博士論文

有機テルル化合物を使用した新規炭素–炭素(C–C)結合形成反応の開発

上村　大護

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   Kamimura, D.; Urabe, D.; Nagatomo, M.; Inoue, M. *Org. Lett.*, **2013**, *15*, 5122.

2. Expanding the scope of Et_3B/O_2-mediated coupling reactions of O,Te-acetal
   Kamimura, D.; Nagatomo, M.; Inoue, M. *Tetrahedron*, **2016**, *72*, 7839.

3. Et_3B-Mediated Two- and Three-Component Coupling Reactions via Radical Decarbonylation of α-Alkoxyacyl Tellurides: Single-Step Construction of Densely Oxygenated Carboskeletons
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略語一覧

Ac : acecyl
An : anisyl
9-BBN : 9-borabicyclo[3.3.1]nonane
Bn : benzyl
Boc : *t*-butoxycarbonyl
Bu : butyl
Bz : benzoyl
DART : direct analysis in real time
DMF : dimethylformamide
Dppf : 1,1'-bis(diphenylphosphino)ferrocene
ESI : electrospray ionization
Et : ethyl
HRMS : high-resolution mass spectrometer
IR : infrared
m.p. : melting point
Me : methyl
n : normal
NMR : nuclear magnetic rsonance
NOE : nuclear overhauser effect
p : para
Ph : phenyl
PTSA : p-toluenesulfonic acid
TBAF : tetrabutylammonium fluoride
TBDPS : *tert*-butyldiphenylsilyl
TBS : *tert*-butyldimethylsilyl
TEMPO : 2,2,6,6-tetramethylpiperidine 1-oxyl
tert, *t* : tertiary
THF : tetrahydrofuran
TLC : thin layer chromatography
TMS : Trimethylsilyl
TOF : time of flight
Tol : tolyl
Ts : tosyl
TTMSS : Tris(trimethylsilyl)silane
UV : ultraviolet

第一章 序論

1-1. テルル(Te)の特徴

テルル(Te)は原子番号 52 の元素であり、周期表において第 16 族に属する、無臭で銀白色の個体半金属である。単体テルルはラットでの経口毒性(LD_{50} 値)は 84 mg/kg と高い毒性を有しており、眼および皮膚への刺激性を有するため、取り扱いには注意が必要である [1)]。

テルルは様々な分野で使用されている。例えば、テルルの添加による快削性等の改善により、自動車部品や精密機械部品として、光ディスクの相変化材料(Ge-Sb-Te)として、太陽電池の半導体(Ge-Te)として、また冷却装置用としてのペルチェ素子(Bi-Te-Sb)として、幅広い分野で使用されている [2)]。一方、有機化学の分野においては、近年までその使用は限られたものであった。

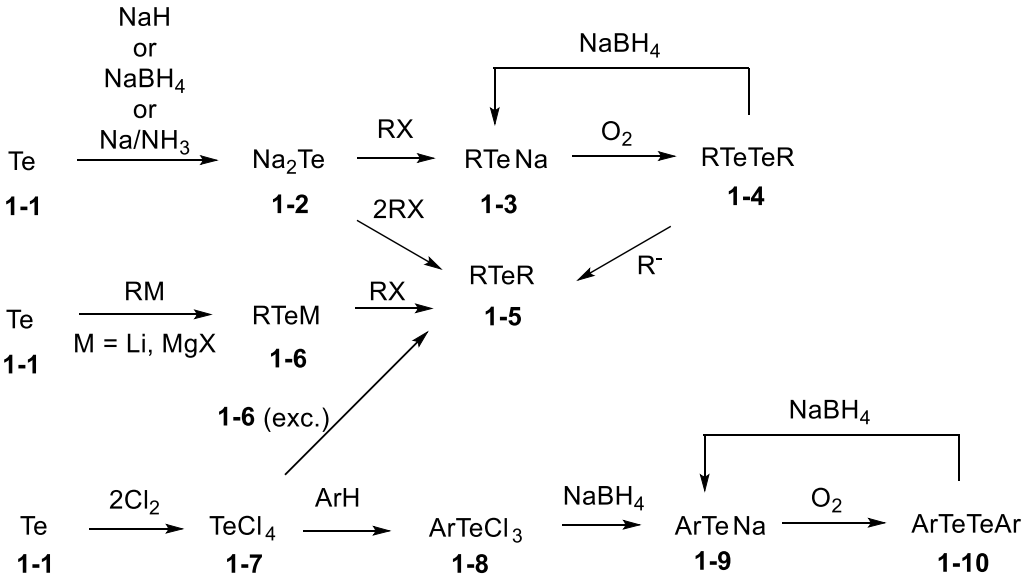
1-2. 有機テルル化合物の調製

有機テルル化合物の調製法について代表的なものを以下に示した [3)](Scheme 1-1)。単体テルル(**1-1**)に水素化ナトリウム、水素化ホウ素ナトリウム、もしくは液体アンモニア中金属ナトリウムを作用させることで、テルル化ナトリウム **1-2** へと変換できる。**1-2** はハロゲン化アルキルとの反応により、有機テルル化合物 **1-3** や **1-5** へと導くことができる。**1-3** は酸素雰囲気下で酸化することでジテルリド **1-4** へ、またジテルリド **1-4** は水素化ホウ素ナトリウムを用いた還元により、再び **1-3** へと変換可能である。**1-4** はアルキルアニオンとの反応により、**1-5** を与える。

1-5 は、単体テルル(**1-1**)にアルキルリチウムやアルキル Grignard 試薬のような有機金属化合物を作用させて得られる **1-6** とハロゲン化アルキルとの反応によっても調製可能である。

1-1 は塩素と反応し、四塩化テルル(**1-7**)を与える。**1-7** は芳香族炭化水素との求電子置換反応により、芳香族テルル化合物 **1-8** となる。**1-8** に水素化ホウ素ナトリウムを反応させることによりテルル化ナトリウム **1-9** へと導くことができ、**1-9** は酸化によりジテルリド **1-10** へと変換できる。

Scheme 1-1. Preparation of organotelluride.



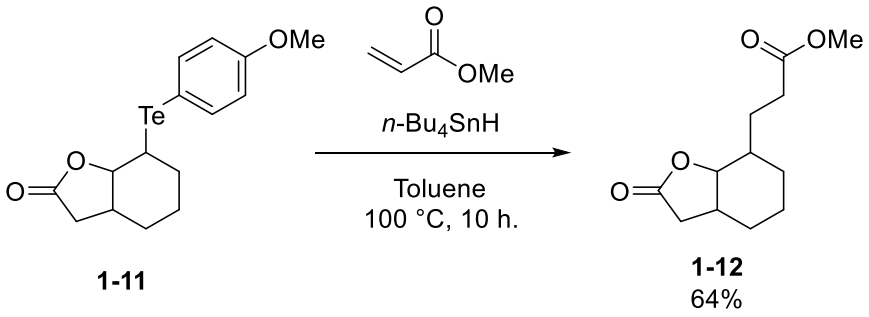
1-3. 有機テルル化合物の反応

1980 年に Clieve らは有機テルル化合物のラジカル反応への適用を初めて報告した [4]。C-Te 結合はその低い結合エネルギー[5]により比較的容易に均等開裂を起こすことから、有機テルル化合物はしばしば Giese 反応 [6]等のラジカル付加反応に使用されている。以下に代表的な反応例を示した。

1-3-1. アルキルテルリドの反応

Comasseto および Gaeta らは、トルエン中テトラノルマルブチルスズヒドリド(*n*-Bu_3SnH)存在下、テルリド **1-11** とアクリル酸メチルを反応させることで、エステル **1-12** を 64%の収率で得ることに成功している(Scheme 1-2)[7]。

Scheme 1-2. Reaction of telluride **1-11** with methyl acrylate

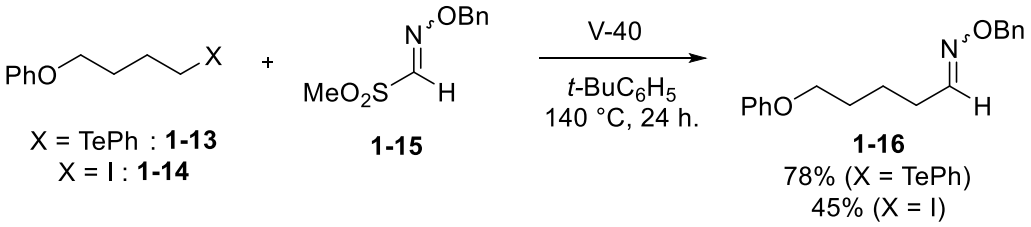


1-11

1-12
64%

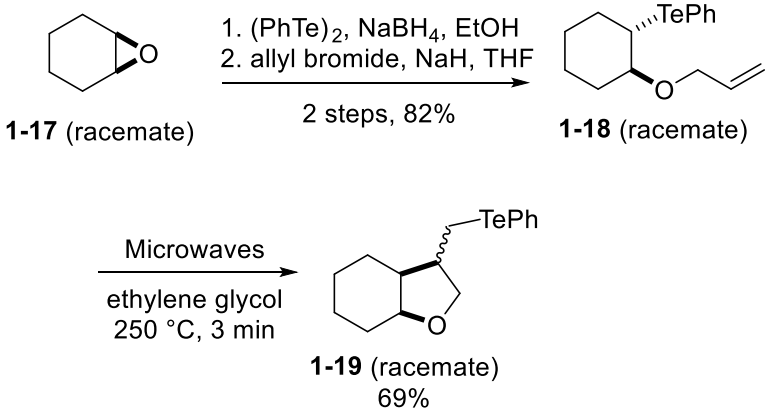
また、Kim および Yoon らは、*tert*-ブチルベンゼン中、V-40 存在下でメタンスルホニルオキシム **1-15** とテルリド **1-13** を反応させることで、二酸化硫黄の脱離を経て 78%の収率で **1-16** を取得した[8]。また、同論文において **1-13** の代わりに、対応するヨウ素体 **1-14** を用いると、オキシム **1-16** の収率は 45%であることが報告されている(Scheme 1-3)。

Scheme 1-3. Radical acylation reactions with methane sulfonyl oxime ether



Ericsson および Engman らは、エポキシド **1-17** から容易に調製可能なテルリド **1-18** を使用し、エチレングリコール中、マイクロウェーブ照射下 250℃で加熱することで、グループ移動型環化体 **1-19** へと導いている(Scheme 1-4)[9]。

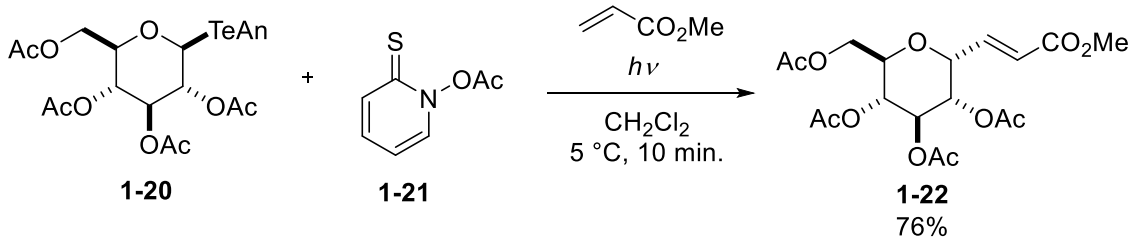
Scheme 1-4. Microwave-assisted group-transfer cyclization



1-3-2. O,Te-アセタールの反応

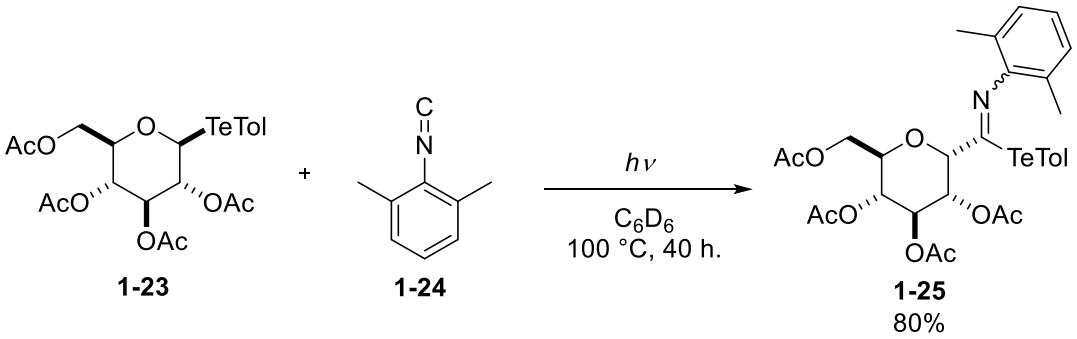
1990 年に Barton らはジクロロメタン中、アクリル酸メチル存在下、O,Te-アセタール **1-20** および *O*-Ac ピリチオン(**1-21**)に対し UV 照射を行うことで、付加体 **1-22** が得られることを報告した(Scheme 1-5)[10]。

Scheme 1-5. Radical reaction of O,Te-acetal



1999 年に山子らは、重ベンゼン中 O,Te-アセタール **1-23** およびイソニトリル **1-24** に対し、UV 照射下 100℃で加熱することで、イミド **1-25** を合成することに成功している(Scheme 1-6)[11]。

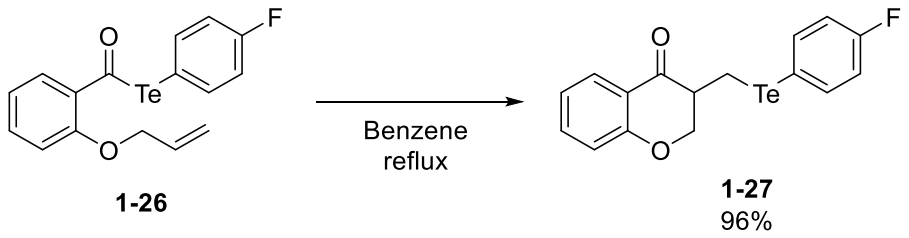
Scheme 1-6. Radical-mediated imidoylation of O,Te-acetal



1-3-3. アシルテルリドの反応

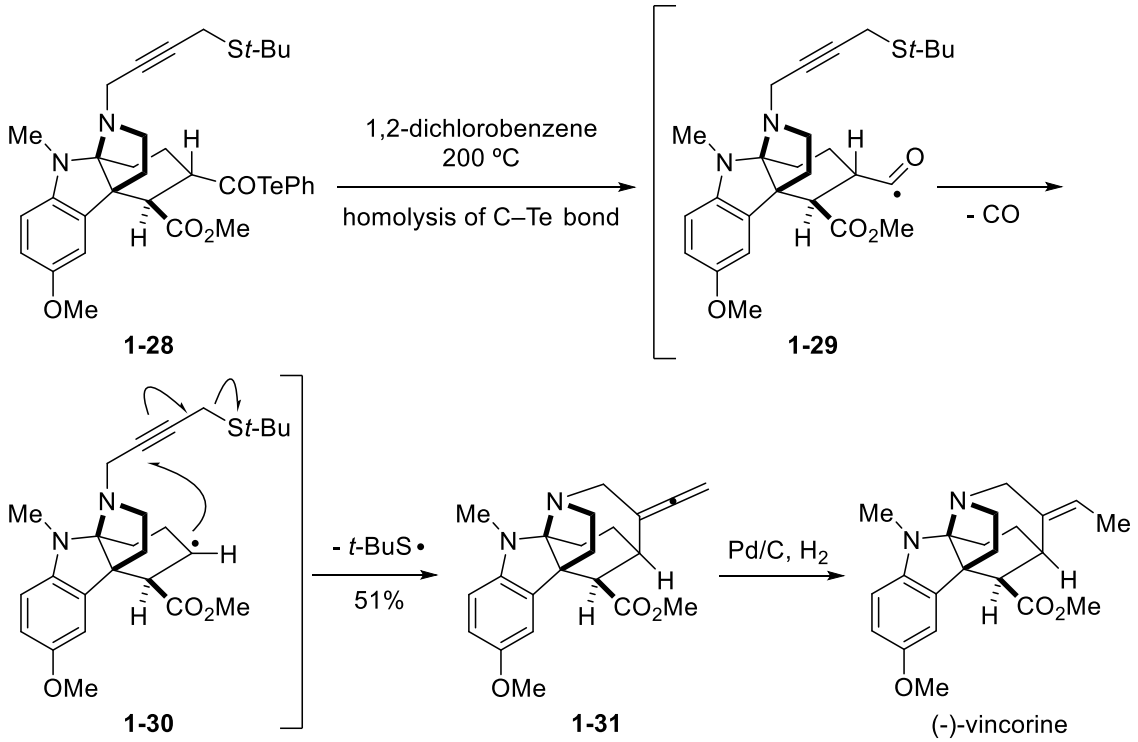
1992 年に Papadatos および Crich らは、アシルテルリド **1-26** をベンゼン中加熱還流することで、グループ移動型ラジカル環化反応を経て、環化体 **1-27** へと導いている(Scheme 1-7)[12]。

Scheme 1-7. Radical cyclization of acyl telluride



2013 年、MacMillan らは、アシルテルリド **1-28** を用いた分子内ラジカル環化反応を鍵反応とした(-)-vincorine の全合成を報告した(Scheme 1-8)[13]。本全合成にでは、アシルテルリドの脱一酸化炭素を経る分子内ラジカル環化反応を鍵反応として用いている。すなわち、アシルテルリド **1-28** を 1,2-ジクロロベンゼン中 200℃に加熱することで、C-Te 結合が開裂し、アシルラジカル **1-29** が生じる。続いてアシルラジカル **1-29** の脱一酸化炭素が進行し炭素ラジカル **1-30** となる。生じた炭素ラジカルが分子内のアルキンに付加することで、7 員環アレン **1-31** が生成する。最後に MacMillan らはアレン **1-31** を還元することで(-)-vincorine の全合成を達成している。

Scheme 1-8. Radical reaction of acyl telluride for total synthesis of (-)-vincorine



以上のように、有機テルル化合物は一般的なラジカル開始剤だけでなく、加熱条件下や光照射下において、有用なラジカル前駆体として働くことが報告されている。この性質を利用することで、複雑な構造を有する骨格形成や天然物への応用が期待できる。しかし、有機テルル化合物の反応性に関する情報は決して十分とは言えず、より詳細な情報収集が求められている。そこで筆者は有機テルル化合物をラジカル前駆体とし、その一般的な反応性情報の収集と新規な炭素−炭素(C−C)結合形成反応の開発を志向し、研究に着手した。

1-4. 参考文献

(1) ナカライテスク株式会社、安全データシート、テルル、2016.

(2) 独立行政法人石油天然ガス・金属鉱物資源機構、マテリアルフロー分析、テルル、2010.

(3) Petragnani, N.; Stefani, H. A. *Tellurium in Organic Synthesis*, 2nd, updated and enlarged ed.; Elsevier: New York, **2007**.

(4) Clive, L. J.; Chittattu, G. J.; Farina, V.; Kiel, W.; Menchen, S. M.; Russell, C. G.; Singh, A.; Wong, C. K.; Curtis, N. *J. Am. Chem. Soc.* **1980**, *102*, 4438.

(5) Akiba, K. *Phosphorus, Sulphur, and Silicon* **1998**, *136*, 239.

(6) Giese, B.; Gonzalez-Gomez, J. A.; Witzel, T. *Angew. Chem. Int. Ed.* **1984**, *23*, 69.

(7) Comasseto, J. V.; Ferraz, H. M. C.; Brandt, C. A.; Gaeta, K. K. *Tetrahedron Letters*, **1989**, *30*, 1209.

(8) Kim, S.; Song, H. J.; Choi, T. L.; Yoon, J. Y. *Angew. Chem. Int. Ed.* **2001**, *40*, 2524.

(9) Ericsson, C.; Engman, L. *J. Org. Chem.* **2004**, *69*, 5143.

(10) Barton, D. H. R.; Ramesh, M.; *J. Am. Chem. Soc*. **1990**, *112*, 891.

(11) Yamago, S.; Miyazoe, H.; Goto, R.; Yoshida, J. *Tetrahedron Lett*. **1999**, *40*, 2347.

(12) Chen, C.; Crich, D.; Papadatos, A. *J. Am. Chem. Soc*. **1992**, *114*, 8313.

(13) Horning, B. D.; MacMillan, D. W. C. *J. Am. Chem. Soc.* **2013**, *135*, 6442.

第二章　有機テルル化合物をラジカル前駆体とした炭素–炭素結合形成反応の開発

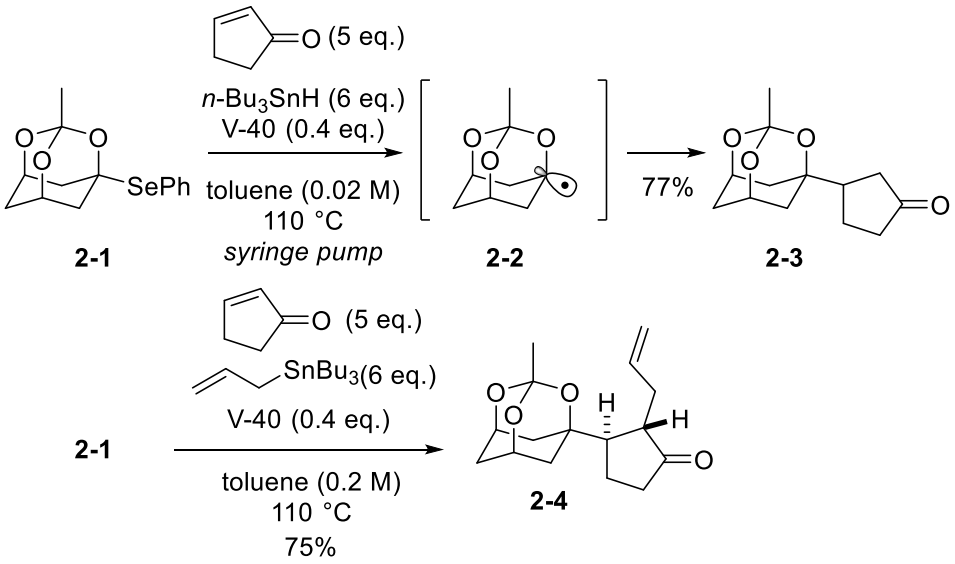
2-1. 研究背景

ラジカル反応は、多置換の炭素環構築に対し有用な反応であり、様々な天然物の全合成に応用されている[1)]。炭素ラジカル種は、高い反応性を示すが電子的に中性である。そのため、多くの極性官能基に直交性を示す。すなわち、極性反応と相補的に組み合わせることで、複雑な構造を有する化合物の構築が可能となる。

天然物合成化学教室(旧　有機反応化学教室、以下、当研究室)では、高度に酸素官能基化された天然物の合成研究に注力しており、このような天然物を合成するにあたり、興味深い特長を有する橋頭位ラジカル[2)]に着目し研究を行っている。橋頭位ラジカルは、非環状の炭素ラジカルと比較し合成上有利な点がある。一つは、高い反応性である。橋頭位ラジカルはかご型構造の頂点にあるため、立体障害が最小化され、結果として高い反応性が期待できる。もう一つは、立体特異的に反応が進行する点である。非環状の炭素ラジカルを用いた場合、ラジカル中心は直ちに立体反転する。一方で、橋頭位ラジカルは立体反転が出来ない第 3 級ラジカルを活性種としており、立体特異的に第 4 級炭素を構築できる。高度に酸素官能基化された天然物の合成には、橋頭位ラジカルの中でも特にα-アルコキシ橋頭位ラジカルを用いることが有用である。

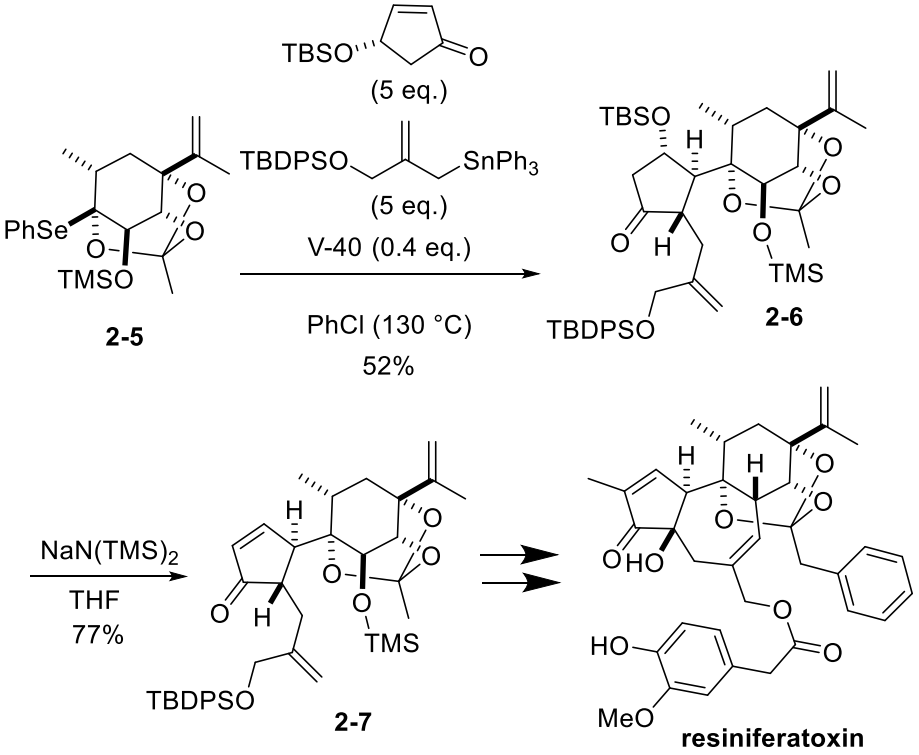
当研究室では、2011 年に O,Se-アセタール **2-1** に対しトルエン中シクロヘキサノン存在下、V-40 および *n*-Bu_3SnH をシリンジポンプで滴下することで、橋頭位ラジカル **2-2** を経てシクロヘキセノン付加体 **2-3** を 77%で得ることに成功している[3)]。合わせて、*n*-Bu_3SnH の代わりにアリルトリブチルスズを用いることで、3 成分連結体 **2-4** を 75%の収率で得ることにも成功している(Scheme 2-1)。

Scheme 2-1. Radical reaction of O,Se-acetal **2-1**



また当研究室では、Scheme 2-1 の反応を鍵反応とした天然物 resiniferatoxin の全合成を達成している(Scheme 2-2)[4]。すなわち、クロロベンゼン中 V-40 存在下、セレニド **2-5**、光学活性シクロペンテノンおよびアリルトリフェニルスズ誘導体を反応させることで、5, 7, 6 員環(A, B, C 環)が縮環した複雑な構造を有するダフナン骨格の A, C 環連結体 **2-6** の取得に成功している。続く塩基によるエノン **2-7** の形成を経て resiniferatoxin へと導いている。

Scheme 2-2. Radical-based annulation of daphnane skeleton



2-2. 作業仮説

O,Se-アセタール **2-1** を用いたラジカル反応は、その高い有用性の一方でいくつかの課題を有している。1 つ目は、過剰量のスズ試薬の使用である。スズ試薬は、しばしば生成物との分離が困難であり、精製作業に手間を要する[5]。また、非常に強い水生環境有害性(GHS 分類:区分 1)や皮膚腐食性/刺激性(GHS 分類:区分 2)を有しており、取り扱いに注意が必要である。2 つ目は、高い反応温度である。*n*-Bu_3SnH 存在下における高温反応では、スズラジカルの付加反応[6]や、還元体の生成[7]等の問題が生じることが多く、収率の低下を招くことから、その回避が望まれる[8]。3 つ目は、厳密な脱気やシリンジポンプを使用した煩雑な反応操作である。このように制限された反応条件は、応用展開の妨げになるだけでなく、再現性の低下やスケールアップ時のトラブルに繋がる。

上記課題のうち、スズ試薬の使用については、他の適切な還元剤を検討することで解決できる可能性が高い。そこで筆者は、高い反応温度の解決のため、C-Se 結合(234 kJ mol^{-1})[9]より結合エネルギーが低く、穏和な条件でラジカルを発生させることが可能であると考えられる C-Te 結合(200 kJ mol^{-1})[9]に着目し、O,Te-アセタール[10] **2-8** を橋頭位ラジカル前駆体として設計した(Figure 2-1)。

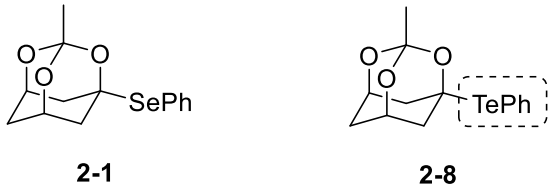
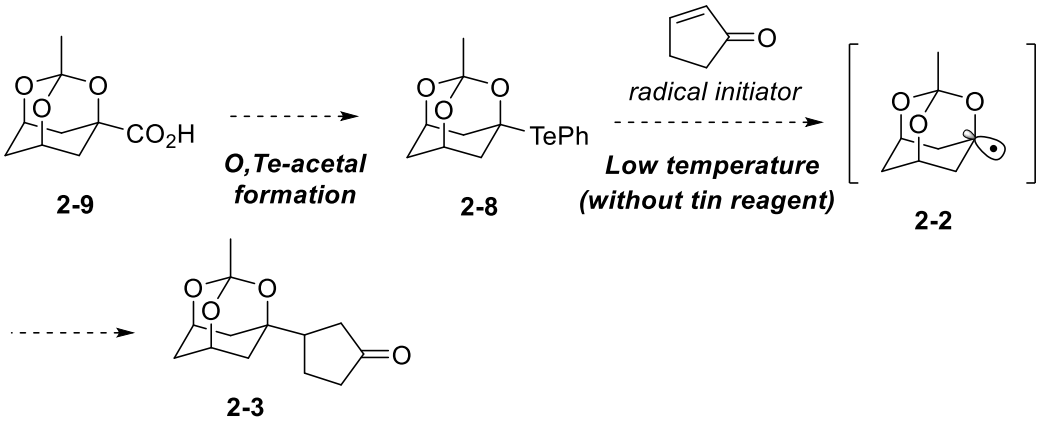


Figure 2-1. Design of O,Te-acetal

Scheme 2-3 に **2-8** を使用したラジカル付加反応の作業仮説を示す。**2-8** は先行研究 [3]に従い、カルボン酸 **2-9** より合成する。続いて **2-8** の低い結合解離エネルギーを利用し、高温条件を必要とすることなく橋頭位ラジカル **2-2** を発生させる。生じた **2-2** に適切なラジカル受容体(下図ではシクロペンテノン)を作用させることで **2-3** を得る。

上記仮説の検証に並行して、適切な還元剤および、煩雑な作業過程を経ることのない反応条件の探索も実施する。

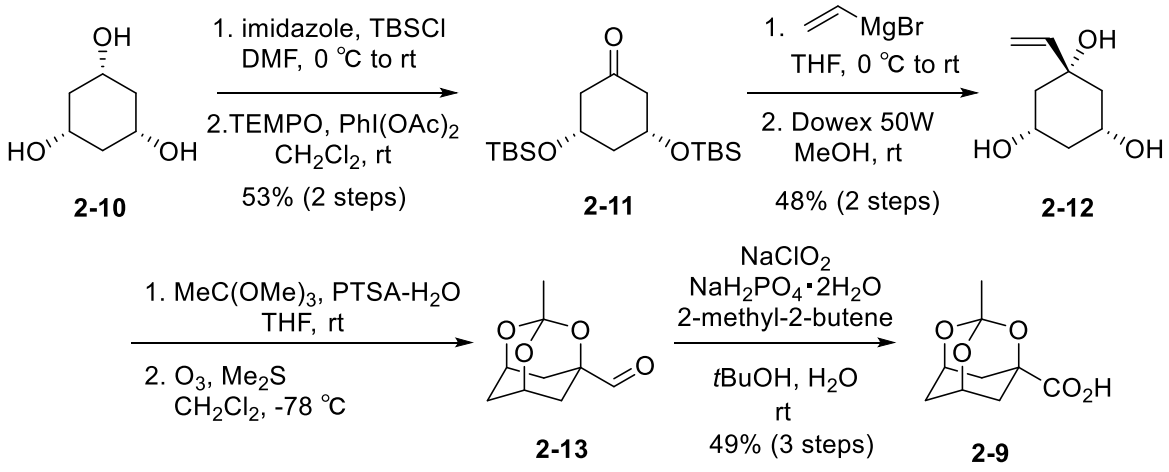
Scheme 2-3. Working hypothesis



2-3. O,Te-アセタールの合成

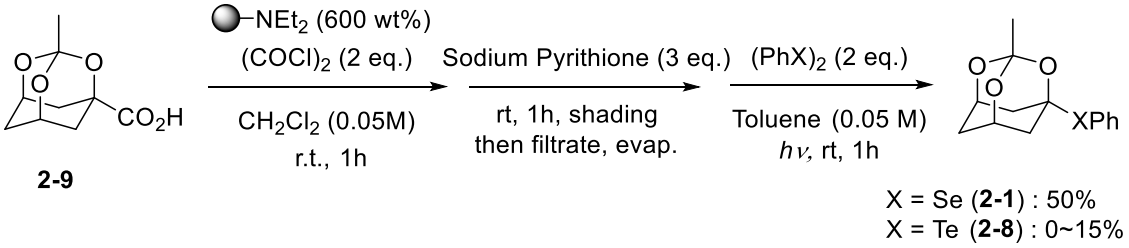
カルボン酸 **2-9** の合成は、**2-1** の合成法 [3]を参考に合成した(Scheme 2-4)。すなわち、1,3,5-シクロヘキサントリオール(**2-10**)の 2 つのアルコールを TBS 保護し、TEMPO 酸化によりケトン **2-11** へと導いた。Grignard 試薬をケトン **2-11** に対して作用させることで、ビニル基を導入した。続く Dowex を用いた TBS 基の除去によりトリオール **2-12** を得た。トリオール **2-12** をオルトエステルとして保護し、ビニル基のオゾン分解により、アルデヒド **2-13** へと変換した。最後に、**2-13** の Pinnick 酸化によりカルボン酸 **2-9** を合成した(7 工程、12%)。

Scheme 2-4. Synthesis of carboxylic acid **2-18**



カルボン酸 **2-9** を使用し、O,Se-アセタール **2-1** と同様の合成法により O,Te-アセタール **2-8** の調製を試みた(Scheme 2-5)。すなわち、カルボン酸 **2-9** に対し、ジクロロメタン中ポリマー担持型ジエチルアミン存在下、オキサリルクロリドを用いて酸クロリドを調製した。続くピリチオンナトリウムとの反応によりバートンエステルへと導いた。その後、濾過によりポリマーを除去し、ジクロロメタンを減圧下で留去した。残留物をトルエンに溶解後、ジフェニルジセレニドもしくはジフェニルジテルリドを添加し、中圧水銀ランプを用いた UV 照射により、O,Se-アセタール **2-1** および O,Te-アセタール **2-8** を得た。

Scheme 2-5. Synthesis of O,Te-acetal **2-8**

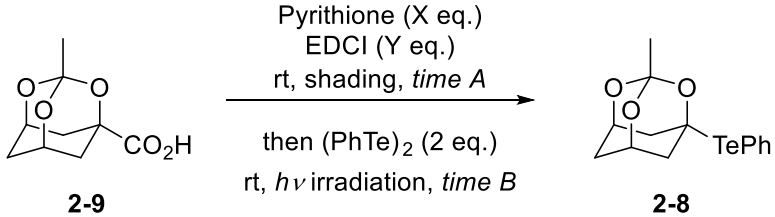


しかし、本手法における **2-8** の収率は低く再現性に乏しかった。再現性の低さが O,Te-アセタールの安定性の低さに起因していると考え、O,Se-アセタール **2-1** の調製の再現性を確認したところ、比較的安定な O,Se-アセタール **2-1** の調製における再現性も乏しかった。そのため、この低い再現性は中間体である Barton エステルの安定性に起因するものであると推察した。本手法では、Barton エステルを形成したのち、ろ過および濃縮工程を経てセレンの導入を行う。そのため、本反応における収率は、調製時の気温や湿度、実験操作に要する時間等に左右される可能性がある。

筆者は、上記の問題点を克服するため、Barton エステルを系内から取り出すことなく、続く付加反応へと進行することが望ましいと考えた。このような手法は Barton らにより DCC を使用した方法[11]が報告されている。そこで、本法を O,Te-アセタール **2-8** の調製に適用した(Table 2-1)。なお、縮合剤はより除去の容易な EDCI を用いることとした。

最初に、カルボン酸 **2-9** に小過剰の Pyrithione(1.25 eq.)および EDCI(1.2 eq)を加え、ベンゼン溶媒中で 1 時間撹拌後、ジフェニルジテルリドを加え 2 時間加熱還流した(entry 1)。合わせて、ジクロロメタン溶媒中、同様の縮合反応を実施した後、ジフェニルジテルリドを加え、中圧水銀ランプによる UV 照射を 2 時間行った(entry 2)。その結果、いずれの条件においても目的の O,Te-アセタール **2-8** を得ることが出来たが、低収率であった。生成した C-Te 結合の光による開裂とそれに伴う分解の可能性を考慮し、entry 2 の条件を基に、UV 照射時間を短くした(entry 3)。すると、わずかに収率の改善が見られたが、満足のいく結果ではなかった。そこで、低い収率は縮合の進行度合いに起因すると想定し、Pyrithione および EDCI を 1.5 当量使用し、反応を実施した(entry 4)。その結果、収率は 56%まで改善した。さらに、溶媒の濃度を 0.05M から 0.1M にすることで収率は 83%となった(entry 5)。また、本反応条件における再現性は良好であった。

Table 2-1. Preparation of O,Te-acetal



2-9　　**2-8**

entry	X (eq.)	Y (eq.)	solvent	time A/time B	result
1	1.25	1.2	benzene (0.05M)	1 h/⊿2 h[a]	18%
2	1.25	1.2	CH_2Cl_2 (0.05M)	1 h/2 h	30%
3	1.25	1.2	CH_2Cl_2 (0.05M)	1 h/1 h	41%
4	1.5	1.5	CH_2Cl_2 (0.05M)	1 h/1 h	56%
5	1.5	1.5	CH_2Cl_2 (0.1M)	1 h/1 h	83%

[a] no *hν* irradiation

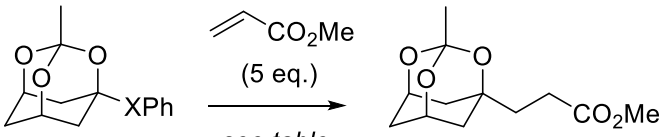
2-4. 反応条件の検討

O,Te-アセタール **2-8** の安定供給が可能となったため、続いてラジカル付加反応への適用を行った(Table 2-2)。最初に、ラジカル受容体をアクリル酸メチルに固定し、反応条件の検討を実施した。Entry 1 には O,Se-アセタール **2-1** をラジカル前駆体として用いた際に最適化された V-40/*n*-Bu_3SnH の条件[12]を示した。同様の条件を使用し、O,Te-アセタール **2-8** とアクリル酸メチルの反応を実施したが、目的の付加体 **2-14** は全く得られなかった(entry 2)。

種々検討の結果、ベンゼン(0.2M)中 60℃で、ラジカル開始剤に V-70 を、還元剤にトリストリメチルシリルシラン(Tris(trimethylsilyl)silane：TTMSS)を用い反応を行うことで、**2-14** を良好な収率(71%)で取得することに成功した。上記反応条件により、スズ試薬の使用を回避することができたが、煩雑な実験操作(シリンジポンプの使用及び厳密な脱気)が必要であるため、さらなる反応条件の改善を試みた。

ラジカル開始剤として低温かつ脱気不要な条件で反応が実施可能なトリエチルボラン[13]を用い反応を行った結果、反応は空気中室温で速やかに進行し、76%の収率で **2-14** を取得することに成功した(entry 4)。また本条件の再現性は良好であった。以上の結果から、O,Se-アセタール **2-1** をラジカル前駆体として用いた反応における 3 つの課題(1.過剰量のスズ試薬の使用、2.高い反応温度、3.煩雑な実験操作)をいずれも解決することが出来た。なお、O,Se-アセタール **2-1** にトリエチルボランを作用させた場合、反応は全く進行しなかった(entry 5)。

Table 2-2. Optimization of two-component coupling reaction with methyl acrylate



X = Se : **2-1**
X = Te : **2-8**

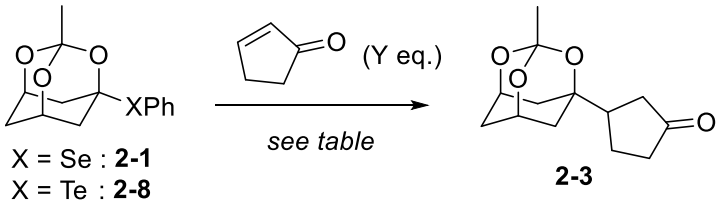
2-14

entry	SM	conditions	Yield (**2-14**)
1[a]	**2-1**	*n*-Bu_3SnH (2 eq.) V-40 (0.4 eq.) Toluene (0.02M), 110 ℃	66%
2	**2-8**	*n*-Bu_3SnH (2 eq.) V-40 (0.4 eq.) Toluene (0.02M), 110 ℃	0%
3	**2-8**	TTMSS (5 eq.) V-70 (0.5 eq.) Benzene (0.2 M), 60 ℃	71%
4	**2-8**	TTMSS (5 eq.) Et_3B in Hex (5 eq.) CH_2Cl_2 (0.2 M), rt.	76%
5[b]	**2-1**	*n*-Bu_3SnH (2 eq.) Et_3B Toluene (0.02M), 110 ℃	0%

[a] ref 5
[b] ref 14

続いて、ラジカル受容体にシクロペンテノンを用いて反応を行った(Table 2-3)。Entry 1 には O,Se-アセタール **2-1** を用いた際の反応条件を示した。本条件では、還元体の生成を抑制するためシリンジポンプを使用する必要がある。Entry 2 では、O,Te-アセタール **2-8** に対し、ジクロロメタン中、シクロペンテノン存在下トリエチルボランをラジカル開始剤として用い反応を行った。その結果、0℃、15 分で反応は進行し、88%の高収率でシクロペンタノン付加体 **2-3** を得ることに成功した。また検討の結果、本反応では還元剤として TTMSS を添加する必要がないことが明らかとなった。O,Se-アセタール **2-1** を entry 2 と同様の条件に付したところ、反応は進行せず原料を 97%の収率で回収するのみであった。本反応性の違いは、C-Se 結合と C-Te 結合の結合解離エネルギー[9]の差に起因すると考えられる。

Table 2-3. Optimization of two-component coupling reaction with cyclopentenone



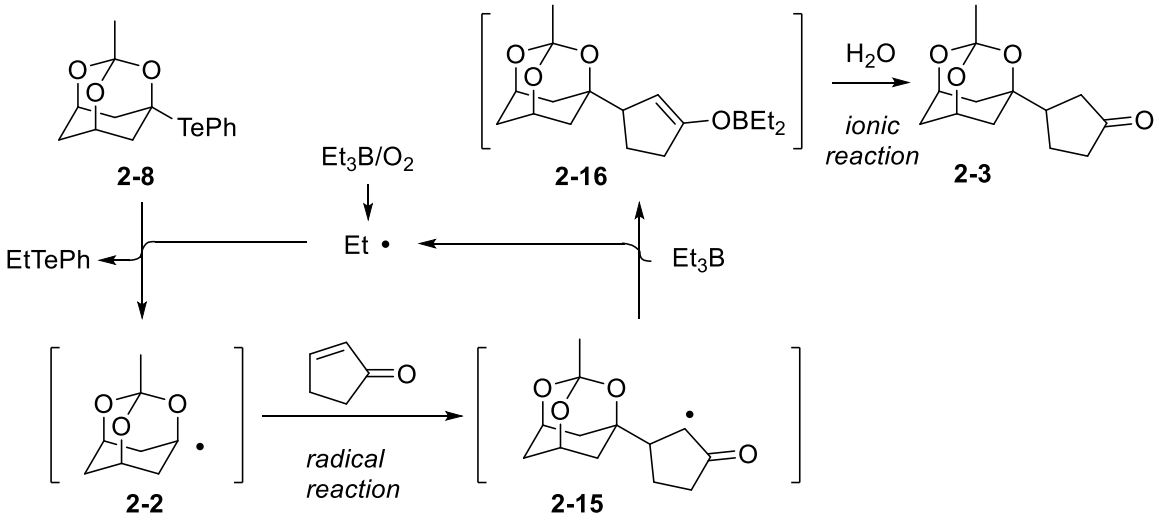
entry	SM	Y (eq.)	condition	Yield (**2-3**)
1[a]	**2-1**	5	n-Bu_3SnH (6 eq.) V-40 (0.6 eq.) Toluene (0.02M), 110 ℃	77%
2	**2-8**	3	Et_3B (3 eq.) CH_2Cl_2 (0.1M), 0 ℃	88%
3	**2-1**	3	Et_3B (3 eq.) CH_2Cl_2 (0.1M), 0 ℃	0%[b]

[a] ref 5
[b] **2-7** was recovered in 97% yield.

2-5. 反応機構

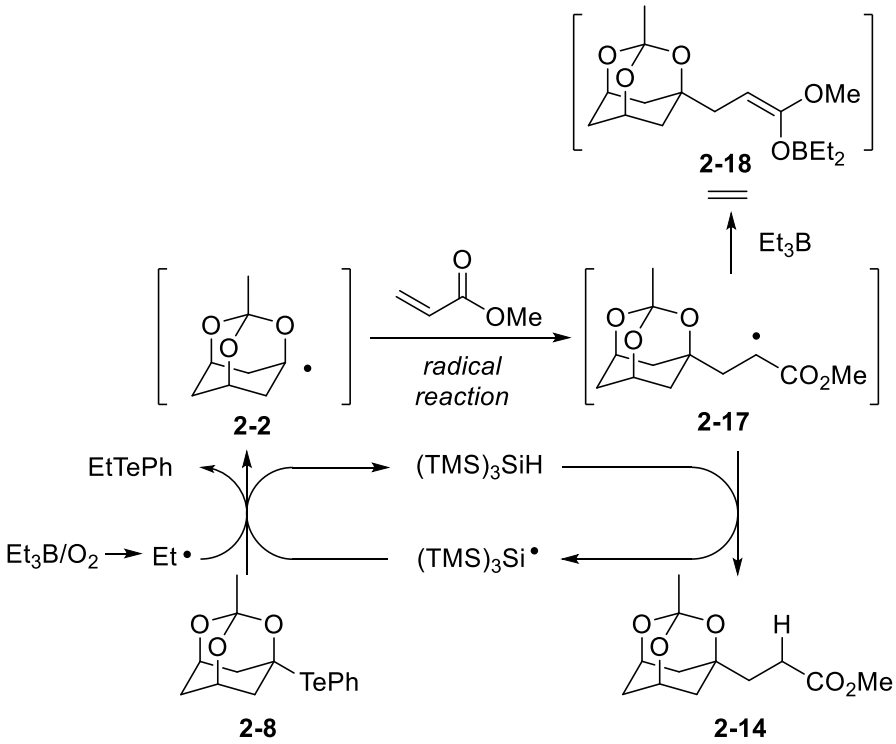
トリエチルボランをラジカル開始剤とした O,Te-アセタール **2-8** のシクロペンテノンに対するラジカル付加反応の反応機構を Scheme 2-6 に示した。本反応はラジカル–極性交差反応[14), 15)]により進行すると考える。すなわち、トリエチルボランと酸素が反応し、エチルラジカルを生じる。エチルラジカルは O,Te-アセタール **2-8** と反応し、橋頭位ラジカル **2-2** を生じる。橋頭位ラジカル **2-2** は、シクロペンテノンにラジカル付加し、α-ケトラジカル **2-15** を与える。**2-15** はトリエチルボランに捕捉され、ボロンエノラート **2-16** が生成し、プロトン化を経て **2-3** となる。

Scheme 2-6. Radical-polar crossover reaction



一方、アクリル酸メチルをラジカル受容体として使用すると、ラジカル–極性交差反応は進行せず、還元剤として TTMSS が必要となる。その理由として、エノンと α,β-不飽和エステル化合物の Lewis 塩基性の差を考慮すると、わずかにエノンのほうが Lewis 塩基性が高いためであると考察した(Scheme 2-7)[16]。すなわち、エノンはトリエチルボランが配位するのに十分な Lewis 塩基性を有しているため、ボロンエノラートが形成可能であるのに対し、α,β-不飽和エステルは Lewis 塩基性が不足していることから、トリエチルボランは配位できず、ボロンエノラートが形成できないためではないかと考えた。実際に、シクロペンテノンに対しては、Scheme 2-6 に示したように、ボロンエノラートを経由していると考えられる反応が進行し、還元剤は不要である。一方、アクリル酸メチルは、ボロンエノラート **2-18** を経由していないためか、エステルの α 位に生じたラジカルを補足する還元剤(TTMSS 等)が必要であるという実験結果が得られている。

Scheme 2-7. Radical reaction with methyl acrylate



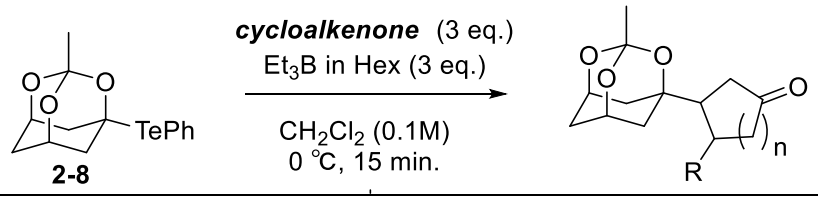
2-6. 基質一般性

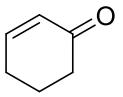
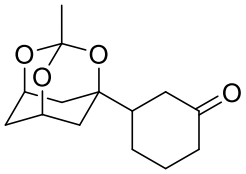
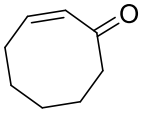
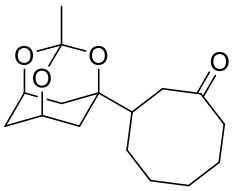
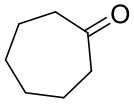
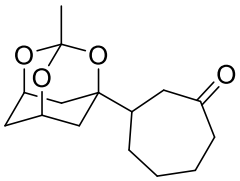
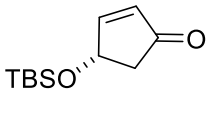
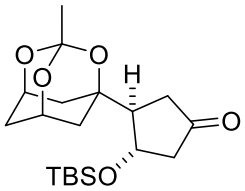
O,Te-アセタール **2-8** を用いたラジカル付加反応が簡便な実験操作かつ温和な条件で進行することを明らかとしたため、続いて基質一般性を調査した。

2-6-1. シクロアルカン類への付加反応

最初に、シクロアルカン類への付加反応を検討した(Table 2-4)。Entry 1~3 では、無置換のシクロヘキサノン、シクロヘプテノンおよびシクロオクテノンをラジカル受容体として使用し反応を検討した。その結果、**2-19**、**2-20** および **2-21** をそれぞれ 80%、78%および 87%の収率で目的の付加体を得ることに成功した。Entry 4 では、光学活性な TBS オキシ基を導入したシクロペンテノンに対する付加反応を行い、付加体 **2-22** を単一のエナンチオマーで得ることが出来た。本反応における選択性発現の理由は、嵩高いトリオキサアダマンタンが TBS 基を避けるよう反対側の面から付加した結果であると想定している。

Table 2-4. Addition to cycloalkenone



entry	cycloalkenone	result	entry	cycloalkenone	result
1		 **2-19** 80% (56%)[a]	3		 **2-21** 87% (45%)[a]
2		 **2-20** 78% (26%)[a]	4		 **2-22** 72% (65%)[a]

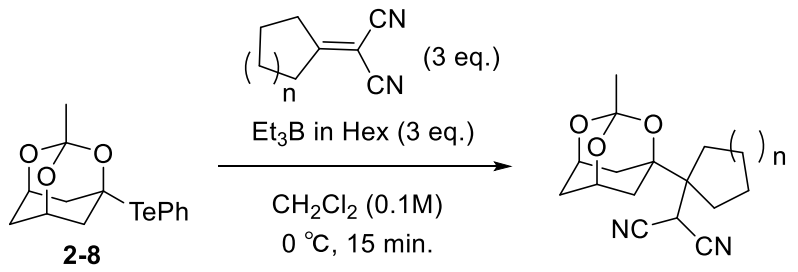
[a] The conditions with O,Se-acetal **2-1** indicated on entry 1 in table 1

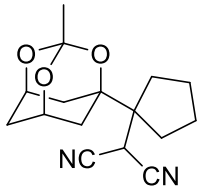
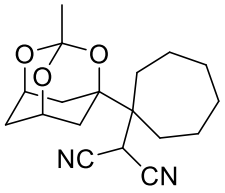
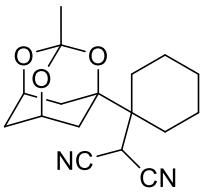
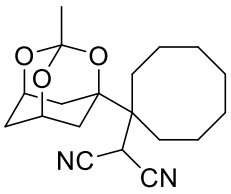
上記付加反応では、前述の O,Se-アセタール **2-1** と V-40 を用いた条件における収率(Table 中、カッコ内)と比較すると、いずれも改善がみられる。この反応性の差は、トリエチルボランの Lewis 酸性に起因していると想定している。すなわち、トリエチルボランがカルボニル酸素に配位することで、共役している二重結合の LUMO エネルギーを低下[17]させ、その結果反応性が向上したのではないかと考える。

2-6-2. マロノニトリル類への付加反応

続いて、シクロアルキリデンマロノニトリル類への付加反応を検討した(Table 2-5)。本反応の進行により、二つの連続する四置換炭素を構築することが出来る。5、6、7 および 8 員環を有するシクロアルキリデンマロノニトリルをラジカル受容体として用い反応を検討した結果、**2-23**、**2-24**、**2-25** および **2-26** をそれぞれ 58%、88%、87%および 33%の収率で付加体を得ることに成功した。

Table 2-5. Addition to malononitrile derivatives



entry	n	result	entry	n	result
1	n = 1	 **2-23** 58% (54%)[a]	3	n = 3	 **2-25** 87% (57%)[a]
2	n = 2	 **2-24** 88% (72%)[a]	4	n = 4	 **2-26** 33% (27%)[a]

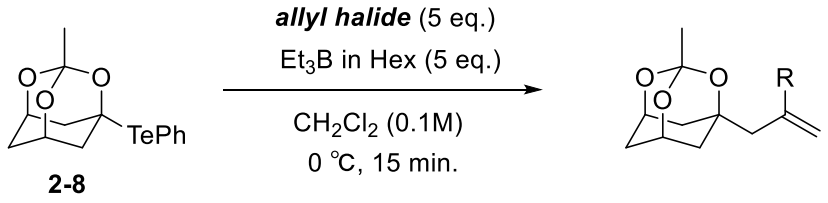
[a] The conditions with O,Se-acetal **2-1** indicated on entry 1 in table 1

本付加反応においても Table 2-4 と同様、O,Se-アセタール **2-1** および V-40 を使用した条件と比較し、一部収率の向上が確認された。しかし、マロノニトリル誘導体に関しては、先のシクロアルケノンのようにトリエチルボランが関与するとは考え難い。おそらく、V-40/*n*-Bu_4SnH の条件では、高い反応温度と *n*-Bu_3SnH の存在により、還元等の副反応が起こっているのではないかと考える。

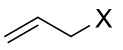
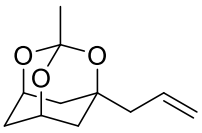
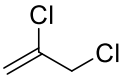
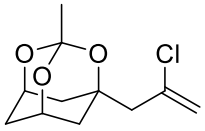
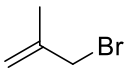
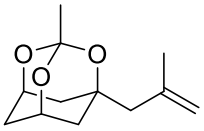
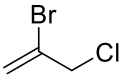
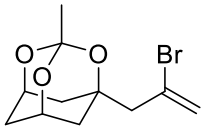
2-6-3. アリルハライド類への付加反応

さらなる汎用性の確認のため、アリルハライド類への付加[18]を検討した(Table 2-6)。Entry 1 では、アリルクロリドおよびアリルブロミドをラジカル受容体として使用し、反応を行った。その結果、アリル体 **2-27** をそれぞれ 65%および 57%で得ることに成功した[19]。同様に、2 位にメチル基が置換したアリルブロミドを使用し反応を行うことで、**2-28** を 61%の収率で得ることができた(entry 2)。ビニル位にクロロ基およびブロモ基を有するアリルクロリドを用いた場合、ビニル位の置換基には影響することなく、それぞれ 79%および 71%の収率で目的の付加体 **2-29** および **2-30** を与えた(entry 3, 4)。なお、entry 3 の反応において、O,Se-アセタール **2-1** および V-40 を用いた条件で反応を実施した場合の収率は 12%(entry 3、カッコ内)であった。これは、過剰量の *n*-Bu_3SnH による還元が影響していると考えられる。

Table 2-6. Addition to allyl halide



2-8

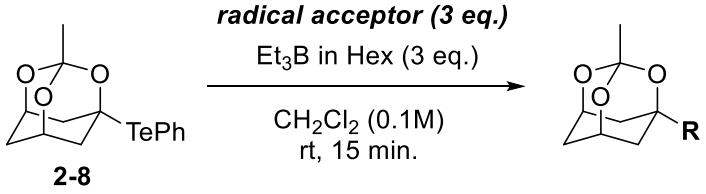
entry	halide	result	entry	halide	result
1		 **2-27 (X=Cl) :** 65% **(X=Br) :** 57%	3		 **2-29** 79% (12%)
2		 **2-28** 61%	4		 **2-30** 71%

2-6-4. C=N 結合への付加反応

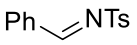
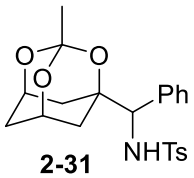
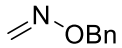
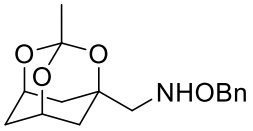
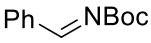
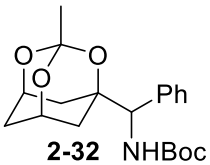
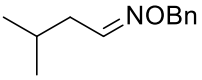
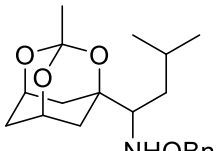
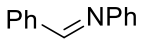
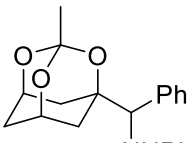
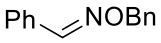
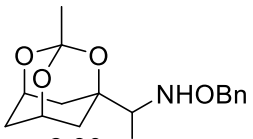
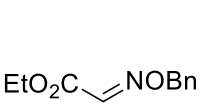
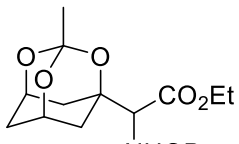
イミンやオキシム類およびヘテロ芳香環は、ラジカル受容体として機能することが知られている[20), 21)]。そこで、上記ラジカル受容体に対し、本反応系を適用できるかを検討した。

最初にイミンおよびオキシム類への付加を検討した(Table 2-7)。まず entry 1~3 では、イミン類への付加を検討した。Boc 基や Ts 基といった電子吸引基を有するものだけでなく、Ph 基が置換したイミンへの付加反応も進行し、目的の付加体 **2-31**、**2-32** および **2-33** をそれぞれ 76%、93%および 60％で得た。続いて、entry 4〜7 は *O*-ベンジルオキシム類への付加を検討した。Entry 4 では、ホルムアルデヒド *O*-ベンジルオキシムをラジカル受容体として用い反応を行った。その結果、良好な収率で目的の付加体 **2-34** を得ることができた。一方、イソプロピル基が置換したベンジルオキシムにおいては、付加体 **2-35** は 38%と低収率であった(entry 4, 5)。低い収率の原因として、イソプロピル基の立体障害とアルキル側鎖の有する電子供与性によるオキシムの求電子性の減少が考えられる。Ph 基やエチルエステルが置換したベンジルオキシムを用いた結果、それぞれ **2-36** および **2-37** を良好な収率で与えた。

Table 2-7. Addition to imine derivatives



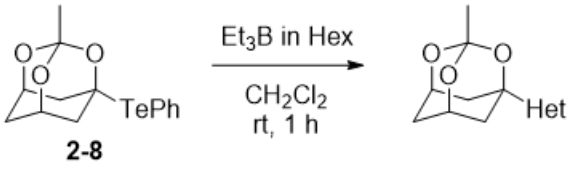
2-8

entry	radical acceptor	result	entry	radical acceptor	result
1		 **2-31** 76%	4		 **2-34** 93%
2		 **2-32** 93%	5		 **2-35** 38%
3		 **2-33** 60%	6		 **2-36** 70%
			7		 **2-37** 89%

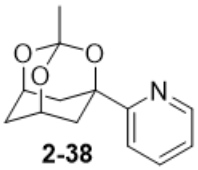
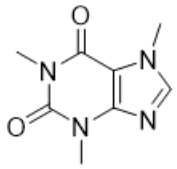
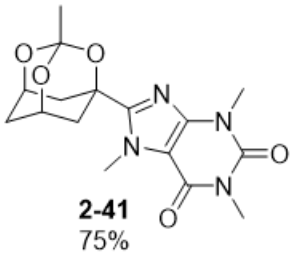
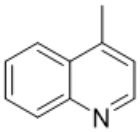
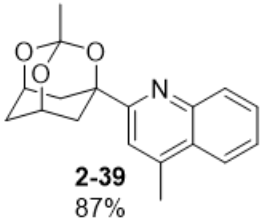
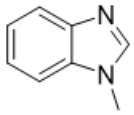
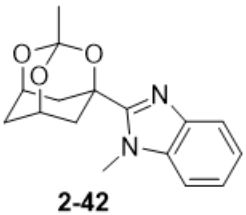
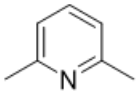
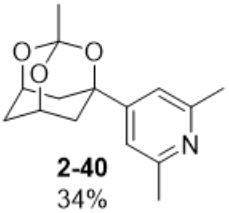
イミン類への反応が効率的に進行したため、続いて含窒素系芳香環類への付加反応を実施した(Table 2-8)。なお、使用するヘテロ芳香環は、Zard、Barton および東郷らの報告 [21b]を参考に、カンファースルホン酸(camphorsulfonic acid:CSA)塩とした。

まず、ピリジンを用い反応を実施した結果、2 位付加体 **2-38** を 45%の収率で得た(entry 1)。Lynch らにより、ピリジンの酸性溶媒中におけるフェニルラジカルの付加は 2 位が優先して進行することが報告されており [22]、本報告とおおよそ一致した結果が得られた。収率の向上を目的に、4 位に置換基を有するレピジンを用いると、2 位付加体 **2-39** を高収率(87%)で得ることができた(entry 2)。一方、ピリジンの 2 位および 6 位が置換された 2,6-ルチジンを使用すると 4 位付加体 **2-40** が得られたが、収率は 34%に留まった(entry 3)。また、カフェイン(entry 4)やベンズイミダゾール(entry 5)のような複数個の窒素原子を有する芳香環においても反応は進行し、目的の付加体 **2-41** および **2-42** を得ることができた。

Table 2-8. Addition to heteroaromatics



2-8

entry	radical acceptor	result	entry	radical acceptor	result
1[a]	 CSA salt	 **2-38** 45%	4[b]	 CSA salt	 **2-41** 75%
2[b]	 CSA salt	 **2-39** 87%	5[a]	 CSA salt	 **2-42** 52%
3[a]	 CSA salt	 **2-40** 34%			

[a] reaction condition : radical acceptor (5 eq.), Et_3B (5 eq.), CH_2Cl_2 (0.2M)
[b] reaction condition : radical acceptor (3 eq.), Et_3B (3 eq.), CH_2Cl_2 (0.1M)

本反応の反応機構は Minisci 反応 [21a]と同様、プロトン化されてより求電子性の高まった含窒素芳香環に求核的にラジカル付加が進行する機構で進行していると考える。

2-7. 小括

以上、筆者は O,Te-アセタール **2-8** の効率的な調製方法の確立、O,Se-アセタール **2-1** を用いたラジカル反応における課題の解決を含む、**2-8** を使用した反応条件の最適化、および **2-8** と種々のラジカル受容体との反応による基質一般性の確認を行った。また、O,Se-アセタール **2-1** との反応性の違いや反応機構の考察を行い、その反応性を明らかとした。

2-8. 参考文献および脚注

(1) For recent reviews on radical reactions, see: (a) Rowlands, G. J. *Tetrahedron* **2009**, *65*, 8603; (b) Rowlands, G. J. *Tetrahedron* **2010**, *66*, 1593.

(2) For a review on bridgehead radicals, see: Walton, J. C. *Chem. Soc. Rev.* **1992**, 105.

(3) Urabe, D.; Yamaguchi, H.; Inoue, M. *Org. Lett*. **2011**, *13*, 4778.

(4) Hashimoto, S.; Katoh, S.; Kato, T.; Urabe, D.; Inoue, M. *J. Am. Chem. Soc*. **2017**, *139*, 16420.

(5) For a review on purification of a product from tin residues, see: Grognec, E. L.;Chrèetien, J.-M.; Zammattio, F.; Quintard, J.-P. *Chem. Rev.* **2015**, *115*, 10207.

(6) van der Kerk, G. J. M.; Noltes, J. G.; Luijten, J. G. A. *J. Appl. Chem*. **1957**, *7*, 356.

(7) For reviews on organotin-mediated radical reactions, see: (a) Neumann, W. P. *Synthesis* **1987**, 665; (b) Curran, D. P. *Synthesis* **1988**, 417.

(8) For reviews on tin-free radical reactions, see: (a) Studer, A.; Amrein, S. *Synthesis* **2002**, 835; (b) Quiclet-Sire, B.; Zard, S. Z. *Chem.-Eur. J.* **2006**, *12*, 6002; (c) Kim, S.; Kim, S. *Bull. Chem. Soc. Jpn.* **2007**, *80*, 809; (d) Akindele, T.; Yamada, K.; Tomioka, K. *Acc. Chem. Res*. **2009**, *42*, 345.

(9) Chivers, T.; Laitinen, R. S. *Chem. Soc. Rev.* **2015**, *44*, 1725.

(10) For reviews on radical reactions of organotellurium compounds, see: (a) Yamago, S. *Synlett* **2004**, 1875; (b) Petragnani, N.; Stefani, H. A. *Tellurium in Organic Synthesis*, 2nd, updated and enlarged ed.; Elsevier: New York, **2007**.

(11) Barton, D. H. R.; Crich, D.; Motherwell, W. B. *Tetrahedron*, **1985**, *41*, 3901.

(12) 山口　裕樹, 東京大学修士論文, 2011 年.

(13) (a) Furukawa, J.; Tsuruta, T.; Inoue, S. *J. Polym. Sci*. **1957**, *26*, 234; (b) Suzuki, A.; Arase, A.; Matsumoto, H.; Itoh, M.; Brown, H. C.; Rogic, M. M.; Rathke, M. W. *J. Am. Chem. Soc*. **1967**, *89*, 5708; (c) Brown, H. C.; Kabalka, G. W. *J. Am. Chem. Soc*. **1970**, *92*, 714; (d)　He, W.; Togo, H.; Waki, Y.; Yokoyama, M. *J. Chem. Soc. Perkin Trans*. **1998**, *1*, 2425; (e) Nozaki, K.; Oshima, K.; Utimoto, K. *Bull. Chem. Soc. Jpn*. **1991**, *64*, 403; For a review, see: (f) Ollivier, C.; Renaud, P. *Chem. Rev*. **2001**, *101*, 3415.

(14) For representative examples, see: (a) Shono, T.; Nishiguchi, I.;Sasaki, M. *J. Am. Chem. Soc*. **1978**, *100*, 4314. (b) Nozaki, K.; Oshima,K.; Utimoto, K. *Tetrahedron Lett.* **1988**, *29*, 1041. (c) Murakami, M.;Kawano, T.; Ito, H.; Ito, Y. *J. Org. Chem*. **1993**, *58*, 1458. (d) Takai, K.;Ueda, T.; Ikeda, N.; Moriwake, T. *J. Org. Chem*. **1996**, *61*, 7990. (e) Takai, K.; Matsukawa, N.; Takahashi, A.; Fujii, T. *Angew. Chem., Int. Ed*. **1998**, *37*, 152. (f) Bazin, S.; Feray, L.; Siri, D.; Naubron, J.-V.; Bertrand, M. P. *Chem. Commun*. **2002**, 2506. (g) Yamamoto, Y.; Nakano, S.; Maekawa, H.; Nishiguchi, I. *Org. Lett*. **2004**, *6*, 799. (h) Godineau, E.; Landais, Y. *J. Am. Chem. Soc*. **2007**, *129*, 12662.

(15) For reviews on the radical-polar cross over reactions, see: (a) Ryu, I. *Chem. Rec*. **2002**, *2*, 249. (b) Murphy, J. A. The Radical-Polar Crossover Reaction. In *Radicals in Organic Synthesis*, Renaud, P., Sibi, M. P., Eds.;Wiley-VCH:Weinheim, 2001; Vol. 1, pp 298. (c) Godineau, E.; Landais, Y. *Chem.-Eur. J*. **2009**, *15*, 3044.

(16) Filgueiras, C. A. L.; Huheey, J. E. *J. Org. Chem.* **1976**, *41*, 49.

(17) Beraud, V.; Gnanou, Y.;Walton, J. C.; Maillard, B. *Tetrahedron Lett.* **2000**, *41*, 1195.

(18) Huval, C. C.; Singleton, D. A. *Tetrahedron Lett.* **1993**, *34*, 3041.

(19) 本付加反応において、アリルハライドおよびトリエチルボランは 5 当量用いて反応を実施しているが、一部の反応において、当量を減らすことで原料が残存し、生成物との分離が困難となる傾向があったためである。

(20) (a) Booth, S. E.; Jenkins, P. R.; Swain, C. J.; Sweeney, J. B. *J.Chem. Soc., Perkin Trans. 1*, **1994**, 3499. (b) Bertrand, M. P.; Feray, L.; Nouguier, R.; Stella, L. *Synlett* **1998**, 780. (c) Miyabe, H.; Ueda, M.; Naito, T. *Chem. Commun*. **2000**, *20*, 2059. (d) Miyabe, H.; Ueda, M.; Naito, T. *J. Org. Chem*. **2000**, *65*, 5043.

(21) (a) Minisci, F.; Bernardi, R.; Bertini, F.; Galli, R.; Perchinummo, M. *Tetrahedron* **1971**, *27*, 3575. (b) Barton, D. H. R.; Garcia, B.; Togo, H.; Zard, S. *Tetrahedron Lett*, **1986**, *27*, 1327.

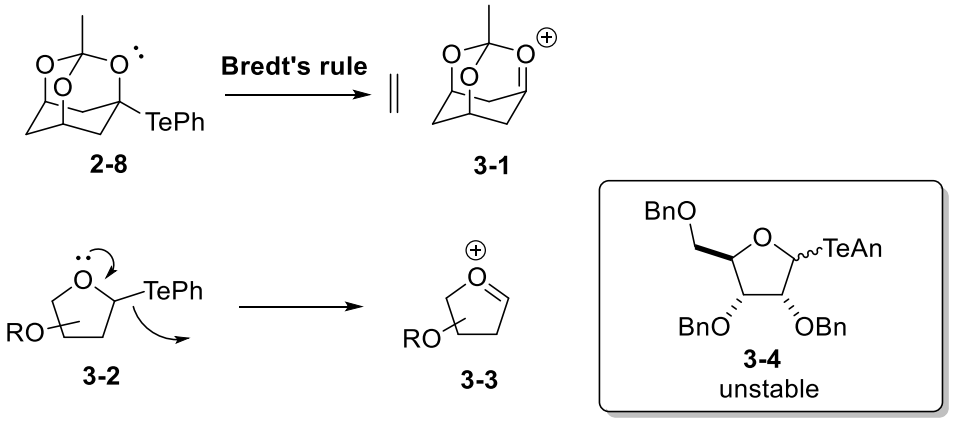
(22) Lynch, B. M.; Chang, H. S. *Tetrahedron Lett*. **1964**, *5*, 2965.

第三章　アシルテルリドをラジカル前駆体とした炭素–炭素結合形成反応の開発

3-1. 研究背景

2 章において筆者は、O-Te アセタール **2-8** より生じたラジカルを使用し、さまざまなラジカル受容体に対する付加反応を実施した。しかし、本反応は O,Te-アセタールの不安定性により、基質が限定される。例えば、O,Te-アセタール **2-8** は Bredt 則に反する橋頭位オキソカルベニウムイオン(**3-1**)を与えないため、本手法が有用な反応である一方、糖類に代表される **3-2** のような構造は、容易にオキソカルベニウムイオン(**3-3**)を生じることから不安定であり、本反応の適用に制限がある。実際、東郷らにより、糖テルリド **3-4** は、その不安定性から精製後速やかに使用することが好ましいと報告されている [1](Scheme 3-1)。

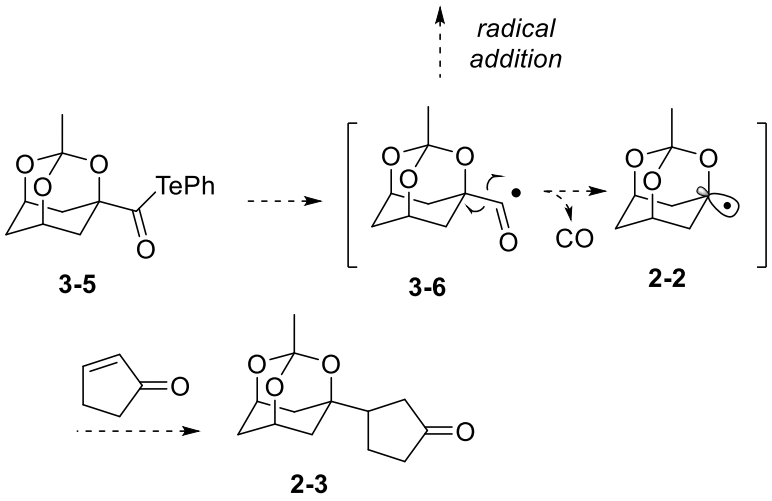
Scheme 3-1. Stability of O,Te-acetal



3-2. 作業仮説

上記不安定性を解消し、汎用性の高い反応を確立するため、筆者はアシルテルリド **3-5** を設計した。**3-5** に対し、適切なラジカル開始剤を作用させることで、アシルラジカル **3-6** が生じる。このアシルラジカル **3-6** が反応するより前に脱カルボニルが進行すれば、橋頭位ラジカル **2-2** となることから、O,Te-アセタール **2-8** と同様に、アシルテルリド **3-5** はラジカル **2-2** の前駆体となることが期待できる(Scheme 3-2)。

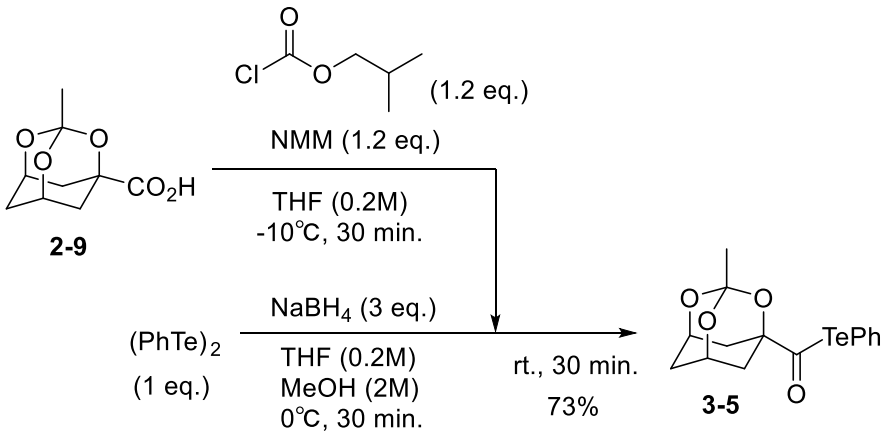
Scheme 3-2. Working hypothesis



3-3. アシルテルリドの合成

アシルテルリドは、MacMillan らにより報告されている手法 [2)]を参考に合成した。すなわち、トリオキサアダマンタンカルボン酸 **2-9** に対し、クロロギ酸イソブチルを用い混合酸無水物を調製したのち、別途調製したテルルアニオン種を反応させることでアシルテルリド **3-5** を得た(Scheme 3-3)。

Scheme 3-3. Preparation of acyl telluride



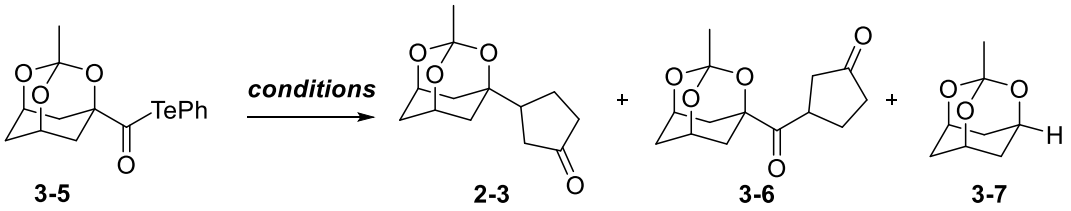
3-4. アシルテルリドの反応性

アシルテルリド **3-5** を用い、シクロペンテノンをラジカル受容体として反応性の調査を行った(Table 3-1)。2 章にて最適化した反応条件(Table 2-3 を参照)を基に、脱カルボニルの進行速度を考慮に入れ反応温度は 0℃から室温に変更し行った。すなわち、0.1 M のジクロロメタン中、シクロペンテノン 3 当量存在下、室温でトリエチルボラン 3 当量を加え、15 分撹拌した(entry 1)。その結果、収率は低いながら、脱カルボニルが進行し、トリオキサアダマンチルラジカル **2-2** がシクロペンテノンに付加した化合物 **2-3** を得ることに成功し、本反応条件(室温)においても脱カルボニルが進行することを明らかにした。一方、主生成物はアシルラジカルがシクロペンテノンへ付加した **3-6** であったため、詳細な反応条件の検討を行った。Entry 1 の結果から、脱カルボニルの速度よりアシルラジカルのシクロペンテノンへの付加速度がわずかに早いと考えられる。そこで、希釈条件下もしくはシクロペンテノンを減量し、ラジカル受容体の濃度を下げることで、アシルラジカルの付加速度を遅延することができれば、脱カルボニルが優先して進行し、**2-3** が優位に得られると想定した。

entry 2 ではシクロペンテノンを 2 当量、ジクロロメタンを 0.005 M とし反応を実施したところ、予想通り **2-3** を優先的に得ることに成功した。しかし、同時に還元体 **3-7** が副生物として得られた。溶媒濃度の検討の結果、溶媒濃度を 0.02 M とし反応を行うことで、還元体 **3-7** の生成が抑制され、目的の付加体 **2-3** を 60%の収率で得ることに成功した(entry 3)。続いて、反応温度と脱カルボニルの関連を調べるため、0℃で反応を行った。本反応温度においても脱カルボニルは完全に抑制されることなく進行することが明らかとなった(entry 4)。

反応溶媒にベンゼンを使用し、トリエチルボラン非存在下、加熱条件にて反応を実施したが、反応は進行しなかった(entry 5)。また、entry 5 の条件にトリエチルボランを添加すると、アシルラジカル付加体 **3-6** の生成は抑制され、付加体 **2-3** を 32%の収率で与えた(entry 6)。

Table 3-1. Investigating reactivity of acyl telluride **3-5**



entry	cyclopentenone	conditions	temp./time	**2-3**	**3-6**	**3-7**
1	3 eq.	Et_3B in Hex (3 eq.) CH_2Cl_2 (0.1M)	rt./15 min.	25%	50%	0%
2	2 eq.	Et_3B in Hex (3 eq.) CH_2Cl_2 (0.005M)	rt./5 h	52%	<17% (1:1 mixture)	<17%
3	2 eq.	Et_3B in Hex (3 eq.) CH_2Cl_2 (0.02M)	rt./15 min.	60%	26%	0%
4	2 eq.	Et_3B in Hex (3 eq.) CH_2Cl_2 (0.02M)	0℃/15 min.	<35%	47%	0%
5	2 eq.	benzene (0.005M)	60℃/30 min.	No Reaction		
6	2 eq.	Et_3B in Hex (3 eq.) benzene (0.005M)	60℃/30 min.	32%	trace	0%

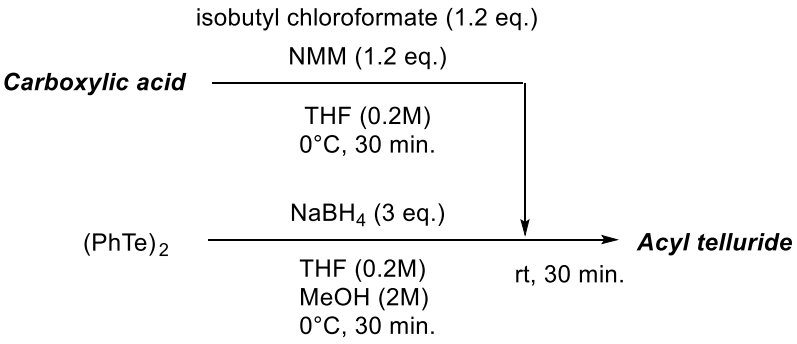
3-5. アシルテルリドの糖類への応用

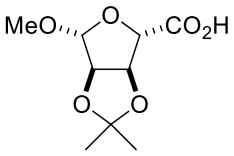
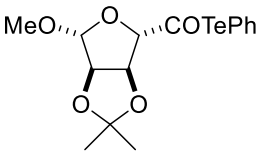
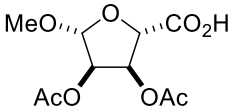
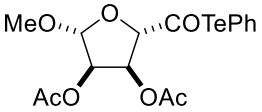
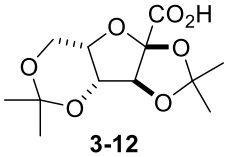
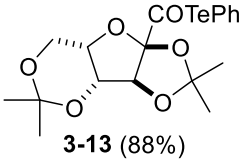
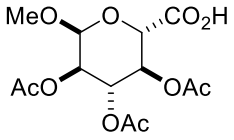
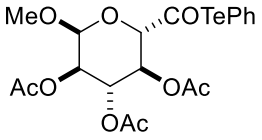
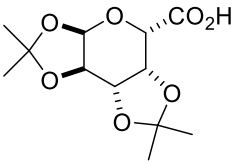
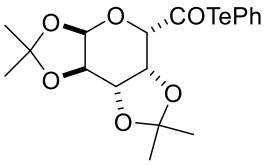
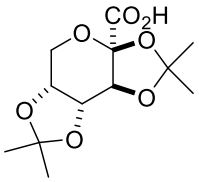
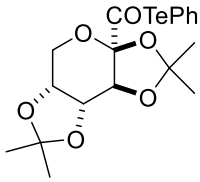
アシルテルリド **3-5** を用いた脱カルボニル反応が室温で進行することが明らかになった一方で、アシルラジカルの付加は完全には抑制できなかった。そこで筆者は本反応の脱カルボニルの反応速度は、生成するラジカル(ここでは橋頭位ラジカル **2-2**)の安定性に依存すると仮定した。すなわち、脱カルボニルにより生じるラジカルが、より安定なものであれば、アシルラジカルの付加より優先して脱カルボニルが進行すると考えた。そこで、立体電子効果が得られる化合物として糖類を選択し、アシルテルリドの脱カルボニル反応の検討を開始した。

3-5-1. 各種糖類のアシルテルリドの調製

種々の糖類のアシルテルリドを、Scheme 3-3 の手法に習って合成した(Table 3-2)。リボース誘導体 **3-9**[3]および **3-11**[4](entry 1, 2)、2-ケトグロン酸 **3-13**(entry 3)、グルコース誘導体 **3-15**[5](entry 4)、ガラクトース誘導体 **3-17**[3](entry 5)、およびフルクトース誘導体 **3-19**(entry 6)を基質とし反応を行った結果、いずれも良好な収率で対応するアシルテルリドを得た。

Table 3-2. Preparation of acyl telluride

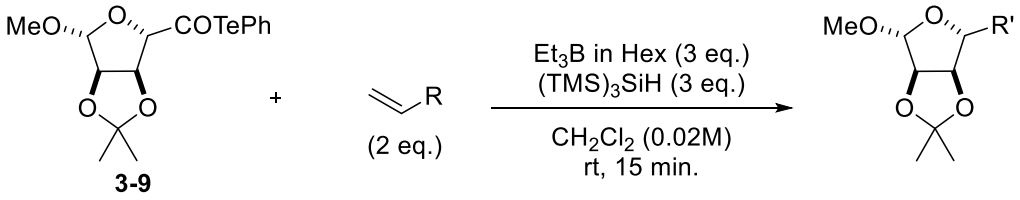


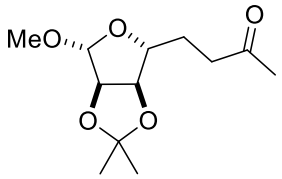
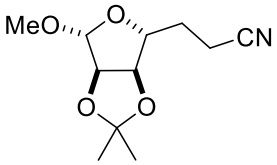
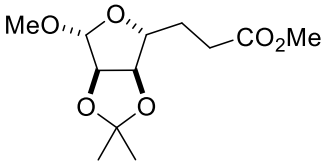
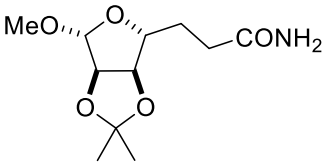
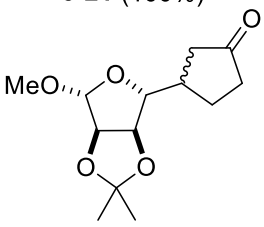
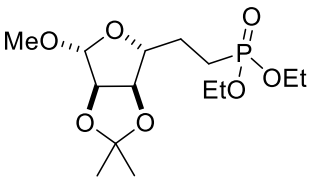
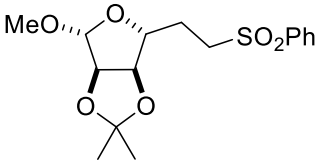
entry	carboxylic acid	acyl telluride
1	 **3-8**	 **3-9** (90%)
2	 **3-10**	 **3-11** (90%)
3	 **3-12**	 **3-13** (88%)
4	 **3-14**	 **3-15** (90%)
5	 **3-16**	 **3-17** (86%)
6	 **3-18**	 **3-19** (78%)

3-5-2. リボース誘導体の反応性

まず、リボースより誘導したアシルテルリド **3-9** を用い、各種ラジカル受容体に対する付加反応[6]を検討した(Table 3-3)。なお、本検討では必要に応じて$(TMS)_3SiH$ を添加した(Scheme 2-6, 2-7 を参照)。最初に、メチルビニルケトンをラジカル受容体として利用し反応を行った(entry 1)。その結果、想定通り速やかに脱カルボニルが進行し、付加体 **3-20** を 92%の収率で得ることに成功した(entry 1)。同様に、各種求電子オレフィン類への付加はいずれもアシルラジカル付加体を生じることなく、高い収率で進行した(entries 2~7)。

Table 3-3. Investigating reactivity of acyl telluride **3-9**

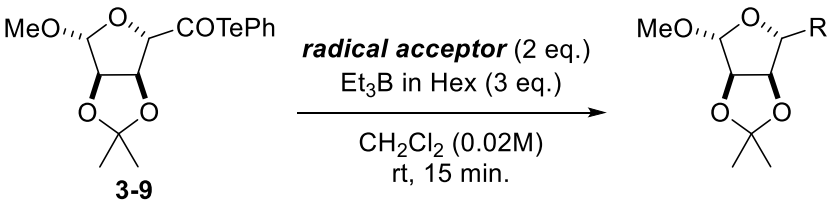


entry	result	entry	result
1[a]	 **3-20** (92%)	5	 **3-24** (73%)
2	 **3-21** (100%)	6	 **3-25** (92%)
3[a]	 **3-22** (82%)	7	 **3-26** (77%)
4	 **3-23** (100%)		

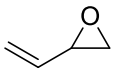
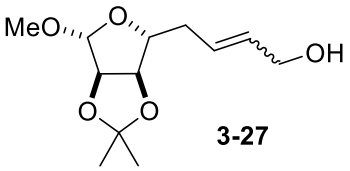
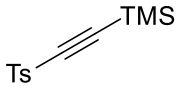
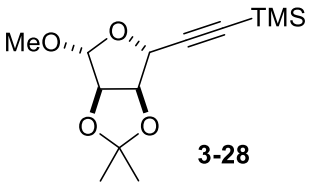
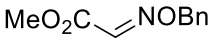
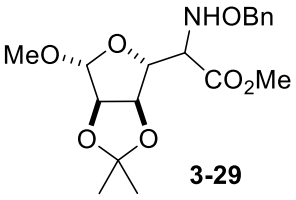
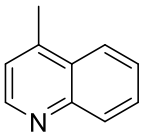
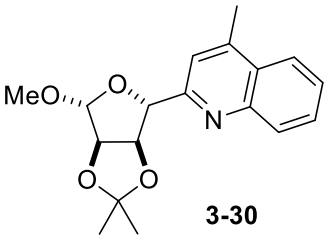
[a] $(TMS)_3SiH$ was not used

アシルテルリド **3-9** の更なる反応性を確認するため、ビニルオキシラン、アルキン、および C=N 結合に対する反応を行った(Table 3-4)、まず、2-ビニルオキシランをラジカル受容体として用い反応を行った結果、アリルアルコール体 **3-27** を 28%の収率で得ることができた(entry 1)。また、トリメチル(トシルエチニル)シラン [7]をラジカル受容体として用いることで、TMS アルキニル体 **3-28** を 34%の収率で得た(entry 2)。C=N 結合に対する付加も進行し、良好な収率でアミノ酸誘導体 **3-29** およびレピジン付加体 **3-30** を得ることに成功した(entry 3, 4)。

Table 3-4. Investigating reactivity of acyl telluride **3-9**



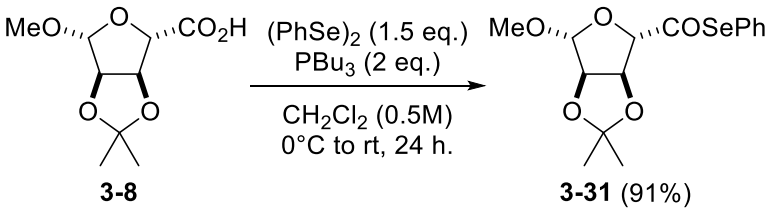
3-9

entry	radical acceptor	result	yield
1		 **3-27**	28%
2		 **3-28**	34%
3		 **3-29**	75%
4	 CAS salt	 **3-30**	66%

3-5-3. アシルセレニドを用いた反応性の確認

本反応におけるテルルの必要性の確認のため、アシルセレニド **3-31** を調製し [8]、その反応性を確認した(Scheme 3-4)。

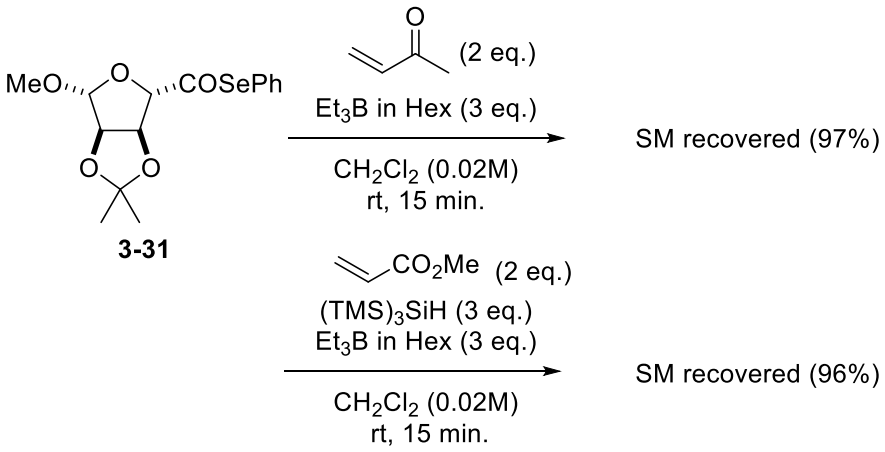
Scheme 3-4. Preparation of acyl selenide **3-31**



3-8 **3-31** (91%)

アシルセレニド **3-31** に対し、Table 3-3 の条件下メチルビニルケトンおよびアクリル酸メチルとの反応を行った。その結果、いずれも反応は全く進行せず原料回収に終った(Scheme 3-5)。よって、本反応にはテルルの存在が重要であることが明らかになった [8]。

Scheme 3-5. Reactivity of acyl selenide

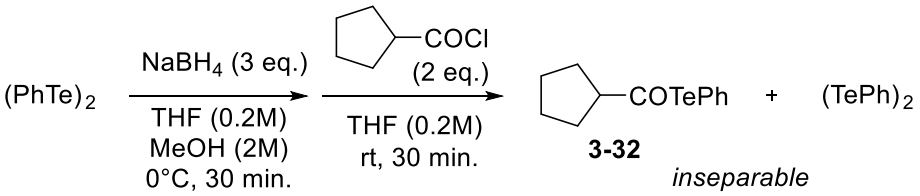


3-31

3-5-4. 酸素原子が脱カルボニルへ与える影響の確認

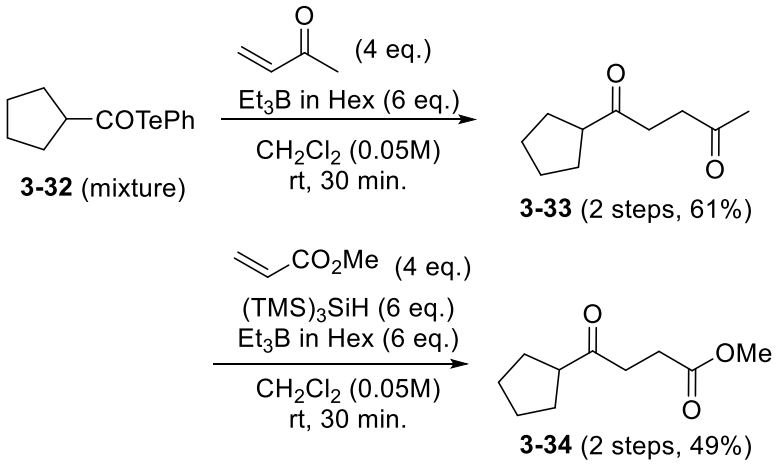
続いて酸素原子の重要性の確認を行った。本検討には、シクロペンタン誘導体 **3-32** をモデル基質として設計し調製を行った(Scheme 3-6)。しかし、目的とする **3-32** は得られるものの、わずかにジフェニルジテルリドが残存し、その分離が困難であった。そのため、分離することなくそのまま付加反応へと適用した。

Scheme 3-6. Preparation of acyl telluride **3-32**



アシルテルリド **3-32** とメチルビニルケトンおよびアクリル酸メチルとの反応を行った結果、脱カルボニルは進行せず、アシルラジカル付加体 **3-33** および **3-34** のみが得られた(Scheme 3-7)。以上の結果から、酸素原子の存在が脱カルボニルに寄与している事が示唆された。

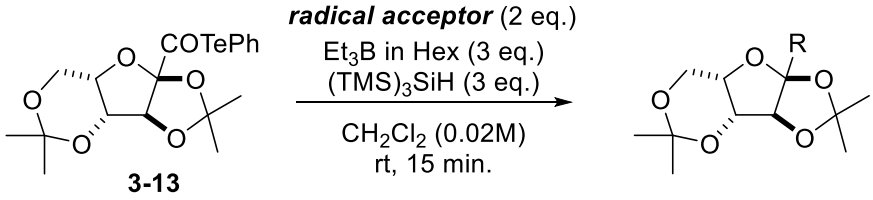
Scheme 3-7. Reactivity of acyl telluride **3-32**

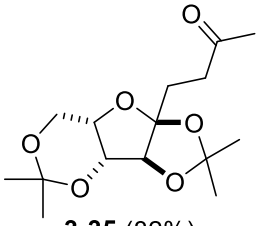
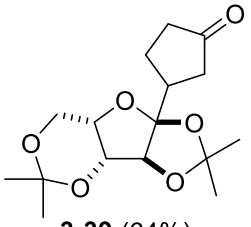
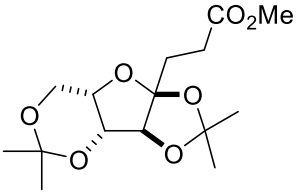
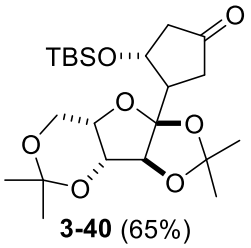
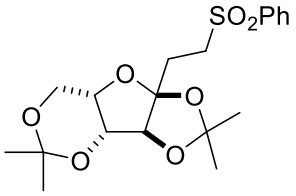
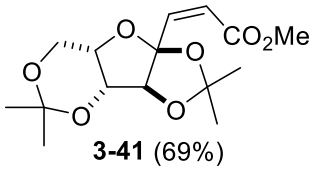
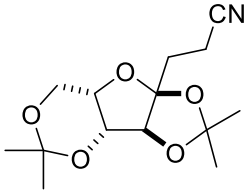
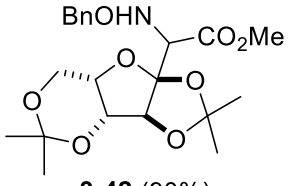


3-5-5. 2-ケト-L-グロン酸誘導体の反応性

本反応を用いた 4 級炭素の構築を志向し、2-ケト-L-グロン酸誘導体 **3-13** を基質として選択し、反応を行った(Table 3-5)。Table 3-3 同様、種々の電子不足オレフィン類を用い反応を実施した結果、目的の付加体を高収率で与えた(entry 1~5)。光学活性エノンに対する付加も、Table 2-4、entry 4 と同様の選択性にて反応は進行し、良好な収率かつ立体選択的に進行した(entry 6)。プロピオール酸メチルをラジカル受容体として用いた場合、シス体選択的に付加体が得られた(entry 7)。本立体選択性は、1,2-ジエン中間体の TTMSS による還元が立体的に空いている面より進行するためであると推定した。オキシムとの反応も 90%と高い収率で進行した(entry 8)。

Table 3-5. Investigating reactivity of acyl telluride **3-13**



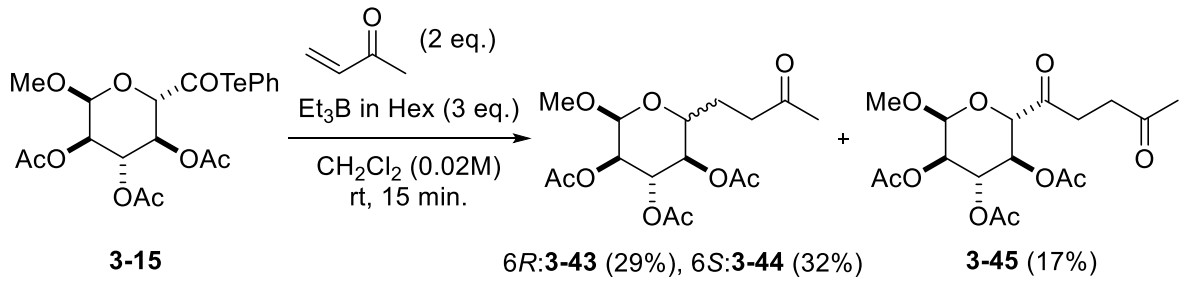
entry	result	entry	result
1[a]	 **3-35** (82%)	5[a]	 **3-39** (64%)
2	 **3-36**(91%)	6[a]	 **3-40** (65%)
3	 **3-37** (91%)	7	 **3-41** (69%)
4	 **3-38** (71%)	8[b]	 **3-42** (90%)

[a] $(TMS)_3SiH$ was not used
[b] CH_2Cl_2 (0.1M)

3-5-6. 六炭糖類の反応性

さらなる基質の適用性を確認するため、六炭糖への適用を検討した。最初に、グルコース誘導体 **3-15** を基質とし、メチルビニルケトンとの反応を検討した(Scheme 3-8)。その結果、脱カルボニルが進行し、メチルビニルケトンと付加した **3-43** および **3-44** がそれぞれ 32%および 29%、また、アシルラジカル付加体 **4-45** が 17%得られた。本基質において、脱カルボニルが進行せずにアシルラジカル付加体が得られた理由の考察のため、さらなる基質の調査を実施した。

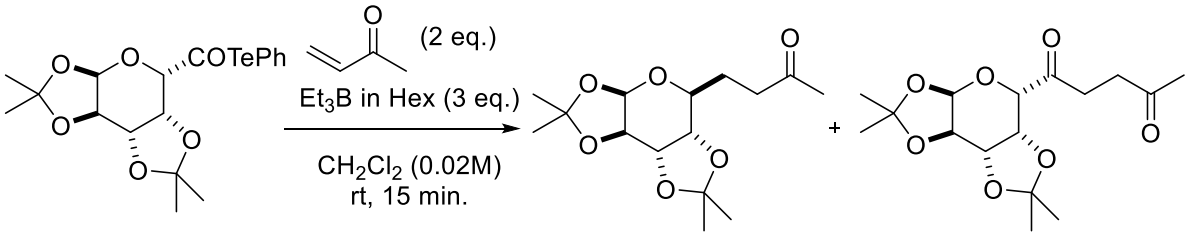
Scheme 3-8. Investigating reactivity of acyl telluride **3-15**



3-15　　6*R*:**3-43** (29%), 6*S*:**3-44** (32%)　　**3-45** (17%)

より立体が強固に固定されたガラクトース誘導体 **3-17** を基質とし反応を行った結果、脱カルボニルが進行した付加体 **3-46** が 21%のみ得られ、アシルラジカル付加体 **3-47** が 45%の収率で得られた(Scheme 3-9)。

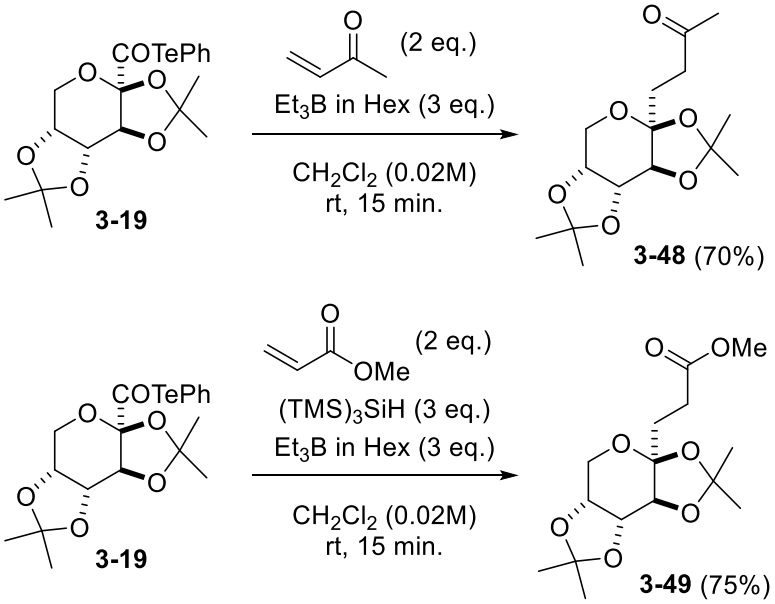
Scheme 3-9. Investigating reactivity of acyl telluride **3-17**



3-17　　**3-46** (21%)　　**3-47** (45%)

一方、フルクトース誘導体 **3-19** を用いると、アシルラジカル付加体は得られず、脱カルボニル体 **3-48** および **3-49** を良好な収率で得た(Scheme 3-10)。

Scheme 3-10. Investigating reactivity of acyl telluride **3-19**



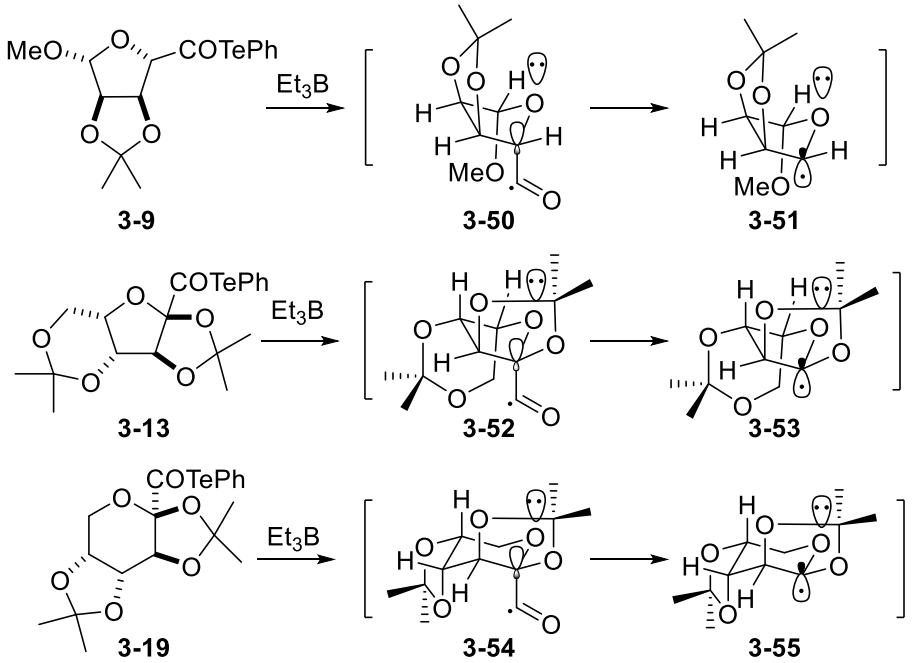
3-19　　**3-48** (70%)

3-19　　**3-49** (75%)

3-6. 反応性の考察

上記の結果を基に、筆者は脱カルボニル反応の反応機構を以下のように考察した(Scheme 3-11)。すなわち、**3-9**、**3-13** および **3-19** は生じるアシルラジカル **3-50**、**3-52** および **3-54** の C-CO 結合の反結合性軌道と隣接する酸素原子の孤立電子対との軌道の重なりが良好であり、立体電子効果 [10] により、脱カルボニルが進行し易くなっている。こうして、速やかに脱カルボニルが生じることで生じたラジカル **3-51**、**3-53** および **3-55** はメチルビニルケトン等のラジカル受容体と反応することにより、立体選択的に目的の付加体を得ることが出来る。

Scheme 3-11. Mechanism of decarbonylation reaction. (part 1)

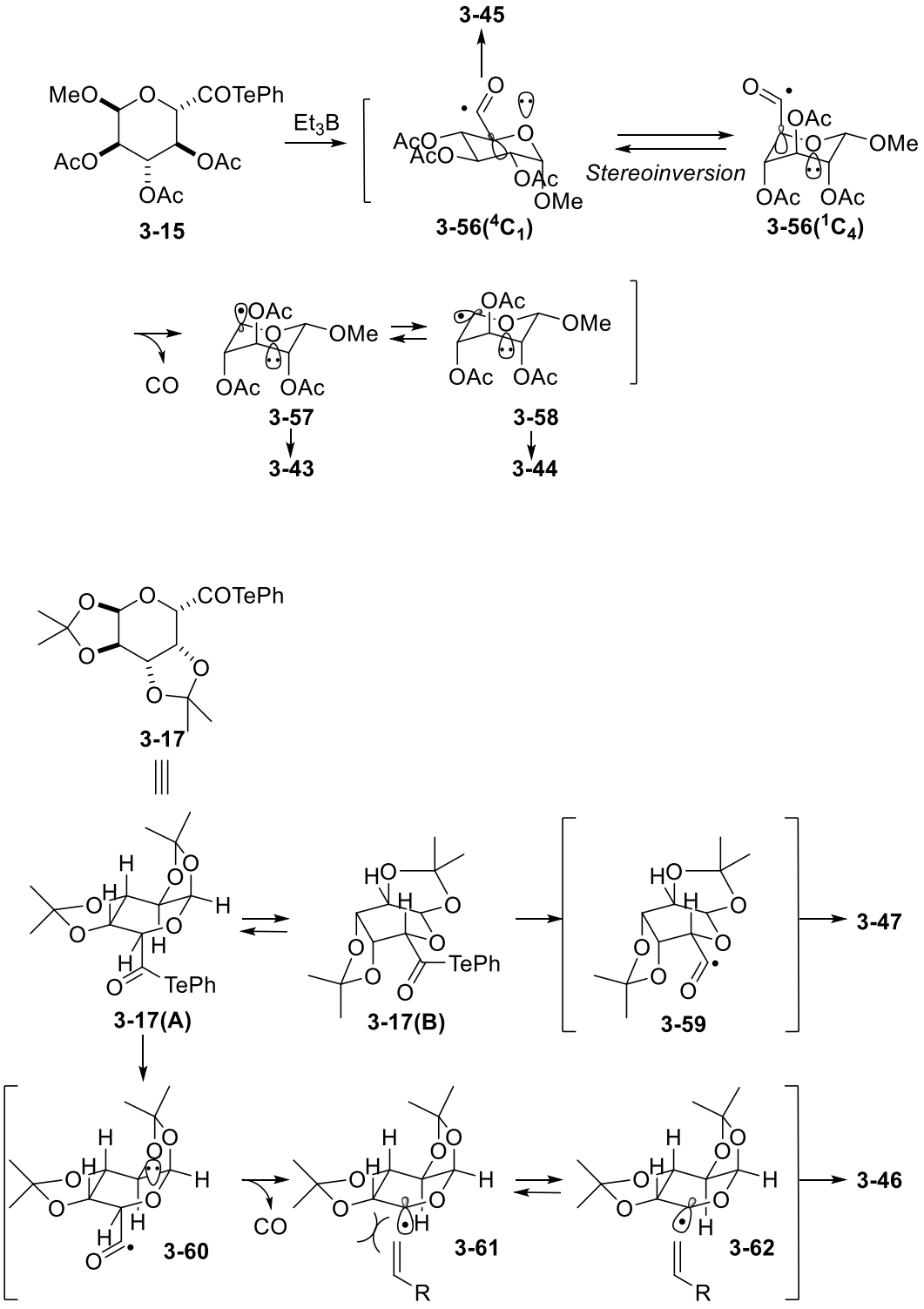


一方、上記化合物とは異なり、グルコース誘導体 **3-15** からは、3 種の化合物が得られる。本反応性に関して、筆者は以下のように考察した。

まず、C-Te 結合が開裂し、アシルラジカル **3-56** が生じる。**3-56** は $^{4}C_{1}$ および $^{1}C_{4}$ の配座間の素早い平衡にあると考えられる [9]。アシルラジカル **3-56(**$^{4}C_{1}$**)**はメチルビニルケトンと反応し **3-45** を生じるのに対し、**3-56(**$^{1}C_{4}$**)**は先に示したように速やかに脱カルボニルが進行する。生じたラジカル **3-57** および **3-58** からそれぞれ **3-43** および **3-44** が得られる。**3-43** と **3-44** の生成比がおおよそ同程度であるのは、立体電子効果および 1,3-ジアキシャル相互作用の影響から各配座間のエネルギー差がほぼ同程度になったためであると想定した。

また、ガラクトース誘導体 **3-17** はねじれ舟形配座 A および B の平衡関係にあると考えられる。配座 B からは、アシルラジカル **3-59** を経て **3-47** が得られる。配座 A からは、アシルラジカル **3-60** を経て脱カルボニル体 **3-61** が生じる。ラジカル **3-61** は concave 面となることから反応が進行せず、**3-62** からの付加が進行し **3-46** が得られたと考える。

Scheme 3-12. Mechanism of decarbonylation reaction (part 2)



3-7. 小括

以上筆者は、各種糖類のアシルテルリドを合成し、その反応性を調査した。その結果、アシルラジカルからの脱カルボニルが穏和な条件かつ速やかに進行しα-アルコキシ炭素ラジカルが生じること、本系内にラジカル受容体を共存させることで、種々の付加体が高収率で得られることを明らかとした。

3-8. 参考文献および脚注

(1) He, W.; Togo, H,; Yokoyama, M. *Tetrahedron Lett*. **1997**, *38*, 5541.

(2) Horning, B. D.; MacMillan, D. W. C. *J. Am. Chem. Soc.* **2013**, *135*, 6442.

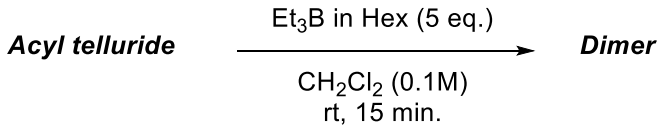
(3) Huang, L.; Teumelsan, N.; Huang, X. *Chem. Eur. J*. **2006**, *12*, 5246.

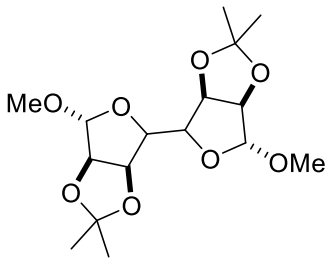
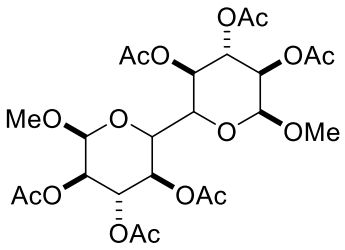
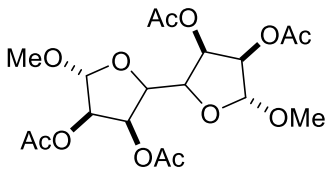
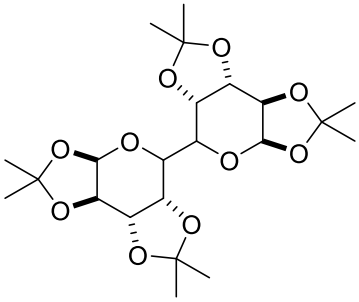
(4) Cai, L.; Li, Q.; Ren, B.; Yang, Z.-J.; Zhang, L.-R.; Zhang, L.-H.*Tetrahedron*, **2007**, *63*, 8135.

(5) Polakova, M.; Pitt, N.; Tosin, M.; Murphy, P. V. *Angew. Chem. Int. Ed*. **2004**, *43*, 2518.

(6) 本反応において、ラジカル受容体を加えずに反応を行うことで、良好な収率で二量体を取得することに成功した(Table 3-6)。

Table 3-6. Investigation of dimerization reaction

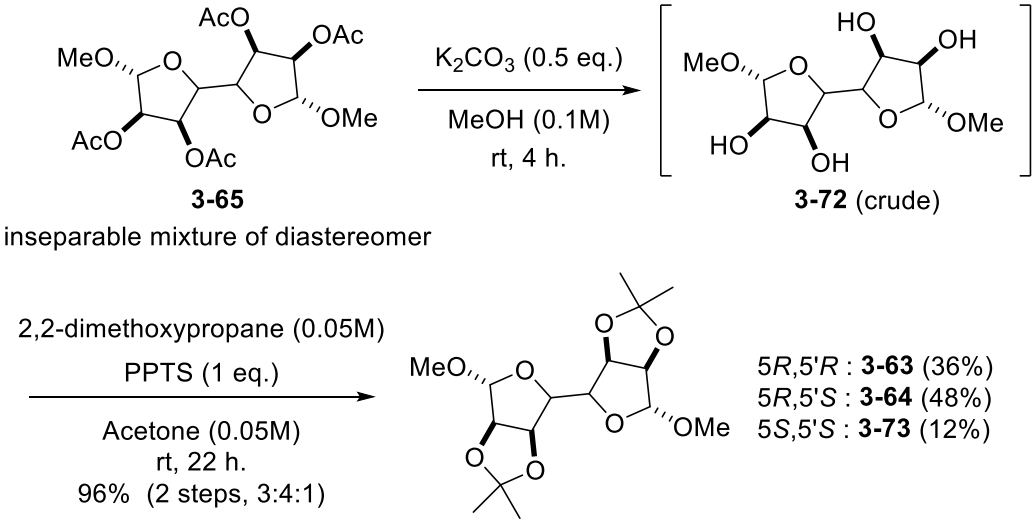


entry	telluride	dimer	entry	telluride	dimer
1	**3-9**		3	**3-15**	
		5*R*,5'*R* : **3-63** (9.4%)[a] 5*R*,5'*S* : **3-64** (13%)[a]			6*S*,6'*S* : **3-66** (31%) 6*S*,6'*R* : **3-67** (27%) 6*R*,6'*R* : **3-68** (11%)
		5*R*,5'*R* : **3-63** (37%) 5*R*,5'*S* : **3-64** (39%)			
2	**3-11**		4	**3-17**	
		3-65 (88%) (*mixture*)			6*R*,6'*R* : **3-69** (22%) 6*R*,6'*S* : **3-70** (44%) 6*S*,6'*S* : **3-71** (22%)

[a] CH_2Cl_2 (0.02M)

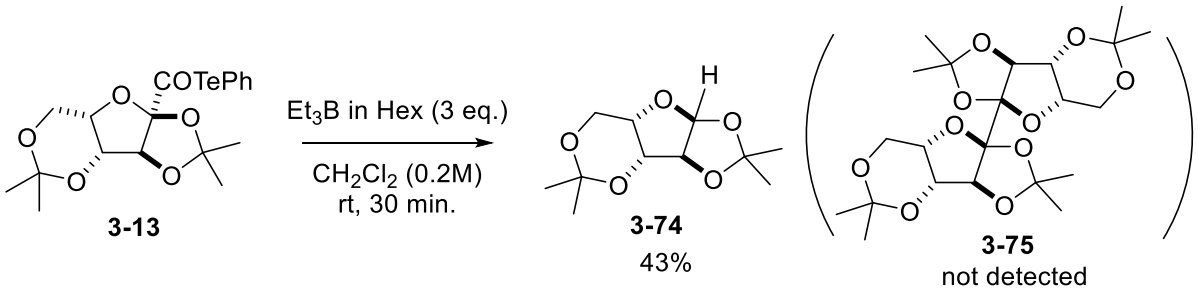
3-65 は保護基を変換することで各異性体を単離し、**3-63**、**3-64** および **3-73** をそれぞれ 36%、48%および 12%で得た(Scheme 3-13)。

Scheme 3-13. Determination of the relative configuration of **3-65**



3-13 の二量化を検討した結果、還元体 **3-74** のみを与え、目的の **3-75** を得ることは出来なかった(Scheme 3-14)。

Scheme 3-14. Application to construction of two vicinal quaternary carbon centers



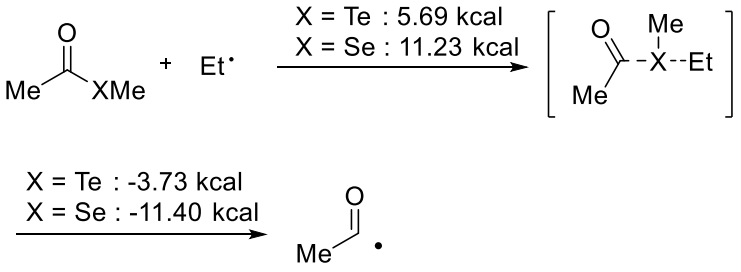
(7) Liautard, V.; Robert, F.; Landais, Y. *Org. Lett.* **2011**, *13*, 2658.

(8) Horvat, S. M.; Schiesser, C. H. *New J. Chem.* **2010**, *34*, 1692.

上記文献では、アシルテルリドおよびセレニドのカルコゲン原子上でのラジカル置換反応における DFT 計算による活性化エネルギーが報告されている。エチルラジカルによる置換反応に関しては、Scheme 3-15 における X がテルルの場合、正反応の活性化エネルギーが 5.69 kcal/mol であり、基底状態のエネルギーも原系であるアシルテルリドよりも生成系であるアシルラジカルの方が 1.96 kcal/mol 低い。一方、X がセレンの場合、正反応の活性化エネルギーが 11.23 kcal/mol であり、アシルテルリドのそれより高い値となっている。また、基底状態のエネルギーは原系のアシルラジカルよりも生成系であるアシルセレニドの方が 0.17 kcal/mol 低い。

以上の結果から、アシルセレニドよりもアシルテルリドの方が高い反応性を示し、下記正反応が進行しやすいことが想定される。また、先に示した実験からは、本計算結果を支持する結果が得られた。

Scheme 3-15. Calculated energy barriers



(9) Boger, D. L.; Mathvink, R. J. *J. Org. Chem*. **1992**, *57*, 1429.

(10) Abe, H.; Shuto, S.; Matsuda, A. *J. Am. Chem. Soc.* **2001**, *123*, 11870.

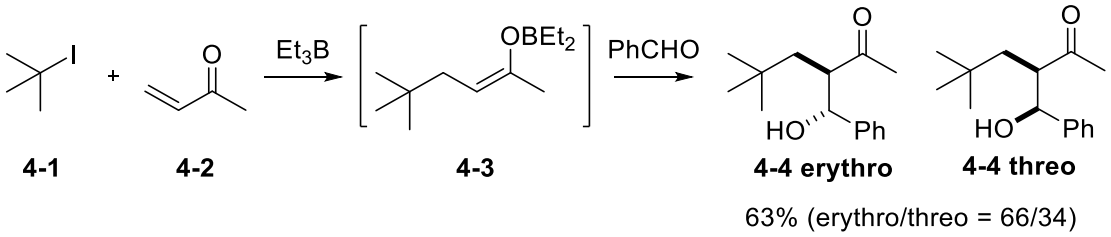
第四章　ラジカル−極性交差反応による連続反応への応用

4-1. O,Te-アセタールを用いた連続反応

4-1-1. 研究背景および作業仮説

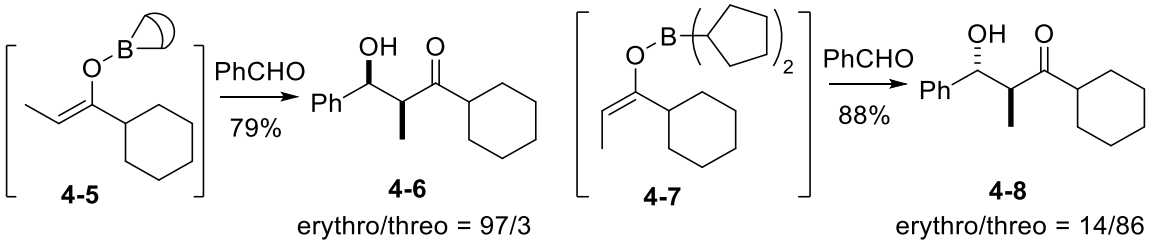
1988 年野崎らによりトリエチルボランを用いたラジカル−極性交差反応による三成分反応 [1]が報告された(Scheme 4-1)。本反応は、ヨウ化 *tert*-ブチル(**4-1**)に対しトリエチルボランを作用させて生じたラジカルがメチルビニルケトン(**4-2**)と反応し、ボロンエノラート **4-3** となる。さらに、本エノラート **4-3** とベンズアルデヒドが反応することで三成分付加体 **4-4** が得られる反応である。

Scheme 4-1. Three component reaction of alkyl iodide

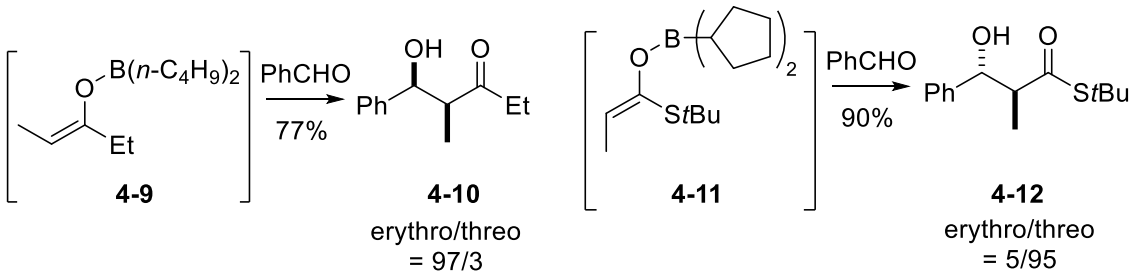


ボロンエノラートを経るアルドール反応における立体選択性に関しては、すでに 1979 年に正宗[2](Scheme 4-2)および Evans[3](Scheme 4-3)等により詳細が報告されている。どちらも、*E*-オレフィンおよび *Z*-オレフィンのボロンエノラートを調製し、ベンズアルデヒドと反応させることにより生成する付加体の選択性を調査している。いずれも *E* 体からはスレオ体が、*Z* 体からはエリスロ体が優先して得られている。

Scheme 4-2. Investigation of aldol reaction using boron enolate by Masamune

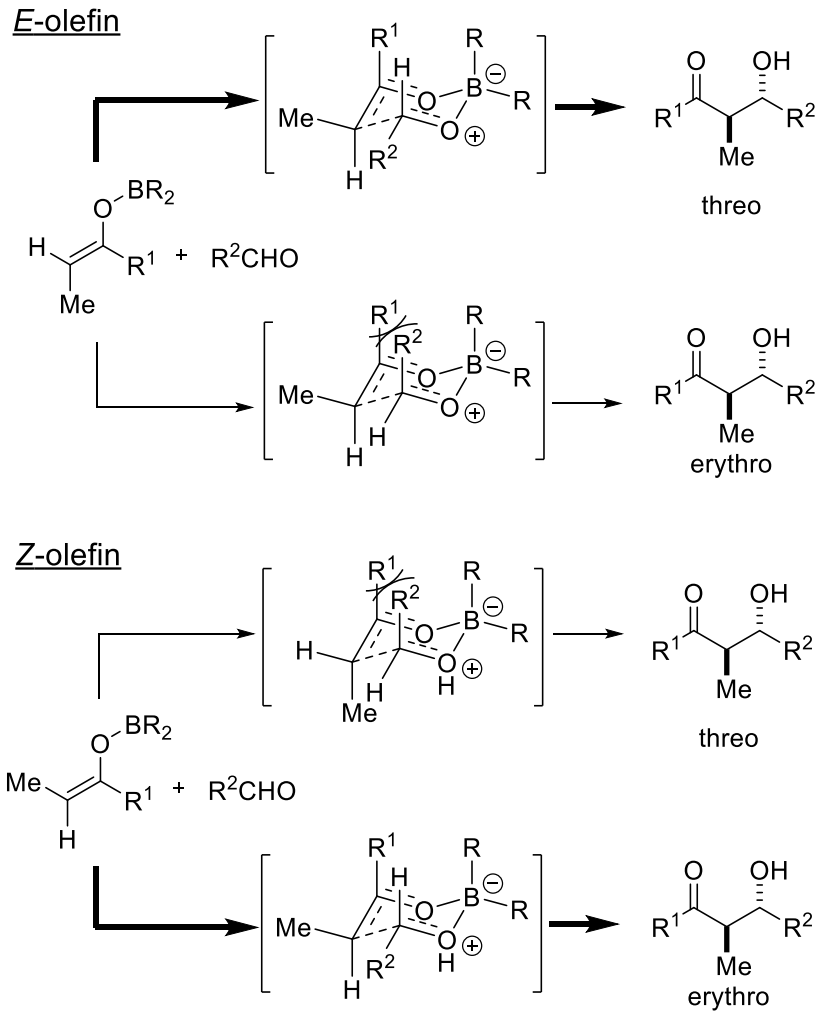


Scheme 4-3. Investigation of aldol reaction using boron enolate by Evans



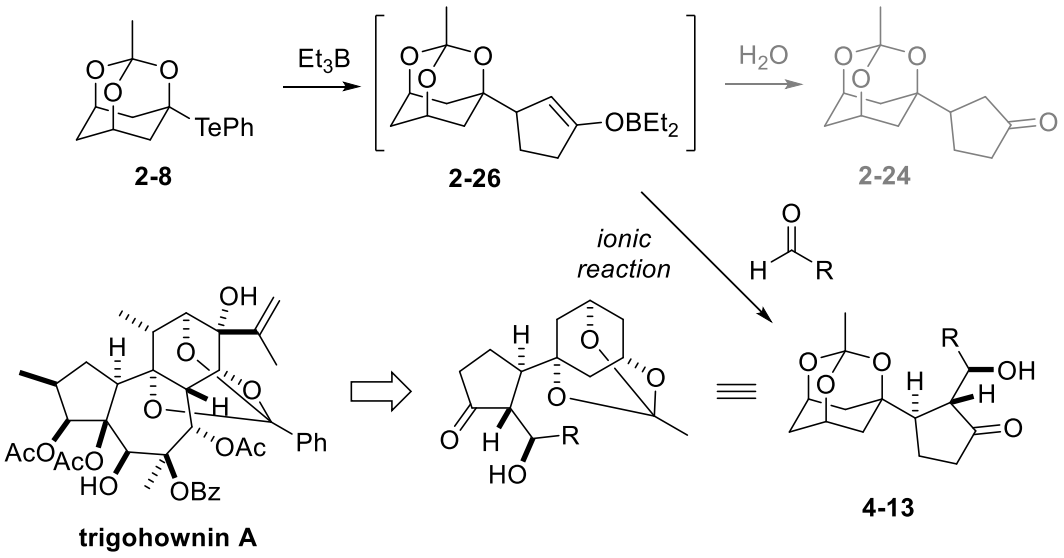
本選択性については、Evans らが同文献内において、ホウ素原子を介した 6 員環遷移状態を経由する反応機構を提唱している(Scheme 4-4)。

Scheme 4-4. Stereoselectivity of aldol reaction via boron enolate



以上の研究背景の基、著者は有機テルル化合物を本反応へ適用することで、trigohownin A[4]のような多官能基化された天然物の部分構造を効率的に合成できると考えた(Scheme 4-5)。合わせて、本反応の汎用性の確認のため、種々の置換基の導入を試みた。

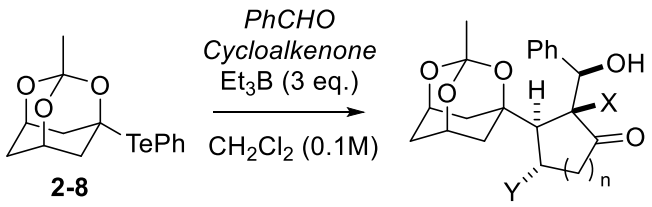
Scheme 4-5. Apply to radical-polar crossover reaction



4-1-2. ベンズアルデヒドを求電子剤とした三成分反応

最初に、求電子剤をベンズアルデヒドに固定し、種々のシクロアルケノンとの反応を行った(Table 4-1)。まず、シクロペンテノンを用い、2 章にて最適化した条件にて反応を行った。その結果、反応は速やかに進行し、目的の三成分付加体を得ることに成功した(Table 4-1、entry 1)。この際、所望の立体異性体である **4-14** およびベンジル位の立体化学が異なる異性体 **4-15** が約 9：1(それぞれ 80%および 9%)で得られた。これらの立体化学は、2 工程の誘導化の後 NOE 測定により決定した[5]。また、光学活性な TBS オキシ基が置換したシクロペンテノンを用いることで、**4-16** を選択的に得た(entry 2)。より高度に酸素官能基化された化合物の取得を志向し、2 位にアセトキシ基を有するシクロペンテノンを用い反応を実施した。その結果、trigohownin A と同様の第 3 級アルコール構造を有する **4-17** を 87%の収率で得ることが出来た(entry 3)。反応の汎用性の確認のため、シクロペンテノンの代わりにシクロヘキサノンを用い反応を行った結果、目的の三成分付加体 **4-18** は 27%と低収率であり、2 成分付加体 **2-19** が 65%の収率で得られた(entry 4)。

Table 4-1. Three-component reaction of **2-8**, PhCHO and cycloalkenone



2-8

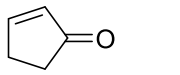
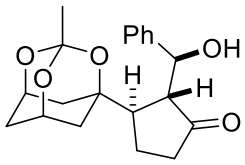
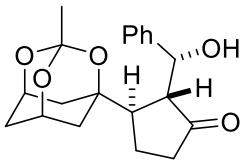
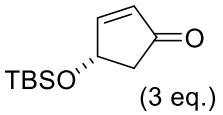
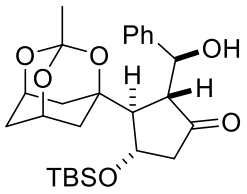
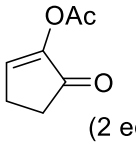
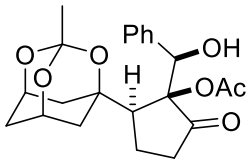
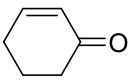
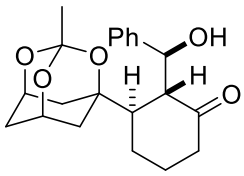
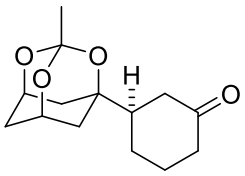
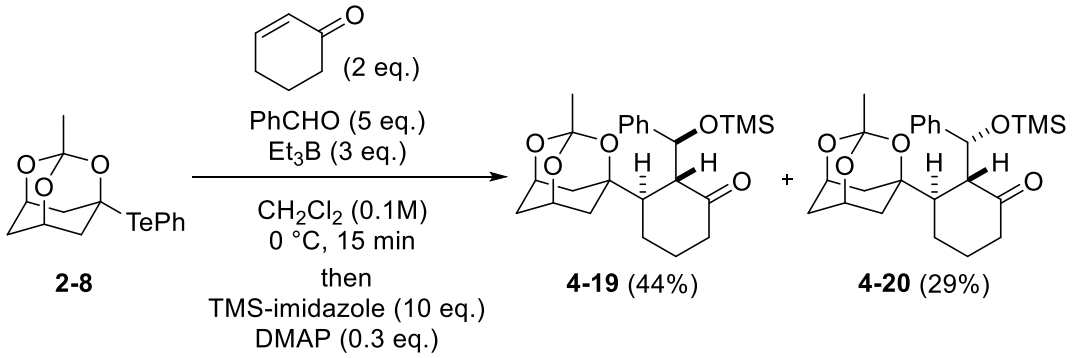
entry	cycloalkenone	PhCHO	result
1	 (3 eq.)	3 eq.	 **4-14** (80%) +  **4-15** (9%)
2	 (3 eq.)	3 eq.	 **4-16** (86%)
3	 (2 eq.)	5 eq.	 **4-17** (87%)
4	 (2 eq.)	5 eq.	 **4-18** (27%) +  **2-19** (65%)

Table 4-1 の entry 4 における低収率の原因を追究した。その結果、反応液の NMR 測定を行うと目的の **4-18** が主生成物として得られていることが明らかとなった。よって、本反応で得られる **4-18** はレトロアルドール反応が容易に進行し、後処理工程により分解していると考えられた。そこで、本反応で生じるアルコールを反応系中で保護することとした。保護に用いる試薬は、穏和な条件で TMS 化が可能な TMS イミダゾール [6]を選択した。検討の結果、選択性は乏しいものの、目的の三成分付加体 **4-19** および **4-20** を良好な収率で得ることが出来た(Scheme 4-6)。

Scheme 4-6 . TMS protection of the alcohol



4-1-3. 三成分反応の反応機構

4-14 の立体選択性は下記 Figure 4-1 に示した反応機構で進行していると考える。すなわち、トリエチルボランにより生じたトリオキサアダマンタンラジカルが、シクロペンテノンに付加し、ボロンエノラート **2-16** を形成する。本エノラート **2-16** に対し、ベンズアルデヒドは嵩高いトリオキサアダマンタン構造とは反対側の面から接近し、椅子型の 6 員環遷移状態 **4-21** を経由することで、優先的に **4-14** が生成すると考える。

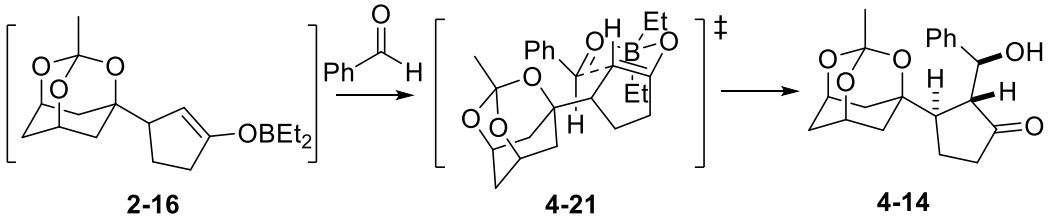
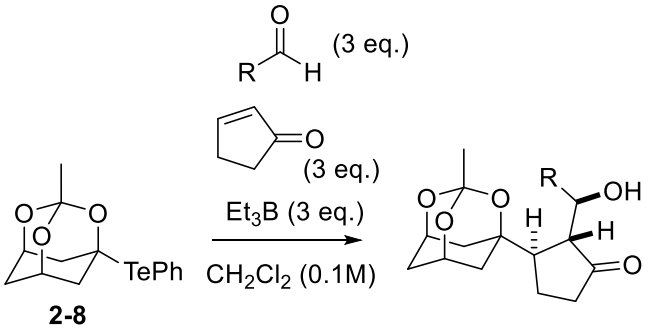


Figure 4-1. Rationale of the stereochemical outcome

4-1-4. アルキルアルデヒドをアルドール受容体とした三成分反応

ベンズアルデヒドに続き、アルキルアルデヒド類に対し反応を行った(Table 4-2)。まず、直鎖のアルキル基を有するアセトアルデヒドやヘプタナールを用い反応を行った。その結果、単一の立体かつ高い収率で目的の三成分付加体 **4-22** および **4-23** を得ることが出来た。環状構造を有するシクロヘキシルアルデヒドを反応させた場合も同様に、高収率かつ高立体選択的に付加体 **4-24** を得た。さらに、ピバルアルデヒドのような嵩高い置換基を有するアルデヒドにおいても、収率を損なうことなく反応は進行した。共役構造を有する *trans*-クロトンアルデヒドを用いると、クロトンアルデヒドがラジカル受容体として作用し、直接付加した化合物が得られるため、三成分付加体 **4-26** の収率は 41%に留まった。共役アルデヒドのラジカル受容体としての作用を低減させるため、他置換オレフィンである 3,3-ジメチルアクロレインを使用した。その結果、共役アルデヒドへの付加は完全に抑制され、三成分付加体 **4-27** を 76%の収率で得ることに成功した。

Table 4-2. Three-component reaction with alkyl aldehyde



2-8

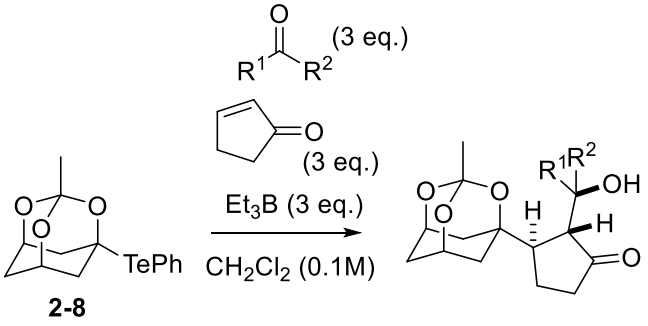
entry	R	result
1 [a]	R = Me	**4-22** : R = Me (86%)
2	R = *n*-C_6H_{13}	**4-23** : R = C_6H_{13} (77%)
3	R = cyclohexyl	**4-24** : R = cyclohexyl (99%)
4 [b]	R = *t*-Bu	**4-25** : R = *t*-Bu (74%)
5 [c]	R = (*E*)-CH=CHMe	**4-26** : R = (*E*)-CH=CHMe (41%)
6 [d]	R = $CH=CMe_2$	**4-27** : R = $CH=CMe_2$ (76%)

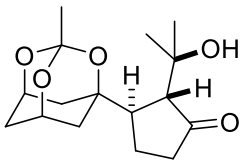
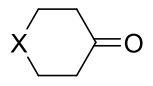
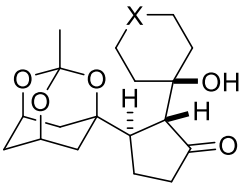
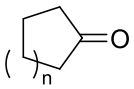
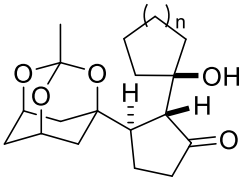
[a] aldehyde (10 eq.). [b] enone (2 eq.), aldehyde (5 eq.). [c] enone (2 eq.). [d] enone (2 eq.), aldehyde (5 eq.).

4-1-5. ケトン類をアルドール受容体とした三成分反応

アルデヒドをアルドール受容体とした三成分反応が進行したため、続いてケトン類の付加の検討を行った(Table 4-3)。最初にアセトンを使用し反応を行った結果、中程度の収率で目的の付加体 **4-28** を得ることが出来た。続いて環上ケトンとの反応を行った。シクロペンタノンを用いた場合、付加体 **4-29** は 22%と非常に低収率であった。本低収率の原因としては、Table 4-1 の entry 4 と同様、レトロアルドール反応が進行しているためであった。一方、シクロヘキサノンを用いた場合、付加体 **4-30** は 86%と高収率であった。本結果を踏まえ、*N*-Boc ピペリジノンやテトラヒドロピラノンとの付加反応を実施した。その結果、いずれも高い収率で三成分付加体を得ることができた。

Table 4-3. Three-component reaction with ketone



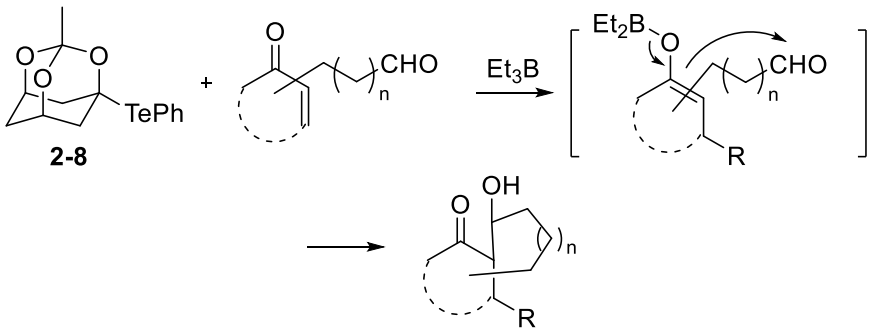
entry	ketone	result	entry	ketone	result
1[a]		 **4-28** (64%)	3	 X = NBoc, O	 X = NBoc : **4-31** (92%) X = O : **4-32** (90%)
2[a]	 n = 1, 2	 n = 1 : **4-29** (22%) n = 2 : **4-30** (86%)			

[a] enone (2 eq.), ketone (5 eq.).

4-1-6. ラジカル付加反応−分子内アルドール連続反応への応用

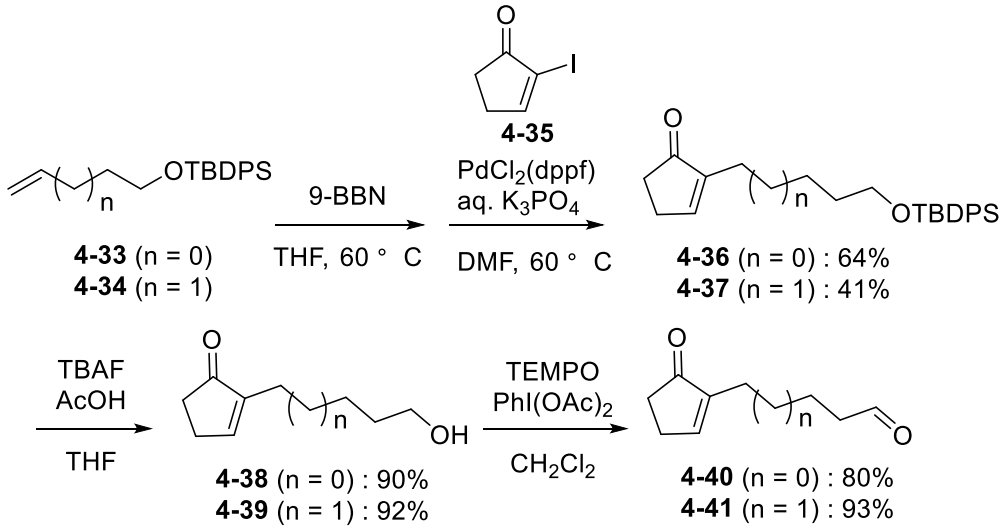
上記検討が効率よく進行したため、続いて分子内反応への応用を試みた。すなわち、ラジカル受容体であるエノンにリンカーを介してアルデヒドを導入することで、エノンへの付加後、ボロンエノラートが同一分子内のアルデヒドに対し付加すると考案した(Scheme 4-7)。

Scheme 4-7. Working hypothesis of intramolecular aldol reaction



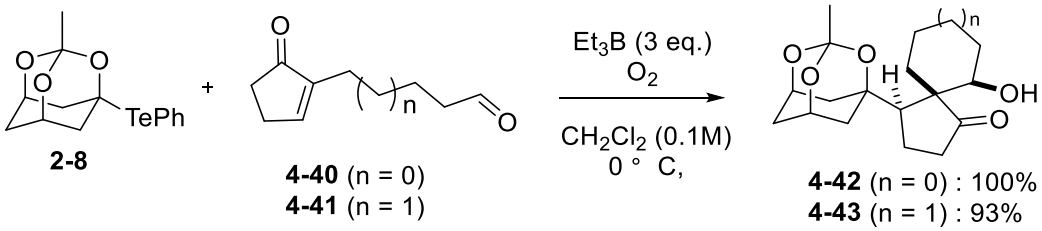
本反応に使用するアルデヒドとして、合成的および立体化学的観点からエノン α 位にペンタナールおよびヘキサナールを有する化合物 **4-40** 及び **4-41** を設計した。まず、文献既知のシリルアルコール **4-33**[7]又は **4-34**[8]と 2-ヨードシクロペンテノン[9](**4-35**)との鈴木カップリングにより、2-置換シクロペンテノン **4-36** および **4-37** へと導いた。続く脱シリル化および酸化により、アルデヒド **4-40** および **4-41** を得た。

Scheme 4-8. Preparation of aldehyde **4-40** and **4-41**



アルデヒド **4-40** および **4-41** を用い、オキサアダマンタンのラジカル付加反応に続く分子内アルドール反応の連続反応の検討を行った。その結果、反応は速やかに進行し、アルドール付加体 **4-42** および **4-43** を高収率および高立体選択的に得ることに成功した(Scheme 4-9)。

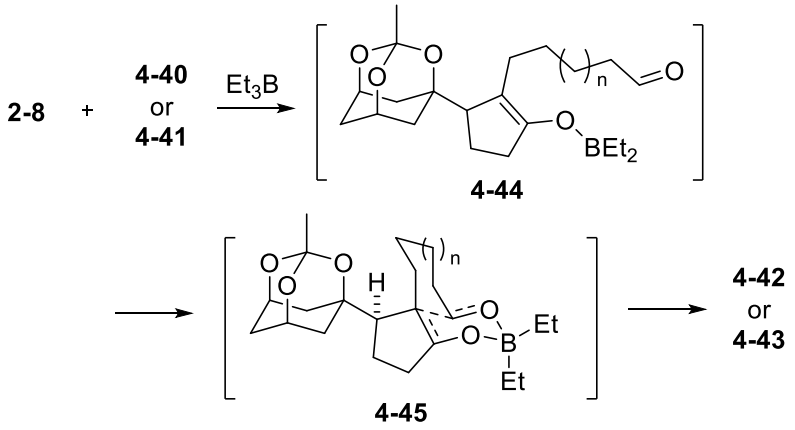
Scheme 4-9. Intramolecular radical polar crossover reaction



4-1-7. ラジカル付加反応-分子内アルドール連続反応の反応機構

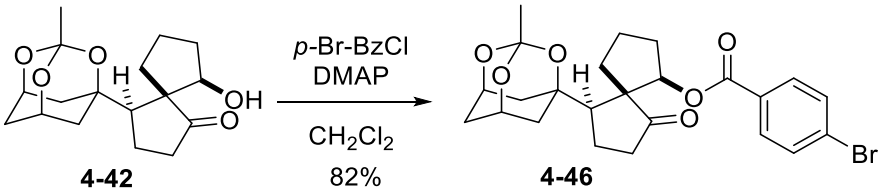
本反応の立体選択性について、以下のように考察した(Scheme 4-10)。すなわち、**2-8** から発生したラジカルが **4-40** もしくは **4-41** へと付加することで、ボロンエノラート **4-44** を生じる。**4-44** はホウ素を介した 6 員環椅子型遷移状態 **4-45** を経ることでそれぞれ **4-42** および **4-43** が得られると考えた。

Scheme 4-10. Proposal mechanism of intramolecular radical polar crossover reaction



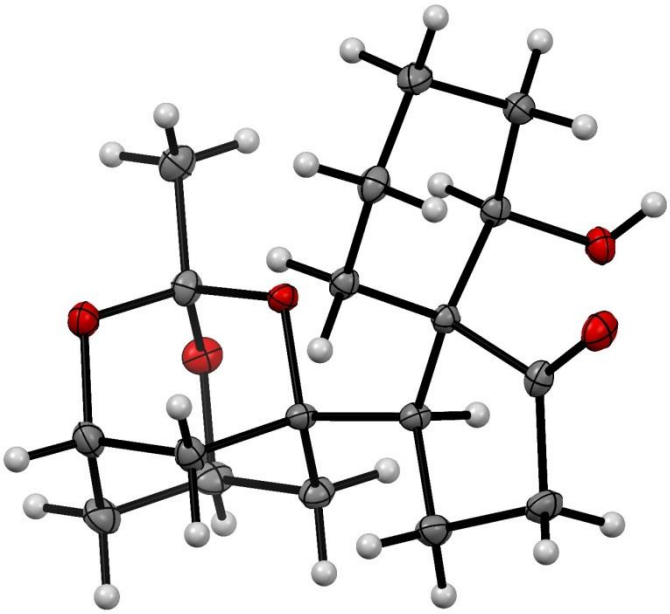
本考察の確認のため、X 線結晶構造の取得を検討した。最初に結晶性の誘導体への変換を指向し、油状物質である **4-42** を *p*-ブロモ安息香酸エステル **4-46** へと変換した(Scheme 4-11)。

Scheme 4-11. Preparation of crystalline derivative

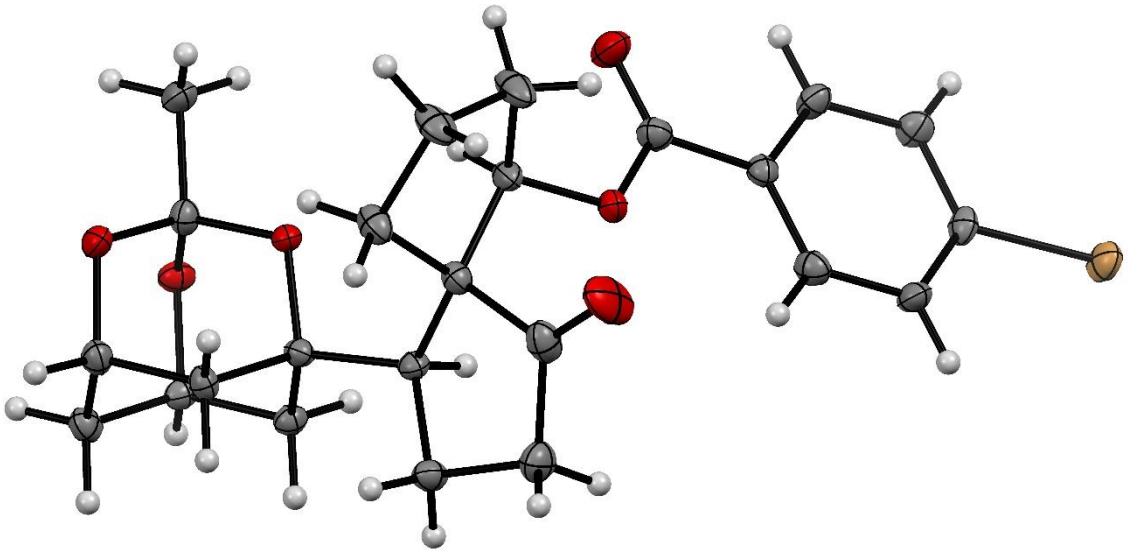


狙い通り **4-46** は結晶として得られた。また、**4-43** が結晶として得られたことから、**4-43** 及び **4-46** の X 線結晶構造の取得を試みた。

いずれも X 線結晶構造の取得に成功し、**4-43** 及び **4-46** は考察通りの立体を有していたことが明らかになった(Figure 4-2)。



The structure of **4-43** (CCDC: 1453970)



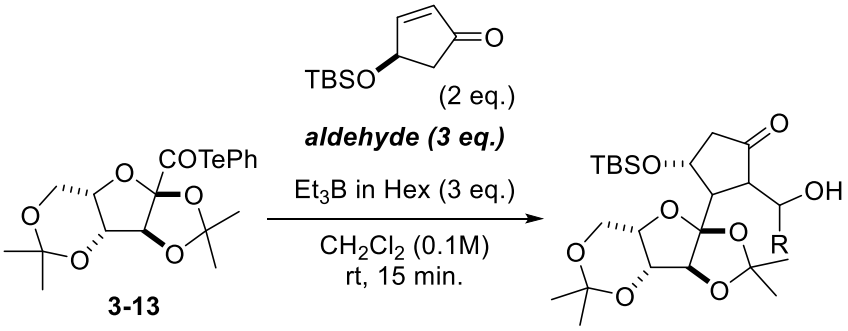
The structure of **4-46** (CCDC: 1453968)

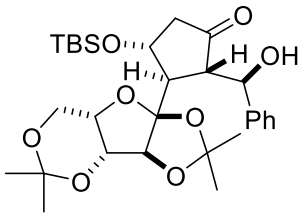
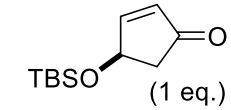
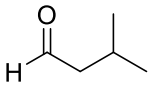
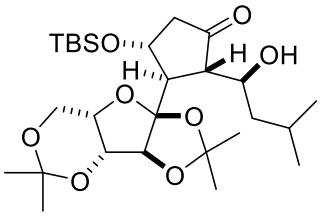
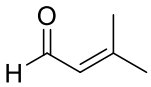
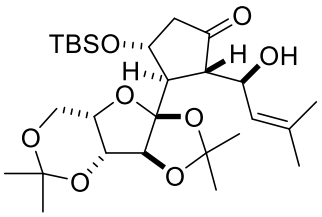
Figure 4-2. X-ray crystallographic analyses of **4-43** and **4-46**

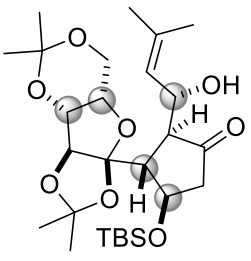
4-2. アシルテルリドを用いた連続反応

O,Te-アセタール **2-8** を基質として行ってきた三成分反応の汎用性の確認のため、3 章にて合成した 2-ケト-L-グロン酸のアシルテルリド **3-13** を基質として用い反応を実施した(Table 4-4)。最初に、光学活性エノンを 2 当量、ベンズアルデヒドを 3 当量用い反応を行った。反応は速やかに進行し、三成分付加体 **4-47** を高収率かつ単一の立体で得ることに成功した(entry 1)。また、本反応はエノンおよびアルデヒドを 1 当量としても問題なく進行し、64%の収率で三成分付加体 **4-47** を与えた。本反応に用いるアルデヒドは、芳香族だけでなく、脂肪族や共役アルデヒドを用いた場合においても速やかに進行し、三成分付加体 **4-48** および **4-49** をそれぞれ 85%および 79%で得ることができた。**4-49** は、超官能基密集型天然物 sororianolide B[10]の部分構造と良い一致を示したことから、本反応は複雑に官能基化された天然物の骨格を一挙に構築できる有用な手法であるといえる(Figure 4-3)。

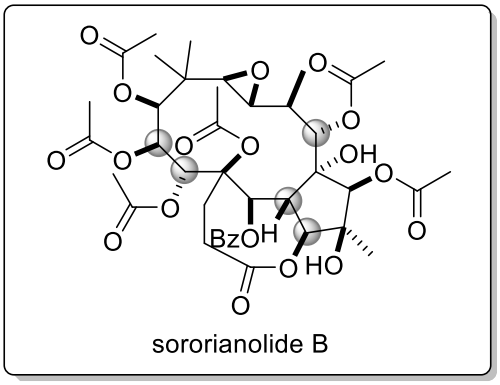
Table 4-4. Three component reaction with acyl telluride



entry	aldehyde	result	yield
1	benzaldehyde		**4-47** (88%)
2	 (1 eq.) benzaldehyde (1 eq.)		**4-47** (64%)
3			**4-48** (85%)
4			**4-49** (79%)



4-49



sororianolide B

Figure 4-3. Structural agreement between **4-49** and sororianolide B

4-3. 小括

以上筆者は、O,Te-アセタール **2-8** をラジカル−極性交差反応へ応用した三成分反応により、複雑な構造を有する化合物へ一挙に導くことに成功した。また、分子内ラジカル−極性交差反応により、立体選択的にスピロ構造が得られることを明らかとした。さらに、アシルテルリド **3-13** を用いた脱カルボニルを経るラジカル−極性交差反応が進行することを見出し、複雑に官能基化された天然物の骨格を一挙に構築することに成功した。

4-4. 参考文献および脚注

(1) Nozaki, K.; Oshima, K.; Utimoto, K. *Tetrahedron Lett.*, **1988**, *29*, 1041.

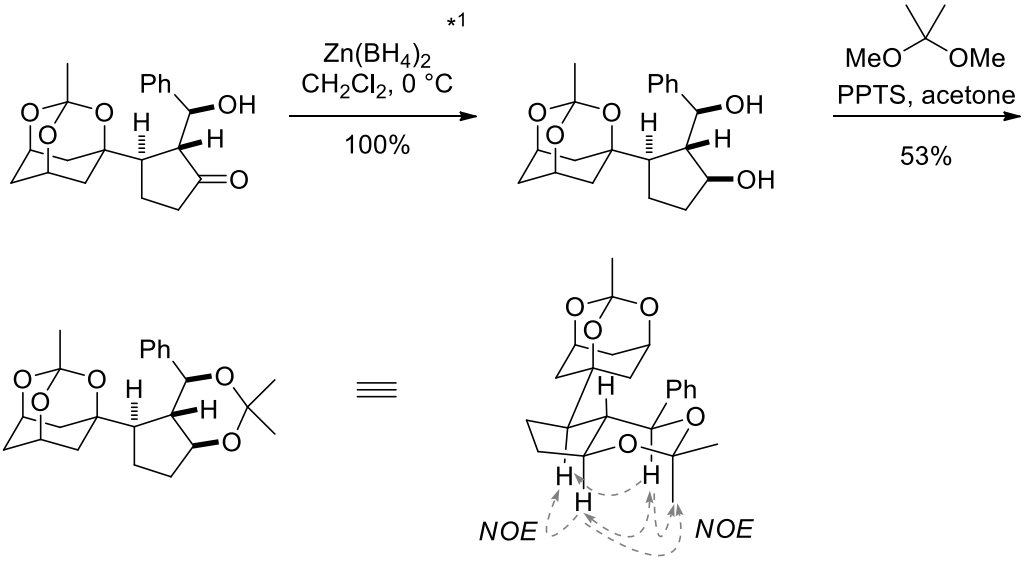
(2) Horn, D. E.; Masamune, S. *Tetrahedron Lett*., **1979**, *20*, 2229.

(3) Evans, D. A.; Vogel, E.; Nelson, J. V. *J. Am. Chem. Soc*., **1979**, *101*, 6120.

(4) Dong, S-H.; Zhang, C. R.; Xu, C-H.; Ding, J.; Yue, J-M. *J. Nat. Prod.*, **2011**, *74*, 1255.

(5) 各化合物の立体は、$Zn(BH_4)_2$ による立体選択的な還元*[1] の後、生じた 1,3-ジオールをアセタール保護し、NOE 測定を行うことで決定した。その他の基質に関しても、同様の方法で立体の検定を行った。詳細は実験項に記載した。

*[1] T. Nakata and T. Oishi, *Tetrahedron Lett.*, 1980, **21**, 1641



(6) Morisaki, M.; Bannal, k.; Ikekawa, N. *Chem. Pharm. Bull*., **1976**, *24*, 1948.

(7) Nemoto, H.; Shiraki, M.; Fukumoto, K. *Tetrahedron Lett*., **1995**, *36*, 8799.

(8) Cook, C.; Guinchara, X.; Liron, F.; Roulland, E. *Org. Lett.*, **2010**, *12*, 744.

(9) Elsa, D.; Pakorn, B.; Edward, M. N. *Synth. Commun*., **1997**, *27*, 2497.

(10) Huang, Y.; Aisa, H. A. *Helv. Chim. Acta*., **2010**, *93*, 1156.

第五章　総論

有機カルコゲン化合物は有用なラジカル前駆体として知られている。中でも筆者は、有機テルル化合物に着目し、これらをラジカル前駆体とした新規反応の開発に着手した。

第二章では O,Te-アセタール **2-8** の合成法の確立、反応条件の最適化および **2-8** から生じる α-アルコキシ橋頭位ラジカル **2-2** の反応性調査を実施した。また、すでに当研究室にて確立されている O,Se-アセタール **2-1** をラジカル前駆体として用いた付加反応との比較を行い、その反応性の違いと本反応の優位性を明らかにした(Figure 5-1)。

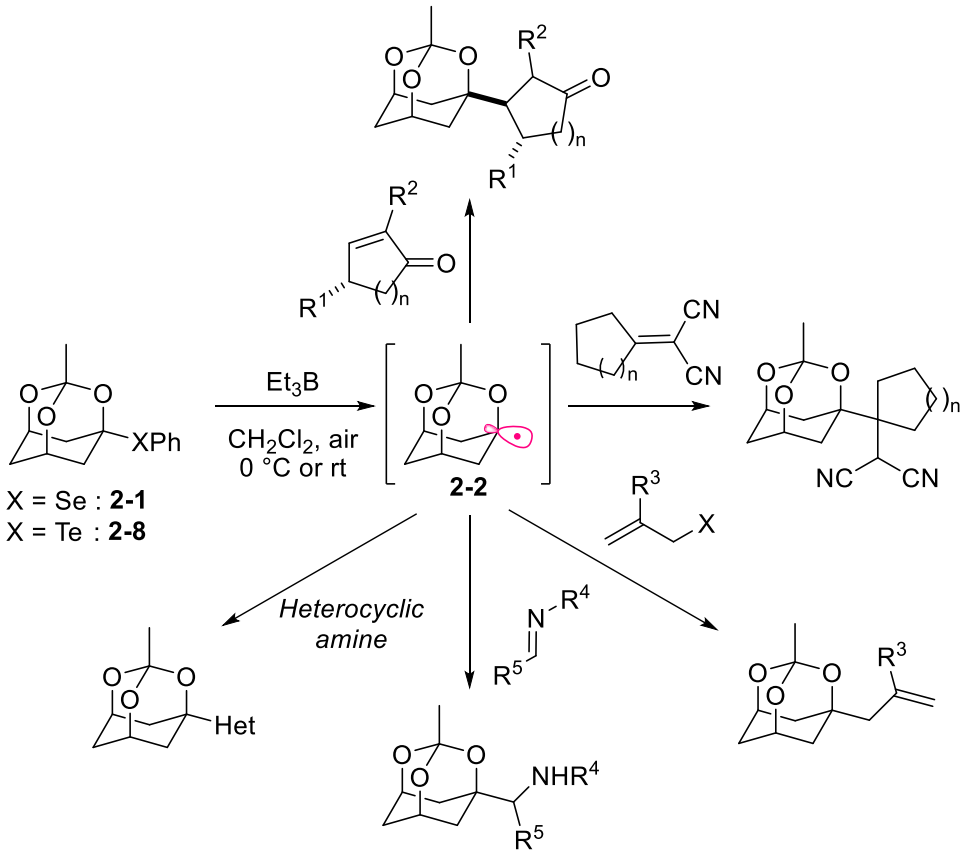


Figure 5-1. Reactivity of O,Te-acetal

第三章では、アシルテルリドの α 位に酸素原子を有する α-アルコキシアシルテルリドをラジカル前駆体として用いることで、アシルラジカルからの脱カルボニルが穏和な条件かつ速やかに進行し α-アルコキシ炭素ラジカルが生じること、本系内にラジカル受容体を共存させることで、種々の付加体が高収率で得られることを明らかとした(Figure 5-2)。

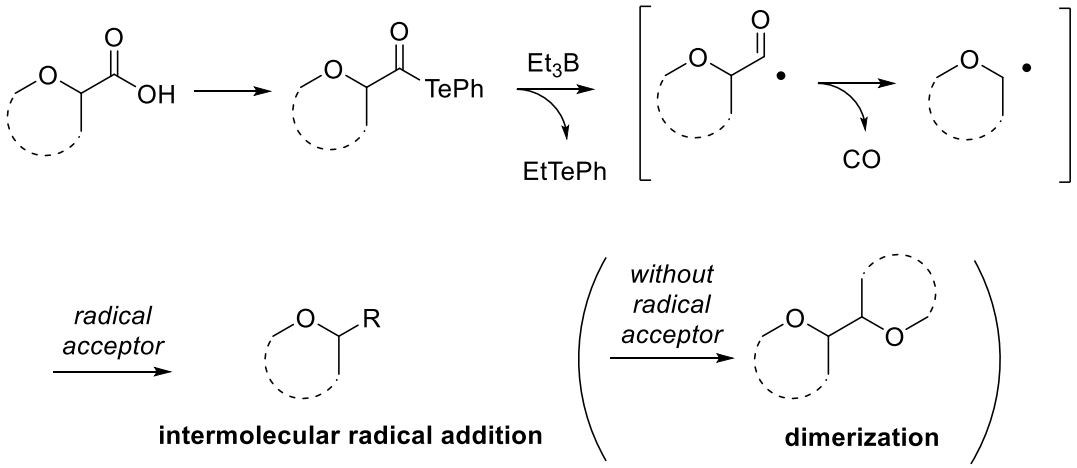


Figure 5-2. C-C bond formation reaction via radical decarbonylation of acyl telluride

第四章では、O,Te-アセタール **2-8** やアシルテルリド **3-13** 等の有機テルル化合物をラジカル一極性交差反応による連続反応へ応用することで、高収率かつ高立体選択的に、複雑な構造を有する誘導体へと変換することに成功した(Figure 5-3, Figure 5-4)。

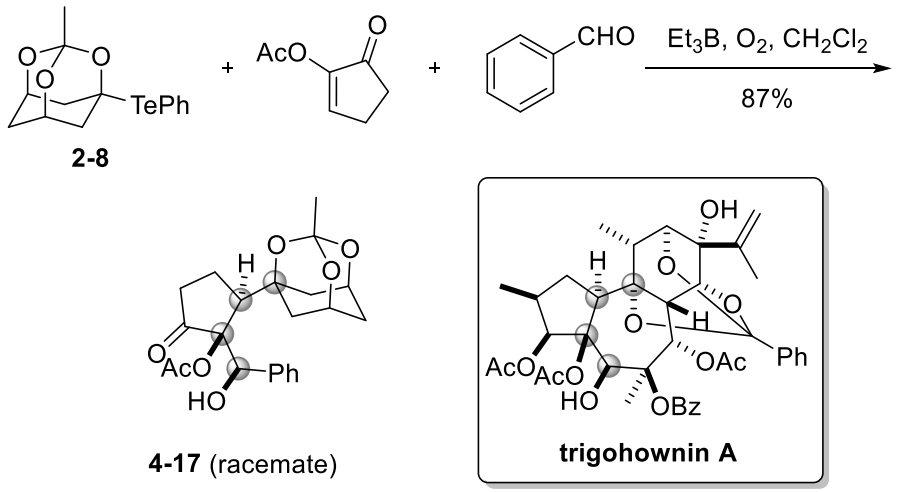


Figure 5-3. Three-component couplings utilizing O,Te-acetal **2-8**

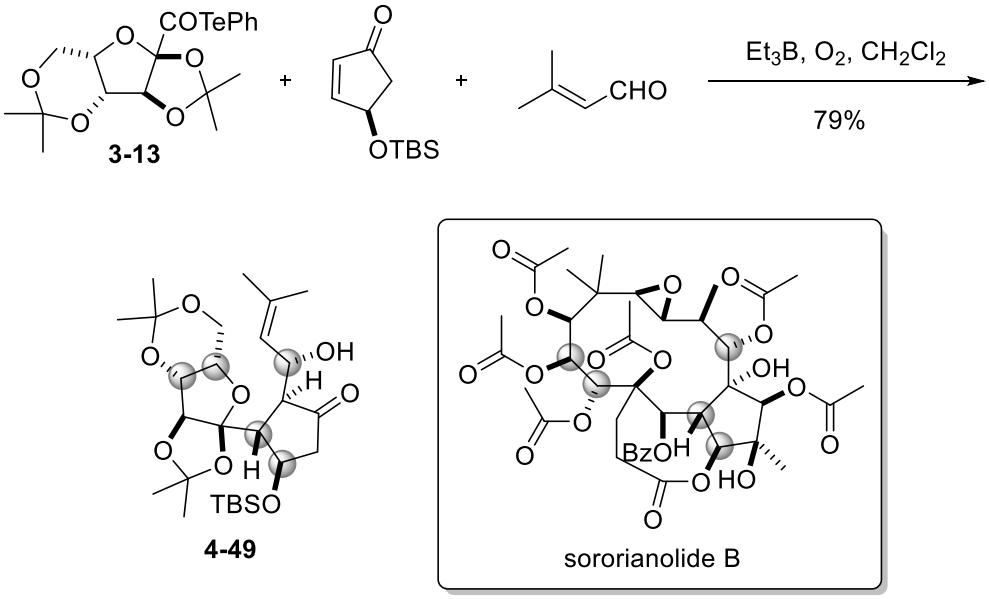


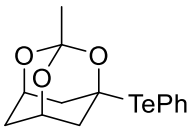
Figure 5-4. Three-component couplings utilizing Acyl telluride **3-13**

以上、本反応は、良好な汎用性と穏和で簡便な反応条件を併せ持ち、複雑な構造を有する骨格の構築にも応用できることから、天然物合成への応用が期待できる。

第六章　実験項

General method: All reactions sensitive to air or moisture were carried out under argon or nitrogen atmosphere in dry solvents under anhydrous conditions, unless otherwise noted. THF, CH2Cl2, toluene, DMF and Et2O were purified by Glass Contour solvent dispensing system (Nikko Hansen & Co., Ltd.). All other reagents were used as supplied. Analytical thin-layer chromatography (TLC) was performed using E. Merck Silica gel 60 F_{254} pre-coated plates (0.25 mm). Column chromatography was performed using 100-210 μm Silica Gel 60 N (Kanto Chemical Co., Inc.), and for flash column chromatography 40-100 μm Silica Gel 60N (Kanto Chemical Co., Inc.) was used. Optical rotations were measured on JASCO DIP-1000 Digital Polarimeter at room temperature using the sodium D line. Melting points were measured on Yanaco MP-J3 micro melting point apparatus, and were uncorrected. Infrared (IR) spectra were recorded on JASCO FT/IR-4100 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on JEOL JNM-ECX-500 (500 MHz), JNM-ECA-500 (500 MHz), or JNM-ECS-400 (400 MHz) spectrometer. Chemical shifts are reported in ppm on the δ scale relative to $CHCl_3$ (δ = 7.26 for ^{1}H NMR), $CDCl_3$ (δ = 77.0 for ^{13}C NMR), C_6D_5H (δ = 7.16 for ^{1}H NMR), and C_6D_6 (δ = 128.0 for ^{13}C NMR). Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad peak. High resolution mass spectra were measured on JEOL JMS-T100LP instrument or Bruker BioTOF-Q instrument (ESI-TOF or DART-TOF).

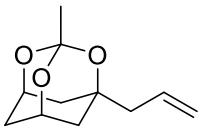
6-1. 第二章の実験項



2-8

Telluride 2-8 [DK-3-006]. A mixture of carboxylic acid **2-9** (100 mg, 0.50 mmol), 1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (144 mg, 0.75 mmol), and 2-Mercaptopyridine *N*-Oxide (95 mg, 0.75 mmol) in CH_2Cl_2 (5.0 mL) were stirred for 1 h at room temperature in the flask was lapped with aluminum foil, and then $(PhTe)_2$ (409 mg, 1.00 mmol) was added to the mixture. The mixture was irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 1 h. The resultant mixture was purified by flash column chromatography on silica gel (30 g, hexane/EtOAc 10:1 to 4:1) to afford O,Te-acetal **2-8** (149 mg, 0.41 mmol) in 83% yield: yellow oil; IR (film) 3051, 3007, 2951, 2849, 1726, 1572, 1472, 1433, 1393, 1349, 1296, 1227, 1141, 1122, 1047, 1017 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 0.87 (1H, dt, *J* = 12.8, 1.8 Hz, CHOC$H_{ax}$$H_{eq}$CHO), 1.73-1.78 (5H, m, C-CH_3, COC$H_{ax}$$H_{eq}$CHO x2), 2.24 (1H, m, CHOC$H_{ax}$$H_{eq}$CHO), 2.80-2.87 (2H, m, COC$H_{ax}$$H_{eq}$CHO x2), 3.87 (1H, m, CH_2C*H*OCH_2 x2), 7.06-7.12 (2H, m, aromatic), 7.12-7.18 (1H, m, aromatic), 8.06 (2H, dd, *J* = 8.2, 1.4 Hz, aromatic); ^{13}C NMR (100 MHz, C_6D_6) δ 26.6, 32.0, 42.3, 68.3, 69.3, 111.9, 112.7, 128.8, 129.4, 141.8; HRMS (ESI-TOF) calcd for $C_{14}H_{17}O_3Te$ $[M+H]^+$ 363.0234, found 363.0249.

Synthesis of olefin **2-27** from O,Te-acetal **2-8** and allyl chloride



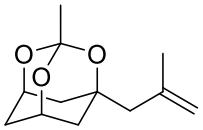
2-27

***General procedure A*: Olefine 2-27 [DK-5-030]**. Et_3B (1.03 mol/L hexane solution, 0.45 mL, 0.47 mmol) was added to a solution of O,Te-acetal **2-8** (33.7 mg, 0.0936 mmol), allyl chloride (38 μL, 0.47 mmol) in CH_2Cl_2 (0.49 mL) at 0 °C. After being stirried for 15 min, the resultant mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5:1) to afford **2-27** (12.0 mg, 0.0611 mmol) in 65% yield: colorless oil; IR (film) 3077, 3008, 2953, 2929, 2850, 1733, 1641, 1445, 1395, 1367, 1325, 1297, 1262, 1229, 1206, 1160, 1127 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.44 (3H, s, CCH_3), 1.54 (1H, d, *J* = 13.3 Hz, CHOC$H_{ax}$$H_{eq}$CHO), 1.59 (2H, d, *J* = 13.3 Hz, COC$H_{ax}$$H_{eq}$CHO x2), 2.16-2.25 (4H, m, COC$H_{ax}$$H_{eq}$CHO x2, C$H_2$CH=$CH_2$), 2.51 (1H, m, CHOC$H_{ax}$$H_{eq}$CHO), 4.40 (2H, m, CH_2C*H*OCH_2 x2), 5.07-5.16 (2H, m, CH_2CH=CH_2), 5.81 (1H, ddt, *J* = 17.8, 10.5, 7.3 Hz, CH_2C*H*=CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.2, 32.3, 36.4, 46.3, 68.1, 72.5, 110.3, 118.8, 131.8; HRMS (ESI-TOF) calcd for $C_{11}H_{17}O_3$ $[M+H]^+$ 197.1172, found 197.1180.

Synthesis of olefin **2-27** from O,Te-acetal **2-8** and allyl bromide

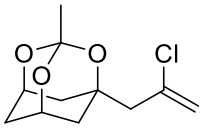
Olefine 2-27 [DK-3-044, DK-5-031]. According to the general procedure A, **2-27** (10.5 mg, 0.0535 mmol) was

synthesized from O,Te-acetal **2-8** (34.0 mg, 0.0945 mmol) and allyl bromide (41 mL, 0.47 mmol) in 57% yield by using Et_3B (1.03 M hexane solution, 0.46 mL, 0.47 mmol) in CH_2Cl_2 (0.94 mL). The reaction mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5/1).



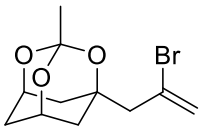
2-28

Olefine 2-28 [DK-3-047, DK-5-036]. According to the general procedure A, **2-28** (13.1 mg, 0.0623 mmol) was synthesized from O,Te-acetal **2-8** (36.7 mg, 0.102 mmol) and 3-bromo-2-methylprop-1-ene (51 µL, 0.51 mmol) in 61% yield by using Et_3B (1.03 M hexane solution, 0.50 mL, 0.51 mmol) in CH_2Cl_2 (1.0 mL). The reaction mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 10/1): colorless oil; IR (film) 3073, 3007, 2951, 2851, 1645, 1445, 1394, 1324, 1297, 1125 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, s, CCH_3), 1.55 (1H, dt, J = 13.3, 1.8 Hz, CHOC$H_{ax}H_{eq}$CHO), 1.56 (2H, d, J = 13.3 Hz, COC$H_{ax}H_{eq}$CHO x2), 1.81 (3H, s, $CH_2C(CH_3)$=CH_2), 2.18 (2H, s, CH_2C(CH_3)=CH_2), 2.25 (2H, m, $COCH_{ax}H_{eq}$CHO x2), 2.51 (1H, dtt, J = 12.8, 4.6, 2.3 Hz, $CHOCH_{ax}H_{eq}$CHO), 4.40 (2H, m, CH_2CHOCH_2 x2), 4.70 (1H, m, $CH_2C(CH_3)$=CH_AH_B), 4.89 (1H, m, $CH_2C(CH_3)$=CH_AH_B); ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.6, 26.2, 32.4, 36.6 (2C), 49.7, 68.2 (2C), 72.8, 110.2, 115.0, 140.9; HRMS (DART-TOF) calcd for $C_{12}H_{19}O_3$ $[M+H]^+$ 211.1329, found 211.1338.



2-29

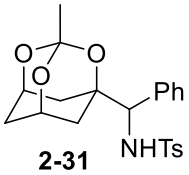
Olefine 2-29 [DK-3-046, DK-5-035]. According to the general procedure A, **2-29** (18.5 mg, 0.0802 mmol) was synthesized from O,Te-acetal **2-8** (36.6 mg, 0.102 mmol) and 2,3-dichloroprop-1-ene (47 µL, 0.51 mmol) in 79% yield by using Et_3B (1.03 M hexane solution, 0.49 mL, 0.51 mmol) in CH_2Cl_2 (1.0 mL). The reaction mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5/1): colorless oil; IR (film) 3009, 2971, 2947, 2930, 2851, 1632, 1444, 1395, 1325, 1298, 1161 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, s, CCH_3), 1.57 (1H, dt, J = 12.8, 1.8 Hz, CHOC$H_{ax}H_{eq}$CHO), 1.80 (2H, d, J = 13.3 Hz, COC$H_{ax}H_{eq}$CHO x2), 2.27 (2H, m, $COCH_{ax}H_{eq}$CHO x2), 2.47-2.55 (3H, m, $CHOCH_{ax}H_{eq}$CHO, CH_2C(Cl)=CH_2), 4.42 (2H, m, CH_2CHOCH_2 x2), 5.25 (1H, s, $CH_2C(Cl)$=CH_AH_B), 5.35 (1H, d, J = 0.9 Hz, $CH_2C(Cl)$=CH_AH_B); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.1, 32.1, 36.4 (2C), 51.0, 68.1 (2C), 72.3, 110.4, 117.2, 135.8; HRMS (DART-TOF) calcd for $C_{11}H_{16}ClO_3$ $[M+H]^+$ 231.0782, found 231.0785.



2-30

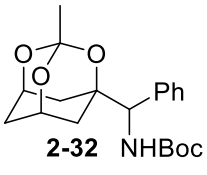
Olefine 2-30 [DK-3-011, DK-5-038]. According to the general procedure A, **2-30** (20.0 mg, 0.0727 mmol) was

synthesized from O,Te-acetal **2-8** (37.1 mg, 0.103 mmol) and 2-bromo-3-chloroprop-1-ene (49 μL, 0.52 mmol) in 71% yield by using Et_3B (1.03 M hexane solution, 0.50 mL, 0.52 mmol) in CH_2Cl_2 (1.0 mL). The reaction mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 10/1): colorless oil; IR (film) 3007, 2953, 2929, 2846, 2359, 1626, 1394, 1324, 1298, 1160, 1147, 1126 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, s, CC*H*$_3$), 1.57 (1H, dt, *J* = 13.3, 1.8 Hz, CHOC*H*$_{ax}$H$_{eq}$CHO), 1.81 (2H, d, *J* = 12.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO x2), 2.28 (2H, dt, *J* = 13.3, 2.3 Hz, COCH$_{ax}$*H*$_{eq}$CHO x2), 2.52 (1H, dtt, *J* = 13.3, 4.6, 2.3 Hz, CHOCH$_{ax}$*H*$_{eq}$CHO), 2.67 (2H, s, C*H*$_2$C(Br)=CH$_2$), 4.42 (2H, m, CH$_2$C*H*OCH$_2$ x2), 5.62 (1H, d, *J* = 1.4 Hz, CH$_2$C(Br)=C*H*$_A$H$_B$), 5.71 (1H, d, *J* = 0.9 Hz, CH$_2$C(Br)=CH$_A$*H*$_B$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.1, 32.1, 36.4 (2C), 52.8, 68.1 (2C), 72.5, 110.4, 122.0, 125.5; HRMS (DART-TOF) calcd for $C_{11}H_{16}BrO_3$ $[M+H]^+$ 275.0277, 277.0257, found 275.0286, 277.0267.



2-31

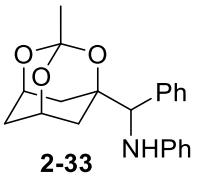
***General procedure B*: Sulfonamide 2-31 [DK-1-156, DK-5-062].** Et_3B (1.03 M hexane solution, 0.30 mL, 0.31 mmol) was added to a solution of O,Te-acetal **2-8** (37.0 mg, 0.103 mmol) and *N*-benzylidene-*p*-toluenesulfonamide (80.0 mg, 0.308 mmol) in CH_2Cl_2 (1.0 mL) at room temperature. The reaction mixture was stirring for 15 min, and then saturated aqueous $NaHCO_3$ (10 mL) was added. The resultant solution was extracted with EtOAc (5 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 3/1) to afford **2-31** (32.5 mg, 0.0782 mmol) in 76% yield: colorless solid; m.p. 177-179 °C; IR (film) 3279, 2954, 1451, 1397, 1324, 1299, 1160, 1128, 1091, 1059 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.35 (1H, dd, *J* = 13.3, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.41 (3H, s, CC*H*$_3$), 1.42-1.48 (2H, m, COC*H*$_{ax}$H$_{eq}$CHO, CHOC*H*$_{ax}$H$_{eq}$CHO), 1.93 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 2.32 (3H, s, ArC*H*$_3$), 2.44 (1H, dtt, *J* = 13.3, 3.6, 1.8 Hz, CHOCH$_{ax}$*H*$_{eq}$CHO), 2.58 (1H, dtt, *J* = 13.3, 2.3, 2.3 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 4.06 (1H, d, *J* = 6.9 Hz, PhC*H*), 4.27 (1H, m, CH$_2$C*H*OCH$_2$), 4.39 (1H, m, CH$_2$C*H*OCH$_2$), 5.53 (1H, d, *J* = 6.4 Hz, N*H*), 7.00-7.06 (4H, m, aromatic), 7.08-7.19 (3H, m, aromatic), 7.39-7.44 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.4, 25.9, 32.1, 33.0, 34.6, 64.9, 67.6, 67.9, 74.4, 110.6, 127.0 (2C), 127.7, 127.9 (2C), 128.6 (2C), 129.1 (2C), 135.4, 137.1, 142.9; HRMS (ESI-TOF) calcd for $C_{22}H_{25}NNaO_5S$ $[M+Na]^+$ 438.1346, found 438.1330.



2-32

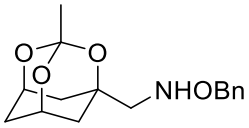
Carbamate 2-32 [DK-1-167, DK-5-061]. According to the general procedure B, **2-32** (35.5 mg, 0.0982 mmol) was synthesized from O,Te-acetal **2-8** (38.3 mg, 0.106 mmol) and *tert*-butyl(phenylmethylene)carbamate (65.0 mg, 0.317 mmol) in 93% yield by using Et_3B (1.03 M hexane solution, 0.31 mL, 0.32 mmol) in CH_2Cl_2 (1.0 mL). The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 3/1): colorless oil; IR (film) 3451,

3354, 2969, 1710, 1495, 1394, 1367, 1324, 1297, 1246, 1167, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34-1.48 (13H, m, *t*-Bu, CCH_3, COC$H_{ax}H_{eq}$CHO), 1.50-1.65 (2H, m, COC$H_{ax}H_{eq}$CHO, CHOC$H_{ax}H_{eq}$CHO), 1.85 (1H, d, *J* = 12.4 Hz, COCH$_{ax}H_{eq}$CHO), 2.48 (1H, dtt, *J* = 13.3, 3.6, 1.8 Hz, CHOCH$_{ax}H_{eq}$CHO), 2.60 (1H, dtt, *J* = 13.3, 2.3, 2.3 Hz, COCH$_{ax}H_{eq}$CHO), 4.27 (1H, m, CH_2CHOCH_2), 4.34-4.49 (2H, m, CH_2CHOCH_2, PhC*H*), 5.53 (1H, br d, *J* = 5.9 Hz, N*H*), 7.24-7.34 (5H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$, 40 °C) δ 26.0, 28.2, 28.3 (3C), 32.3, 34.1, 34.7, 67.7, 68.1, 74.3, 79.6, 110.6, 127.5, 128.1 (2C), 128.5 (2C), 138.4, 155.7; HRMS (ESI-TOF) calcd for $C_{20}H_{27}NNaO_5$ $[M+Na]^+$ 384.1781, found 384.1783.



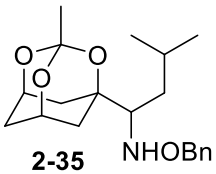
2-33

Aniline 2-33 [DK-3-081, DK-5-052]. According to the general procedure B, **2-33** (21.0 mg, 0.0622 mmol) was synthesized from O,Te-acetal **2-8** (37.4 mg, 0.104 mmol) and *N*-benzylideneaniline[1)] (94.0 mg, 0.519 mmol) in 60% yield by using Et_3B (1.03 M hexane solution, 0.51 mL, 0.52 mmol) in CH_2Cl_2 (1.0 mL). The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5/1): colorless oil; IR (film) 3390, 3053, 3008, 2956, 2931, 2850, 1602, 1503, 1395, 1320, 1298, 1146, 1127 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.39 (1H, dd, *J* = 13.3, 1.4 Hz, COC$H_{ax}H_{eq}$CHO), 1.46 (1H, d, *J* = 13.3 Hz, CHOC$H_{ax}H_{eq}$CHO), 1.51 (3H, s, CCH_3), 1.55 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}H_{eq}$CHO), 2.36 (1H, m, COCH$_{ax}H_{eq}$CHO), 2.43-2.57 (2H, m, COCH$_{ax}H_{eq}$CHO, CHOCH$_{ax}H_{eq}$CHO), 4.13 (1H, s, PhC*H*), 4.37 (1H, m, CH_2CHOCH_2), 4.42 (1H, m, CH_2CHOCH_2), 6.53 (2H, d, *J* = 7.8 Hz, aromatic), 6.66 (1H, t, *J* = 7.3 Hz, aromatic), 7.06 (2H, t, *J* = 7.3 Hz, aromatic), 7.24-7.44 (5H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.1, 32.36, 32.39, 35.4, 66.2, 68.0, 68.1, 74.7, 110.7, 114.2 (2C), 117.8, 127.7, 128.3 (2C), 128.6 (2C), 128.9 (2C), 138.0, 147.6; HRMS (ESI-TOF) calcd for $C_{21}H_{23}NNaO_3$ $[M+Na]^+$ 360.1570, found 360.1562.



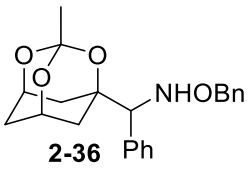
2-34

***O*-Benzylhydroxylamine 2-34 [DK-2-028, DK-5-089].** According to the general procedure B, **2-34** (25.7 mg, 0.0882 mmol) was synthesized from O,Te-acetal **2-8** (34.0 mg, 0.0944 mmol) and formaldehyde *O*-benzyloxime[2)] (38.0 mg, 0.281 mmol) in 93% yield by using Et_3B (1.03 M hexane solution, 0.28 mL, 0.28 mmol) in CH_2Cl_2 (0.94 mL). The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 2/1): colorless oil; IR (film) 3276, 2950, 2929, 2854, 1395, 1323, 1298, 1165, 1149, 1127 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.42 (3H, s, CCH_3), 1.57 (1H, dt, *J* = 13.2, 1.8 Hz, CHOC$H_{ax}H_{eq}$CHO), 1.61 (2H, d, *J* = 13.2 Hz, COC$H_{ax}H_{eq}$CHO x2), 2.35 (2H, m, COCH$_{ax}H_{eq}$CHO x2), 2.51 (1H, dtt, *J* = 13.2, 4.6, 2.3 Hz, CHOCH$_{ax}H_{eq}$CHO), 2.92 (2H, s, NHCH_2), 4.39 (2H, br s, CH_2CHOCH_2 x2), 4.69 (2H, s, PhCH_2), 7.27-7.37 (5H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 32.4, 35.5 (2C), 60.8, 68.0 (2C), 72.4, 75.9, 110.3, 127.8, 128.3 (2C), 128.4 (2C), 137.8; HRMS (ESI-TOF) calcd for $C_{16}H_{21}NNaO_4$ $[M+Na]^+$ 314.1363, found 314.1359.



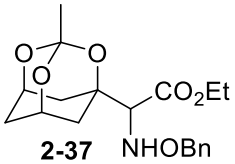
2-35

***O*-Benzylhydroxylamine 2-35 [DK-5-096].** According to the general procedure B, **2-35** (12.8 mg, 0.0363 mmol) was synthesized from O,Te-acetal **2-8** (34.3 mg, 0.0953 mmol) and 3-methylbutanal *O*-benzyl oxime[3] (55.0 mg, 0.288 mmol) in 38% yield by using Et_3B (1.03 M hexane solution, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.95 mL). The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5/1): colorless oil; IR (film) 2952, 2867, 1453, 1394, 1365, 1323, 1298, 1129 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.91 (3H, d, *J* = 6.9 Hz, CHCH_3), 0.95 (3H, d, *J* = 6.9 Hz, CHCH_3), 1.21 (1H, ddd, *J* = 16.6, 9.8, 2.9 Hz, NHCHCH_AH$_B$), 1.42 (3H, s, CCH_3), 1.44 (1H, m, NHCHCH$_A$$H_B$), 1.56 (1H, dt, *J* = 13.2, 1.7 Hz, CHOCH_{ax}H$_{eq}$CHO), 1.66-1.73 (2H, m, COCH_{ax}H$_{eq}$CHO x2), 1.84 (1H, m, $(CH_3)_2$CH), 2.17 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 2.43 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 2.45 (1H, m, CHOCH$_{ax}$$H_{eq}$CHO), 2.74 (1H, dd, *J* = 9.7, 2.3 Hz, NHCH), 4.35-4.43 (2H, m, CH_2CHOCH_2 x2), 4.66 (1H, d, *J* = 11.5 Hz, PhCH_AH$_B$), 4.70 (1H, d, *J* = 11.5 Hz, PhCH$_A$$H_B$), 6.06 (1H, br s, N$H$), 7.27-7.37 (5H, m, aromatic); ^{13}C NMR (100 MHz, CD_3OD) δ 22.1, 24.3, 26.46, 26.52, 33.4, 35.1, 35.3, 36.2, 66.9, 69.8, 69.9, 76.2, 77.0, 111.6, 128.8, 129.3 (2C), 129.5 (2C), 139.3; HRMS (ESI-TOF) calcd for $C_{20}H_{29}NNaO_4$ $[M+Na]^+$ 370.1989, found 370.1999.



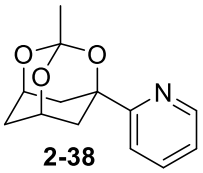
2-36

***O*-Benzylhydroxylamine 2-36 [DK-2-061, DK-5-090].** According to the general procedure B, **2-36** (25.5 mg, 0.0694 mmol) was synthesized from O,Te-acetal **2-8** (35.7 mg, 0.0991 mmol) and benzaldehyde *O*-benzyloxime **6e**[4] (63.0 mg, 0.298 mmol) in 70% yield by using Et_3B (1.03 M hexane solution, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (1.0 mL). The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 4/1): colorless oil; IR (film) 3267, 3061, 3029, 2952, 2864, 1453, 1395, 1323, 1298, 1145, 1127 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.26 (1H, dd, *J* = 13.2, 1.8 Hz, COCH_{ax}H$_{eq}$CHO), 1.37 (1H, dt, *J* = 13.2, 1.7 Hz, CHOCH_{ax}H$_{eq}$CHO), 1.457 (1H, m, COCH_{ax}H$_{eq}$CHO), 1.459 (3H, s, CCH_3) , 2.24 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 2.41 (1H, dtt, *J* = 13.2, 3.6, 1.8 Hz, CHOCH$_{ax}$$H_{eq}$CHO), 2.47 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 4.03 (1H, s, PhC$H$), 4.28-4.35 (2H, m, CH_2CHOCH_2 x2), 4.54 (1H, d, *J* = 11.5 Hz, PhCH_AH$_B$), 4.58 (1H, d, *J* = 11.5 Hz, PhCH$_A$$H_B$), 6.48 (1H, s, N$H$), 7.13-7.18 (2H, m, aromatic), 7.22-7.29 (3H, m, aromatic), 7.30-7.38 (3H, m, aromatic), 7.42-7.47 (2H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 31.2, 32.3, 35.5, 67.97, 68.0, 72.7, 74.4, 76.7, 110.4, 127.6, 127.8, 127.9 (2C), 128.2 (2C), 128.5 (2C), 129.1 (2C), 137.1, 137.7; HRMS (ESI-TOF) calcd for $C_{22}H_{25}NNaO_4$ $[M+Na]^+$ 390.1676, found 390.1681.



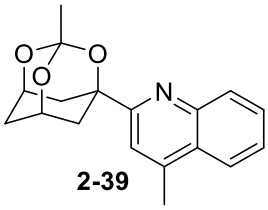
2-37

***O*-Benzylhydroxylamine 2-37 [DK-1-164, DK-5-076]**. According to the general procedure B, **2-37** (12.8 mg, 0.0363 mmol) was synthesized from O,Te-acetal **2-8** (34.3 mg, 0.0953 mmol) and 3-methylbutanal *O*-benzyl oxime[5)] (55.0 mg, 0.288 mmol) in 38% yield by using Et_3B (1.03 M hexane solution, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.95 mL). The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5/1): colorless oil; IR (film) 2952, 2867, 1453, 1394, 1365, 1323, 1298, 1129 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.91 (3H, d, *J* = 6.9 Hz, CHCH_3), 0.95 (3H, d, *J* = 6.9 Hz, CHCH_3), 1.21 (1H, ddd, *J* = 16.6, 9.8, 2.9 Hz, NHCHCH_AH$_B$), 1.42 (3H, s, CCH_3), 1.44 (1H, m, NHCHCH$_A$$H_B$), 1.56 (1H, dt, *J* = 13.2, 1.7 Hz, CHOCH_{ax}H$_{eq}$CHO), 1.66-1.73 (2H, m, COCH_{ax}H$_{eq}$CHO x2), 1.84 (1H, m, $(CH_3)_2$CH), 2.17 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 2.43 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 2.45 (1H, m, CHOCH$_{ax}$$H_{eq}$CHO), 2.74 (1H, dd, *J* = 9.7, 2.3 Hz, NHCH), 4.35-4.43 (2H, m, CH_2CHOCH_2 x2), 4.66 (1H, d, *J* = 11.5 Hz, PhCH_AH$_B$), 4.70 (1H, d, *J* = 11.5 Hz, PhCH$_A$$H_B$), 6.06 (1H, br s, N$H$), 7.27-7.37 (5H, m, aromatic); ^{13}C NMR (100 MHz, CD_3OD) δ 22.1, 24.3, 26.46, 26.52, 33.4, 35.1, 35.3, 36.2, 66.9, 69.8, 69.9, 76.2, 77.0, 111.6, 128.8, 129.3 (2C), 129.5 (2C), 139.3; HRMS (ESI-TOF) calcd for $C_{20}H_{29}NNaO_4$ $[M+Na]^+$ 370.1989, found 370.1999.



2-38

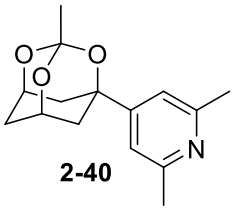
Pyridine 2-38 [DK-1-162, DK-5-013]. According to the general procedure B, **2-38** (9.1 mg, 0.039 mmol) was synthesized from O,Te-acetal **2-8** (31 mg, 0.86 mmol) in 45% yield by using pyridinium (+)-10-Camphorsulfonate (135 mg, 0.43 mmol) and Et_3B (1.03 mol/L hexane solution, 0.42 mL, 0.43 mmol) in CH_2Cl_2 (0.43 mL). The resultant mixture was purified by flash column chromatography on silica gel (5 g, hexane/EtOAc 1:1): colorless solid; m.p. 90-95 °C; IR (film) 3075, 3010, 2963, 2933, 2859, 1591, 1471, 1442, 1397, 1337, 1303, 1261, 1129 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.58 (3H, s, CCH_3), 1.76 (1H, dt, *J* = 13.2, 1.7 Hz, CHOCH_{ax}H$_{eq}$CHO), 2.28-2.35 (4H, m, COCH_2CHO x2), 2.57-2.65 (1H, m, COCH$_{ax}$$H_{eq}$CHO), 4.49-4.55 (2H, m, CH_2CHOCH_2 x2), 7.20 (1H, ddd, *J* = 7.5, 4.6, 1.2 Hz, aromatics), 7.60 (1H, d, *J* = 8.0 Hz, aromatics), 7.74 (1H, td, *J* = 7.5, 1.8 Hz, aromatics), 8.51-8.57 (1H, m, aromatics); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.3, 31.9, 37.2, 68.5, 75.1, 110.8, 118.6, 122.4, 137.0, 148.5, 163.2; HRMS (ESI-TOF) calcd for $C_{13}H_{15}NNaO_3$ $[M+Na]^+$ 256.0944, found 256.0944.



2-39

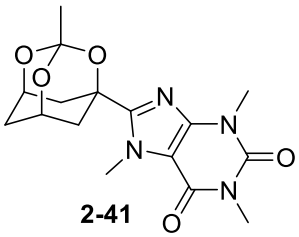
Quinoline 2-39 [DK-3-158, DK-4-024. According to the general procedure B, **2-39** (25 mg, 0.084 mmol) was

synthesized from O,Te-acetal **2-8** (35 mg, 0.97 mmol) in 87% yield by using 4-methylquinolinium (+)-10-Camphorsulfonate (110 mg, 0.29 mmol) and Et3B (1.03 mol/L hexane solution, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.98 mL). The resultant mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 4:1): colorless oil; IR (film) 3063, 3007, 2951, 2929, 2848, 1600, 1561, 1508, 1447, 1396, 1314, 1296, 1147, 1127 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.63 (3H, s, CC*H*$_3$), 1.82 (1H, dt, *J* = 13.3, 1.4 Hz, CHOC*H*$_{ax}$H$_{eq}$CHO), 2.37-2.49 (4H, m, COC*H*$_2$CHO x2), 2.64 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.74 (3H, d, *J* = 0.9 Hz, CC*H*$_3$), 4.56 (2H, m, CH$_2$C*H*OCH$_2$ x2), 7.54 (1H, ddd, *J* = 8.2, 6.9, 1.4 Hz, aromatics), 7.62 (1H, s, aromatics), 7.68 (1H, ddd, *J* = 8.2, 6.9, 1.4 Hz, aromatics), 7.99 (1H, dd, *J* = 8.2, 1.4 Hz, aromatics), 8.03 (1H, d, *J* = 8.7 Hz, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.0, 26.4, 32.0, 37.1, 68.7, 75.7, 110.9, 117.3, 123.7, 126.0, 127.4, 129.0, 129.8, 145.1, 147.2, 162.9; HRMS (ESI-TOF) calcd for $C_{18}H_{19}NNaO_3$ $[M+Na]^+$ 320.1257, found 320.1254.



2-40

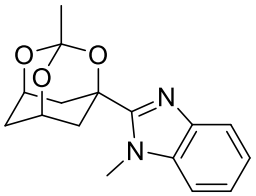
Pyridine 2-40 [DK-1-175, DK-5-018]. According to the general procedure B, **2-40** (7.8 mg, 0.030 mmol) was synthesized from O,Te-acetal **2-8** (32 mg, 0.89 mmol) in 34% yield by using 2,6-dimethylpyridinium (+)-10-Camphorsulfonate (150 mg, 0.44 mmol) and Et3B (1.03 mol/L hexane solution, 0.43 mL, 0.44 mmol) in CH_2Cl_2 (0.44 mL). The resultant mixture was purified by flash column chromatography on silica gel (5 g, hexane/EtOAc 1:2 to 1:3): colorless solid; m.p. 95-100 °C; IR (film) 3007, 2955, 2930, 2854, 1609, 1565, 1396, 1339, 1313, 1293, 1224, 1185, 1128 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.56 (3H, s, CC*H*$_3$), 1.71 (1H, dt, *J* = 13.3, 1.8 Hz, CHOC*H*$_{ax}$H$_{eq}$CHO), 1.98 (2H, d, *J* = 13.3 Hz, COC*H*$_{ax}$H$_{eq}$CHO x2), 2.31-2.38 (2H, m, COCH$_{ax}$*H*$_{eq}$CHO x2), 2.56 (6H, s, CC*H*$_3$ x2), 2.62 (1H, dtt, *J* = 13.3, 4.6, 2.3 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 4.47-4.52 (2H, m, CH$_2$C*H*OCH$_2$ x2), 7.00 (2H, s, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.4, 26.2, 31.9, 37.9, 68.0, 73.1, 110.9, 115.4, 154.6, 157.9; HRMS (ESI-TOF) calcd for $C_{15}H_{20}NO_3$ $[M+H]^+$ 262.1438, found 262.1431.



2-41

Caffeine 2-41 [DK-3-161, DK-5-020]. According to the general procedure B, **2-41** (23 mg, 0.066 mmol) was synthesized from O,Te-acetal **2-8** (32 mg, 0.89 mmol) in 74% yield by using caffeine (+)-10-Camphorsulfonate (114 mg, 0.27 mmol) and Et_3B (1.03 mol/L hexane solution, 0.26 mL, 0.27 mmol) in CH_2Cl_2 (0.89 mL). The resultant mixture was purified by flash column chromatography on silica gel (5 g, hexane/EtOAc 1:3): colorless solid; m.p. 202-206 °C; IR (film) 3006, 2951, 2932, 2853, 1564, 1442, 1396, 1351, 1314, 1296, 1219, 1141, 1131, 1062 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.52 (3H, s, CC*H*$_3$), 1.74 (1H, d, *J* = 13.3 Hz, CHOC*H*$_{ax}$H$_{eq}$CHO), 2.17 (2H, d, *J* =

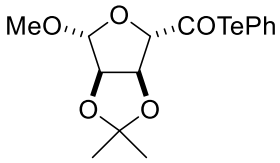
13.3 Hz, COCH_{ax}H$_{eq}$CHO x2), 2.58-2.72 (3H, m, COCH$_{ax}$$H_{eq}$CHO x2, CHOCH$_{ax}$$H_{eq}$CHO), 3.39 (3H, s, CC$H_3$), 3.53 (3H, s, CC$H_3$), 4.20 (3H, s, CC$H_3$), 4.53 (2H, m, CH_2C*H*OCH_2 x2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.0, 27.9, 29.6, 31.8, 34.0, 35.9, 68.0, 73.4, 108.4, 110.5, 147.0, 151.6, 152.0, 155.6; HRMS (ESI-TOF) calcd for $C_{16}H_{20}N_4NaO_5$ $[M+Na]^+$ 371.1326, found 371.1322.



2-42

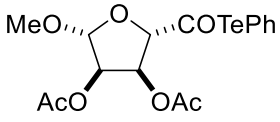
Benzimidazole 2-42 [DK-5-027, DK-5-117]. According to the general procedure B, **2-42** (16 mg, 0.056 mmol) was synthesized from O,Te-acetal **2-8** (34 mg, 0.94 mmol) in 60% yield by using 1-methylbenzimidazolium (+)-10-camphorsulfonate (174 mg, 0.48 mmol) and Et_3B (1.03 mol/L hexane solution, 0.46 mL, 0.47 mmol) in CH_2Cl_2 (0.48 mL). The resultant mixture was purified by flash column chromatography on silica gel (5 g, hexane/EtOAc 1:2): colorless solid; m.p. 139-144 °C; IR (film) 2955, 2927, 2854, 1729, 1468, 1389, 1320, 1298, 1249, 1227, 1146, 1127, 1060, cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.56 (3H, s, CCH_3), 1.77 (1H, dt, *J* = 13.2, 1.8 Hz, CHOCH_{ax}H$_{eq}$CHO), 2.30 (2H, d, *J* = 13.2 Hz, COCH_{ax}H$_{eq}$CHO x2), 2.64 (1H, dtt, *J* = 13.3, 4.6, 2.3 Hz, CHOCH$_{ax}$$H_{eq}$CHO), 2.77-2.86 (2H, m, COCH$_{ax}$$H_{eq}$CHO x2), 4.04 (3H, s, CC$H_3$), 4.54-4.60 (2H, m, CH_2C*H*OCH_2 x2), 7.24-7.34 (2H, m, aromatics), 7.34-7.7.39 (1H, m, aromatics), 7.72-7.78 (1H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.1, 31.8, 32.0, 36.3, 68.3, 73.5, 109.4, 110.5, 119.7, 122.2, 123.0, 136.6, 141.5, 153.7; HRMS (ESI-TOF) calcd for $C_{16}H_{18}N_2NaO_3$ $[M+Na]^+$ 309.1210, found 309.1207.

6-2. 第三章の実験項



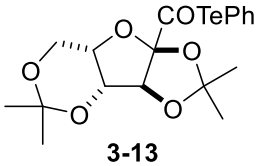
3-9

General procedure C: **Acyl telluride 3-9 [DK-5-023, DK-5-146].** To a solution of carboxylic acid **3-8**[6] (1.5 g, 6.9 mmol) in THF (34 mL) were added *N*-methylmorpholine (0.91 mL, 8.3 mmol) and isobutylchloroformate (0.95 mL, 8.3 mmol) at 0 °C. Then, the reaction mixture was stirred for 30 min at that temperature. To another solution of $(PhTe)_2$ (2.8 g) and $NaBH_4$ (0.78 g) in THF (34 mL) was added MeOH (3.4 mL) at 0 °C. After the latter reaction mixture was stirred for 30 min at the same temperature, the former reaction mixture was added to the latter one. Then, the reaction mixture was stirred for another 30 min at room temperature. The reaction mixture was filtered through a SiO_2 pad (hexane/EtOAc 1:1). The filtrate was concentrated, and residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 10:1) to give **3-9** (2.5 g, 6.2 mmol) in 90% yield: yellow solid; $[\alpha]_D^{27}$ = -89.5 (*c* 1.0, $CHCl_3$); m.p. 53-56 °C; IR (film) 3068, 3054, 2989, 2936, 2839, 1706, 1435, 1382, 1374, 1212, 1199, 1158, 1109, 1091, 1059, 1040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, acetonide), 1.49 (3H, s, acetonide), 3.60 (3H, s, OMe), 4.38 (1H, d, *J* = 0.9 Hz, H4), 4.55 (1H, d, *J* = 6.0 Hz, H2), 5.01 (1H, dd, *J* = 6.0, 1.4 Hz, H3), 5.15 (1H, s, H1), 7.28-7.42 (3H, m, aromatics), 7.67-7.74 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.2, 26.6, 57.8, 80.6, 84.5, 97.8, 111.2, 113.0, 113.8, 128.7, 129.4, 140.0, 206.5; HRMS (ESI-TOF) calcd for $C_{15}H_{18}NaO_5Te$ $[M+Na]^+$ 431.0109, found 431.0101.



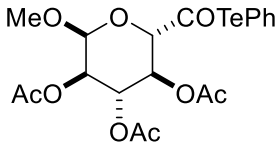
3-11

Acyl telluride 3-11 [DK-6-070]. According to the general procedure C, **3-11** (0.99 g, 1.9 mmol) was synthesized from carboxylic acid **3-10**[7] (0.70 g, 2.1 mmol) in 90% yield by using *N*-methylmorpholine (0.28 mL, 2.5 mmol) and isobutylchloroformate (0.33 mL, 2.5 mmol) in THF (10 mL), and $(PhTe)_2$ (0.86 g) and $NaBH_4$ (0.24 g) in THF (10 mL) and MeOH (1.0 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1): yellow solid; $[\alpha]_D^{25}$ = -83.7 (*c* 1.0, $CHCl_3$); m.p. 87-90 °C; IR (film) 3053, 3001, 2937, 2843, 1754, 1704, 1435, 1372, 1237, 1213, 1081 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.04 (3H, s, OAc), 2.11 (3H, s, OAc), 3.64 (3H, s, OMe), 4.31 (1H, d, *J* = 7.8 Hz, H4), 5.07 (1H, s, H1), 5.27 (1H, d, *J* = 4.6 Hz, H2), 5.45 (1H, dd, *J* = 7.8, 4.6 Hz, H3) 7.28-7.43 (3H, m, aromatics), 7.66-7.74 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.4, 20.5, 57.5, 70.7, 74.4, 89.0, 107.8, 113.6, 128.8, 129.5, 140.0, 169.1, 169.4, 208.1; HRMS (ESI-TOF) calcd for $C_{16}H_{18}NaO_7Te$ $[M+Na]^+$ 475.0007, found 475.0009.



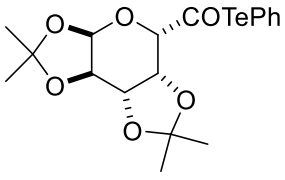
3-13

Acyl telluride 3-13 [DK-5-067, DK-5-170]. According to the general procedure C, **3-13** (1.4 g, 3.0 mmol) was synthesized from carboxylic acid **3-12** (1.0 g, 3.4 mmol) in 88% yield by using *N*-methylmorpholine (0.45 mL, 4.1 mmol) and isobutylchloroformate (0.53 mL, 4.1 mmol) in THF (17 mL), and $(PhTe)_2$ (1.5 g) and $NaBH_4$ (0.39 g) in THF (17 mL) and MeOH (1.7 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1 to 2:1): yellow solid; $[\alpha]_D^{24}$ = -7.03 (*c* 1.0, $CHCl_3$); m.p. 144-146 °C; IR (film) 3056, 2990, 2935, 2870, 1711, 1379, 1177, 1121, 1089, 1037 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, s, acetonide), 1.45 (3H, s, acetonide), 1.49 (3H, s, acetonide), 1.53 (3H, s, acetonide), 4.17 (1H, dd, *J* = 13.7, 2.3 Hz, H5a), 4.22 (1H, d, *J* = 13.7 Hz, H5b), 4.27 (1H, d, *J* = 1.4 Hz, H4), 4.33 (1H, d, *J* = 2.3 Hz, H3), 4.53 (1H, s, H2), 7.29-7.41 (3H, m, aromatics), 7.71-7.77 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 25.8, 27.0, 28.8, 59.9, 72.3, 74.0, 87.2, 98.0, 114.5, 115.0, 116.3, 128.6, 129.4, 139.9, 206.4; HRMS (ESI-TOF) calcd for $C_{18}H_{22}NaO_6Te$ $[M+Na]^+$ 487.0371, found 487.0385.



3-15

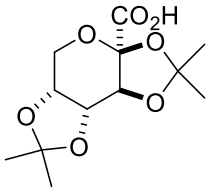
Acyl telluride 3-15 [DK-6-069]. According to the general procedure C, **3-15** (0.99 g, 1.9 mmol) was synthesized from carboxylic acid **3-14**[8] (0.70 g, 2.1 mmol) in 90% yield by using *N*-methylmorpholine (0.28 mL, 2.5 mmol) and isobutylchloroformate (0.33 mL, 2.5 mmol) in THF (10 mL), and $(PhTe)_2$ (0.86 g) and $NaBH_4$ (0.24 g) in THF (10 mL) and MeOH (1.0 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1): yellow solid; $[\alpha]_D^{25}$ = 90.8 (*c* 1.0, $CHCl_3$); m.p. 65-70 °C; IR (film) 3054, 2943, 2845, 1754, 1712, 1434, 1369, 1241, 1219, 1049 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.99 (3H, s, OAc), 2.01 (3H, s, OAc), 2.09 (3H, s, OAc), 3.49 (3H, s, OMe), 3.99 (1H, d, *J* = 10.0 Hz, H5), 4.91 (1H, dd, *J* = 10.5, 3.5 Hz, H2), 5.15 (1H, d, *J* = 3.2 Hz, H1), 5.18 (1H, dd, *J* = 10.0, 10.0 Hz, H4), 5.50 (1H, dd, *J* = 10.0, 10.0 Hz, H3), 7.27-7.41 (3H, m, aromatics), 7.63-7.70 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 20.57, 20.62, 56.2, 67.8, 69.0, 70.4, 77.4, 97.0, 113.3, 128.8, 129.5, 140.1, 169.3, 169.8, 170.1, 204.1; HRMS (ESI-TOF) calcd for $C_{19}H_{22}NaO_9Te$ $[M+Na]^+$ 547.0218, found 547.0204.



3-17

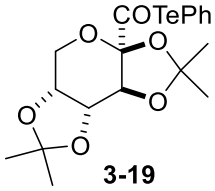
Acyl telluride 3-17 [DK-6-082]. According to the general procedure C, **3-17** (0.90 g, 1.9 mmol) was synthesized

from carboxylic acid **3-16** (0.60 g, 2.2 mmol) in 86% yield by using *N*-methylmorpholine (0.29 mL, 2.6 mmol) and isobutylchloroformate (0.34 mL, 2.6 mmol) in THF (11 mL), and $(PhTe)_2$ (0.90 g) and $NaBH_4$ (0.25 g) in THF (11 mL) and MeOH (1.1 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1): yellow solid; $[\alpha]_D^{28}$ = -206.2 (*c* 1.0, $CHCl_3$); m.p. 96-102 °C; IR (film) 3067, 3053, 2987, 2934, 2902, 1708, 1382, 1255, 1212, 1166, 1110, 1069, 1003 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, acetonide), 1.36 (3H, s, acetonide), 1.50 (3H, s, acetonide), 1.52 (3H, s, acetonide), 4.02 (1H, d, *J* = 1.4 Hz, H5), 4.43 (1H, dd, *J* = 5.0, 2.3 Hz, H2), 4.61 (1H, dd, *J* = 7.8, 1.8 Hz, H4), 4.64 (1H, dd, *J* = 7.8, 1.8 Hz, H3), 5.73 (1H, d, *J* = 5.0 Hz, H1), 7.27-7.40 (3H, m, aromatics), 7.68-7.76 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.4, 24.8, 25.8, 26.0, 70.9, 71.1, 71.9, 77.7, 96.8, 109.4, 110.1, 114.2, 128.5, 129.3, 140.0, 208.0; HRMS (ESI-TOF) calcd for $C_{18}H_{22}NaO_6Te$ $[M+Na]^+$ 487.0371, found 487.0354.



3-18

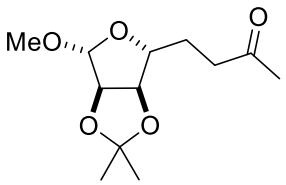
Carboxylic acid 3-18 [DK-6-078]. A solution of commercially available 2,3:4,5-di-*O*-isopropylidene-β-D-fructopyranose (1.0 g, 3.8 mmol) in a mixture of CH_3CN (19 mL) and H_2O (19 mL) was stirred with $PhI(OAc)_2$ (3.7 g, 12 mmol) and 2-Azaadamantane-*N*-oxyl (29 mg, 0.19 mmol) at room temperature for 14 h, and then 1 mol/L HCl (20 mL) was added. The resultant mixture was extracted with EtOAc (10 mL x3), and the combined organic layers were washed with brine (20 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 1:1) to afford carboxylic acid **3-18** (1.1 g, 3.8 mmol) in 100% yield: colorless oil; $[\alpha]_D^{24}$ = -43.4 (*c* 4.2, $CHCl_3$); IR (film) 3218, 2991, 2940, 1776, 1750, 1455, 1382, 1343, 1309, 1254, 1214, 1176, 1165, 1110, 1072, 1042 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, acetonide), 1.43 (3H, s, acetonide), 1.48 (3H, s, acetonide), 1.54 (3H, s, acetonide), 3.88 (1H, dd, *J* = 13.3, 0.9 Hz, H5a), 3.92 (1H, dd, *J* = 13.3, 1.7 Hz, H5b), 4.26 (1H, d, *J* = 7.8 Hz, H4), 4.62 (1H, dd, *J* = 7.8, 2.3 Hz, H3), 4.65 (1H, d, *J* = 2.7 Hz, H2), 9.69 (1H, s, CO_2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.9, 24.3, 25.6, 26.0, 61.7, 69.6, 69.9, 72.7, 98.8, 109.4, 111.1, 169.5; HRMS (ESI-TOF) calcd for $C_{12}H_{17}O_7$ $[M-H]^-$ 273.0980, found 273.0973.



3-19

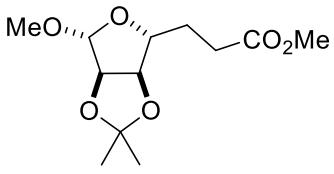
Acyl telluride 3-19 [DK-6-079]. According to the general procedure C, **3-19** (0.85 g, 1.8 mmol) was synthesized from carboxylic acid **3-18** (0.62 g, 2.3 mmol) in 78% yield by using *N*-methylmorpholine (0.30 mL, 2.7 mmol) and isobutylchloroformate (0.35 mL, 2.7 mmol) in THF (11 mL), and $(PhTe)_2$ (0.92 g) and $NaBH_4$ (0.26 g) in THF (11 mL) and MeOH (1.1 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc

10:1): yellow solid; $[\alpha]_D^{22}$ = -73.5 (*c* 1.0, $CHCl_3$); m.p. 150-153 °C; IR (film) 3054, 2989, 2937, 1717, 1452, 1435, 1383, 1314, 1253, 1212, 1173, 1119, 1072, 1019 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, s, acetonide), 1.42 (3H, s, acetonide), 1.54 (3H, s, acetonide), 1.61 (3H, s, acetonide), 3.97 (1H, dd, *J* = 14.6, 1.4 Hz, H5a), 4.01 (1H, dd, *J* = 14.6, 1.4 Hz, H5b), 4.27 (1H, dt, *J* = 7.8, 1.4 Hz, H4), 4.50 (1H, d, *J* = 2.8 Hz, H2), 4.58 (1H, dd, *J* = 7.8, 2.8 Hz, H3), 7.28-7.41 (3H, m, aromatics), 7.69-7.75 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.9, 24.7, 26.0, 26.2, 62.0, 69.7, 70.0, 72.0, 104.5, 109.3, 110.3, 114.4, 128.7, 129.3, 140.0, 206.4; HRMS (ESI-TOF) calcd for $C_{18}H_{22}NaO_6Te$ $[M+Na]^+$ 487.0371, found 487.0371.



3-20

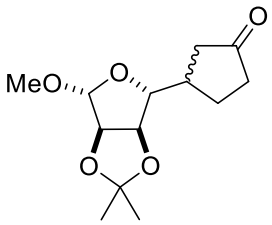
General procedure D: **Ketone 3-20 [DK-4-044, DK-5-150].** To a solution of acyl telluride **3-9** (50 mg, 0.12 mmol) in CH_2Cl_2 (6.2 mL) were added methyl vinyl ketone (20 μL, 0.25 mmol) and Et_3B (1.03 mol/L hexane solution, 0.36 mL) at room temperature. Then, the reaction mixture was stirred for 15 min at rt, and then the reaction mixture was concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 3:1) to give **3-20** (28 mg, 0.11 mmol) in 92% yield: colorless oil; $[\alpha]_D^{18}$ = -50.7 (*c* 0.69, $CHCl_3$); IR (film) 2988, 2937, 2834, 1715, 1372, 1273, 1239, 1210, 1162, 1108, 1092, 1060, 1014 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.29 (3H, s, acetonide), 1.45 (3H, s, acetonide), 1.70-1.88 (2H, m, $CH_3COCH_2CH_2$), 2.14 (3H, s, CH_3COCH_2), 2.46-2.64 (2H, m, CH_3COCH_2), 3.32 (3H, s, OMe), 4.10 (1H, dd, *J* = 9.2, 6.4 Hz, H4), 4.51 (1H, d, *J* = 6.0 Hz, H3), 4.58 (1H, d, *J* = 6.0 Hz, H2), 4.92 (1H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.0, 26.5, 28.8, 30.0, 40.1, 55.1, 84.1, 85.4, 86.3, 109.6, 112.3, 207.7; HRMS (ESI-TOF) calcd for $C_{12}H_{20}NaO_5$ $[M+Na]^+$ 267.1203, found 267.1200.



3-21

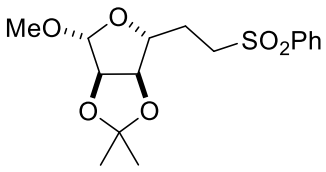
Methyl ester 3-21 [DK-4-046, DK-5-152]. According to the general procedure D, **3-21** (31 mg, 0.12 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol), methyl acrylate (22 μL, 0.25 mmol) and tris(trimethylsilyl)silane (0.11 mL, 0.37 mmol) in 100% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{29}$ = -32.3 (*c* 2.1, $CHCl_3$); IR (film) 2989, 2938, 2835, 1739, 1440, 1373, 1242, 1209, 1164, 1108, 1093, 1061 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30 (3H, s, acetonide), 1.46 (3H, s, acetonide), 1.81-1.92 (2H, m, $CH_3COCH_2CH_2$), 2.42 (1H, dt, *J* = 16.5, 7.8 Hz, $CH_AH_BCO_2CH_3$), 2.49 (1H, dt, *J* = 16.5, 7.8 Hz, $CH_AH_BCO_2Me$), 3.33 (3H, s, OMe), 3.67 (3H, s, $CH_2CO_2CH_3$), 4.15 (1H, t, *J* = 7.8 Hz, H4), 4.53 (1H, d, *J* = 6.0 Hz, H3), 4.60 (1H, d, *J* = 6.0 Hz, H2), 4.93

(1H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 26.4, 30.2, 30.7, 51.6, 55.1, 84.0, 85.4, 86.1, 109.7, 112.3, 173.4; HRMS (ESI-TOF) calcd for $C_{12}H_{20}NaO_6$ [M+Na]$^+$ 283.1152, found 283.1161.



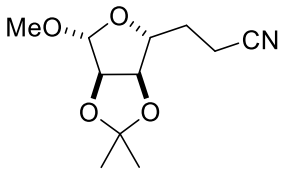
3-22

Ketone 3-22 [DK-4-043, DK-5-151]. According to the general procedure D, **3-22** (25 mg, 0.098 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol) and cyclopentenone (21 μL, 0.25 mmol) in 82% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 3:1). Inseparable diastereomixture (3:1): colorless solid; IR (film) 2984, 2935, 2835, 1743, 1373, 1272, 1240, 1210, 1160, 1107, 1091, 1061, 1029 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.31, 1.33 (3H, s x2, acetonide), 1.46-1.49 (3H, m, acetonide), 1.58-2.48 (7H, m, cyclopentanone), 3.33, 3.39 (3H, s x2, OMe), 3.94-4.02 (1H, m, H4), 4.49-4.65 (2H, m, H3, H2), 4.94, 4.97 (1H, s x2, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.95, 24.99, 25.8, 26.47, 26.48, 26.8, 37.8, 37.9, 40.8, 40.9, 41.6, 42.8, 55.37, 55.42, 82.4, 83.0, 85.4, 85.5, 90.7, 90.9, 109.4, 109.6, 112.55, 112.56, 217.7, 218.3; HRMS (ESI-TOF) calcd for $C_{13}H_{20}NaO_5$ [M+Na]$^+$ 279.1203, found 279.1196.



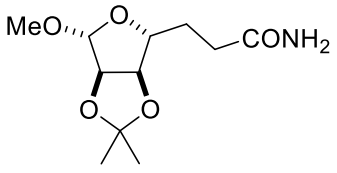
3-23

Phenyl sulfone 3-23 [DK-4-067, DK-5-153]. According to the general procedure D, **3-23** (41 mg, 0.12 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol), phenyl vinyl sulfone (41 mg, 0.25 mmol) and tris(trimethylsilyl)silane (0.11 mL, 0.37 mmol) in 100% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 3:1): colorless solid; $[\alpha]_D^{24}$ = -13.2 (*c* 2.6, $CHCl_3$); m.p. 46-50 °C; IR (film) 3064, 2987, 2936, 2834, 1447, 1381, 1308, 1278, 1212, 1144, 1106, 1090, 1061 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.26 (3H, s, acetonide), 1.41 (3H, s, acetonide), 1.89-1.98 (2H, m, CH_2CH$_2$SO$_2$Ph), 3.10-3.21 (4H, m, CH_AH$_B$SO$_2$Ph, OMe), 3.22-3.32 (1H, m, CH$_A$$H_BSO_2$Ph), 4.10 (1H, dd, *J* = 7.4, 7.4 Hz, H4), 4.48 (1H, d, *J* = 6.3 Hz, H3), 4.54 (1H, d, *J* = 5.7 Hz, H2), 4.88 (1H, s, H1), 7.52-7.59 (2H, m, aromatics), 7.61-7.68 (1H, m, aromatics), 7.86-7.92 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 26.3, 28.5, 53.6, 55.1, 83.8, 85.09, 85.13, 109.7, 112.5, 128.0, 129.3, 133.8, 138.9; HRMS (ESI-TOF) calcd for $C_{16}H_{22}NaO_6S$ [M+Na]$^+$ 365.1029, found 365.1024.



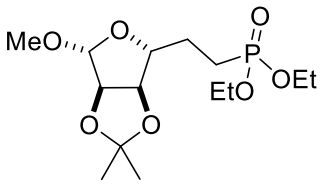
3-24

Cyanide 3-24 [DK-4-066, DK-5-154]. According to the general procedure D, **3-24** (20 mg, 0.088 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol), acrylonitrile (16 μL, 0.25 mmol) and tris(trimethylsilyl)silane (0.11 mL, 0.37 mmol) in 73% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1): colorless oil; $[\alpha]_D^{28}$ = -25.2 (*c* 1.6, $CHCl_3$); IR (film) 2988, 2937, 2836, 2248, 1382, 1374, 1273, 1241, 1210, 1160, 1107, 1093, 1050 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30 (3H, s, acetonide), 1.46 (3H, s, acetonide), 1.88 (1H, t, *J* = 7.8 Hz, C*H*$_A$H$_B$CH$_2$CN), 1.90 (1H, t, *J* = 7.8 Hz, CH$_A$*H*$_B$CH$_2$CN), 2.44 (1H, dt, *J* = 16.9, 7.8 Hz, C*H*$_A$H$_B$CN), 2.52 (1H, dt, *J* = 16.9, 7.8 Hz, CH$_A$*H*$_B$CN), 3.35 (3H, s, OMe), 4.21 (1H, t, *J* = 7.8 Hz, H4), 4.53 (1H, d, *J* = 6.0 Hz, H3), 4.60 (1H, d, *J* = 6.0 Hz, H2), 4.95 (1H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 25.0, 26.4, 30.9, 55.5, 83.7, 85.23, 85.25, 110.0, 112.7, 119.0; HRMS (ESI-TOF) calcd for $C_{11}H_{17}NNaO_4$ $[M+Na]^+$ 250.1050, found 250.1039.



3-25

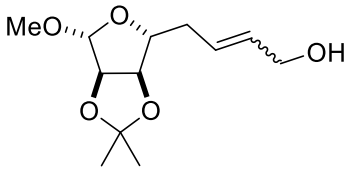
Amide 3-25 [DK-4-068, DK-5-158]. According to the general procedure D, **3-25** (27 mg, 0.11 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol), acrylamide (17 mg, 0.25 mmol) and tris(trimethylsilyl)silane (0.11 mL, 0.37 mmol) in 92% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (EtOAc): colorless oil; $[\alpha]_D^{24}$ = -23.6 (*c* 1.6, $CHCl_3$); IR (film) 3414, 3342, 3203, 2987, 2936, 2836, 1668, 1621, 1419, 1381, 1275, 1242, 1210, 1160, 1105, 1092, 1059 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.29 (3H, s, acetonide), 1.45 (3H, s, acetonide), 1.78-1.94 (2H, m, C*H*$_2$CH$_2$CONH$_2$), 2.25-2.42 (2H, m, C*H*$_2$CONH$_2$), 3.33 (3H, s, OMe), 4.15 (1H, dd, *J* = 9.8, 6.3 Hz, H4), 4.53 (1H, d, *J* = 6.3 Hz, H3), 4.59 (1H, d, *J* = 6.3 Hz, H2), 4.92 (1H, s, H1), 5.71 (1H, s, N*H*$_A$H$_B$), 5.98 (1H, s, NH$_A$*H*$_B$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.9, 26.4, 30.5, 32.3, 55.1, 84.0, 85.4, 86.2, 109.7, 112.3, 174.6; HRMS (ESI-TOF) calcd for $C_{11}H_{19}NNaO_5$ $[M+Na]^+$ 268.1155, found 268.1148.



3-26

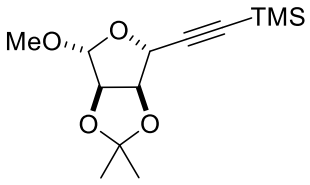
Diethyl phosphonate 3-26 [DK-4-069, DK-5-159]. According to the general procedure D, **3-26** (31 mg, 0.092

mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol), diethyl vinylphosphate (38 mg, 0.25 mmol) and tris(trimethylsilyl)silane (0.11 mL, 0.37 mmol) in 77% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (EtOAc): colorless oil; $[\alpha]_D^{24}$ = -21.1 (*c* 1.8, $CHCl_3$); IR (film) 2984, 2935, 1444, 1380, 1240, 1162, 1095, 1057, 1029 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.29 (3H, s, acetonide), 1.30 (6H, t, *J* = 6.8 Hz, CH_2CH_3 x2), 1.45 (3H, s, acetonide), 1.68-1.99 (4H, m, $CH_2CH_2P(=O)$), 3.33 (3H, s, OMe), 4.00-4.16 (5H, m, H4, OCH_2CH_3 x2), 4.50 (1H, dd, *J* = 6.0, 0.9 Hz, H3), 4.58 (1H, d, *J* = 6.0 Hz, H2), 4.93 (1H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.4 (d, *J* = 5.8 Hz), 22.7 (d, *J* = 143.7 Hz), 24.9, 26.4, 28.0 (d, *J* = 4.8 Hz), 55.1, 61.5 (dd, *J* = 6.7, 1.9 Hz), 83.7, 85.3, 87.0 (d, *J* = 18.2 Hz), 109.6, 112.3; HRMS (ESI-TOF) calcd for $C_{14}H_{27}NaO_7P$ $[M+Na]^+$ 361.1387, found 361.1396.



3-27

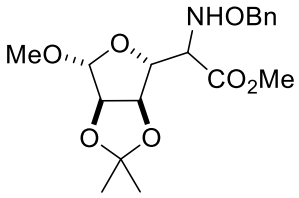
Alcohol 3-27 [DK-5-033, DK-5-173]. According to the general procedure D, **3-27** (8.0 mg, 0.033 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol) and 2-vinyloxirane (20 μL, 0.25 mmol) in 28% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 1:1): colorless oil; $[\alpha]_D^{25}$ = -32.3 (*c* 0.50, $CHCl_3$); IR (film) 3416, 2987, 2935, 2856, 2837, 1377, 1273, 1240, 1211, 1160, 1106, 1092, 1061, 1030 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (3H, s, acetonide), 1.47 (3H, s, acetonide), 2.23-2.47 (2H, m, $CHCH_2CH=CH$), 3.34 (3H, s, OMe), 4.12 (2H, d, *J* = 5.5 Hz, CH_2OH), 4.21 (1H, dd, *J* = 7.8, 7.8 Hz, H4), 4.55 (1H, d, *J* = 6.4 Hz, H3), 4.61 (1H, d, *J* = 6.4 Hz, H2), 4.95 (1H, s, H1), 5.69 (1H, ddd, *J* = 15.6, 5.5, 5.5 Hz, CH=C*H*), 5.75 (1H, ddd, *J* = 15.6, 5.5, 5.5 Hz, CH=C*H*), O*H* missing; ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.0, 26.5, 37.9, 54.9, 63.5, 83.4, 85.5, 86.6, 109.5, 112.3, 128.2, 132.1; HRMS (ESI-TOF) calcd for $C_{12}H_{20}NaO_5$ $[M+Na]^+$ 267.1203, found 267.1192.



3-28

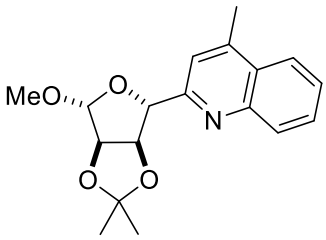
Acetylene 3-28 [DK-4-156, DK-5-193]. According to the general procedure D, **3-28** (11 mg, 0.041 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol) and *p*-Tolyl[2-(trimethylsilyl)ethynyl]sulfone (62 mg, 0.25 mmol) in 34% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 20:1): colorless solid; $[\alpha]_D^{24}$ = -67.1 (*c* 0.61, $CHCl_3$); m.p. 29-32 °C; IR (film) 2988, 2959, 2937, 2833, 2177, 1374, 1250, 1210, 1197, 1161, 1111, 1092, 1044, 1032 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.15 (9H, s, TMS), 1.30 (3H, s, acetonide), 1.45 (3H, s, acetonide), 3.37 (3H, s, OMe), 4.67 (1H, d, *J* = 6.0 Hz, H2), 4.77 (1H, s, H4), 4.87 (1H, d, *J* = 6.0 Hz, H3), 5.03 (1H, s, H1); ^{13}C NMR (100

MHz, $CDCl_3$) δ -0.3, 24.9, 26.3, 54.2, 75.5, 85.2, 85.4, 90.7, 103.0, 109.0, 112.6; HRMS (ESI-TOF) calcd for $C_{13}H_{22}NaO_4Si$ $[M+Na]^+$ 293.1180, found 293.1180.



3-29

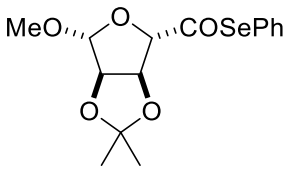
Hydroxyl amine 3-29 [DK-4-171, DK-5-166]. According to the general procedure D, **3-29** (33 mg, 0.090 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol) and methyl 2-(Benzyloxyimino)ethanate[9] (48 mg, 0.25 mmol) in 75% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1). Inseparable diastereomixture (7:3): colorless oil; IR (film) 3259, 3031, 2989, 2937, 2841, 1743, 1454, 1437, 1373, 1239, 1205, 1160, 1107, 1093, 1061 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.27, 1.30 (3H, s x2, acetonide), 1.43, 1.44 (3H, s x2, acetonide), 3.31 (3H, s, OMe), 3.61-3.66 (1H, m, NHC*H*CO_2CH_3), 3.78, 3.79 (3H, s x2, CO_2C*H*$_3$), 4.10 (0.7H, d, *J* = 10.1 Hz, H4a), 4.47 (1H, d, *J* = 6.4 Hz, H2a, H2b), 4.54 (0.3H, d, *J* = 5.5 Hz, H4b), 4.56 (0.3H, d, *J* = 5.5 Hz, H3b), 4.61-4.78 (2.7H, m, NHOC*H*$_2$, H3a), 4.86 (0.3H, s, H1b), 4.92 (0.7H, s, H1a), 7.27-7.38 (5H, m, aromatics), N*H* missing; ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 25.1, 26.4, 52.2, 52.3, 55.7, 55.9, 65.9, 66.4, 76.2, 76.4, 82.1, 82.3, 84.8, 84.9, 85.0, 85.6, 110.0, 111.0, 112.3, 112.5, 127.9, 128.26, 128.29, 128.6, 128.8, 137.4, 137.7, 171.1, 172.5; HRMS (ESI-TOF) calcd for $C_{18}H_{25}NNaO_7$ $[M+Na]^+$ 390.1523, found 390.1513.



3-30

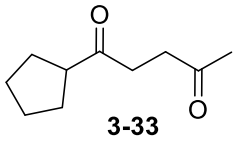
Lepidine 3-30 [DK-4-170, DK-5-174]. According to the general procedure D, **3-30** (25 mg, 0.079 mmol) was synthesized from telluride **3-9** (50 mg, 0.12 mmol) and 4-methylquinolinium (+)-10-camphorsulfonate[10] (92 mg, 0.25 mmol) in 66% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 10:1): colorless oil; $[\alpha]_D^{24}$ = -53.2 (*c* 1.2, $CHCl_3$); IR (film) 3063, 2985, 2933, 2834, 1604, 1564, 1509, 1447, 1376, 1271, 1239, 1209, 1160, 1104, 1092, 1056, 1043 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.42 (3H, s, acetonide), 1.60 (3H, s, acetonide), 2.71 (3H, d, *J* = 0.9 Hz, C*C*H_3), 3.29 (3H, s, OMe), 4.64 (1H, d, *J* = 6.0 Hz, H2), 5.16 (1H, s, H1), 5.42 (1H, s, H4), 5.76 (1H, dd, *J* = 6.0, 1.4 Hz, H3), 7.51 (1H, s, aromatics), 7.53 (1H, ddd, *J* = 8.7, 6.9, 1.4 Hz, aromatics), 7.68 (1H, ddd, *J* = 8.7, 6.9, 1.4 Hz, aromatics), 7.96 (1H, dd, *J* = 8.2, 0.9 Hz, aromatics), 8.08 (1H, dd, *J* = 8.7, 0.9 Hz, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 25.2, 26.7, 55.4, 83.6, 85.3, 88.7, 110.5, 112.4, 119.5, 123.5, 126.1, 127.3, 129.0, 130.0, 144.2, 147.0,

159.5; HRMS (ESI-TOF) calcd for $C_{18}H_{21}NNaO_4$ $[M+Na]^+$ 338.1363, found 338.1363.



3-31

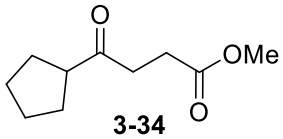
Acyl selenide 3-31 [DK-5-160]. To a solution of compound **3-8** (0.20 g, 0.92 mmol) in CH_2Cl_2 (1.8 mL) were added $(PhSe)_2$ (0.43 g, 1.4 mmol) and tributylphosphane (0.46 mL, 1.8 mmol) at 0 °C. The reaction mixture was stirred for 24 h at room temperature, and was then directly subjected to flash column chromatography on silica gel (hexane/EtOAc 10:1) to afford **3-31** (0.30 g, 0.84 mmol) in 91% yield: light pink solid; $[\alpha]_D^{24}$ = -50.6 (*c* 1.0, $CHCl_3$); m.p. 75-78 °C; IR (film) 3059, 2988, 2935, 2839, 1713, 1440, 1376, 1211, 1159, 1108, 1093, 1042 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.33 (3H, s, acetonide), 1.50 (3H, s, acetonide), 3.59 (3H, s, OMe), 4.57 (1H, d, *J* = 6.0 Hz, H2), 4.66 (1H, s, H4), 5.12 (1H, d, *J* = 6.0 Hz, H3), 5.15 (1H, s, H1), 7.34-7.43 (3H, m, aromatics), 7.47-7.54 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.2, 26.5, 57.1, 81.3, 84.4, 93.5, 110.8, 113.0, 125.6, 128.9, 129.3, 136.0, 201.8; HRMS (ESI-TOF) calcd for $C_{15}H_{18}NaO_5Se$ $[M+Na]^+$ 381.0212, found 381.0200.



3-33

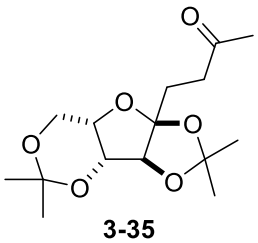
Ketone 3-33 [DK-6-022, DK-6-139]. A solution of $(PhTe)_2$ (0.10 g, 0.24 mmol) and $NaBH_4$ (28 mg, 0.73 mmol) in THF (1.2 mL) was added MeOH (0.12 mL) at 0 °C. After the latter reaction mixture was stirred for 30 min at the same temperature, cyclopentane carbonylchliride (59 μL, 0.49 mmol) in THF (1.2 mL) was added to the mixture at 0°C. Then, the reaction mixture was stirred for another 30 min at room temperature. The reaction mixture was passed through a short pad of silica gel (hexane) to afford the crude telluride **3-32** as a mixture of $(PhTe)_2$. This mixture was used in the next step without further purification.

To a solution of crude **3-32** in CH_2Cl_2 (9.8 mL) were added methyl vinyl ketone (79 μL, 1.0 mmol) and Et_3B (1.03 mol/L hexane solution, 1.4 mL, 1.5 mmol) at room temperature. Then, the reaction mixture was stirred for 30 min at rt, and then the reaction mixture was concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5:1) to give **3-33** (51 mg, 0.30 mmol) in 61% yield: colorless oil; IR (film) 2955, 2913, 2870, 1710, 1451, 1399, 1363, 1227, 1194, 1166, 1105, 1021 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.50-1.88 (8H, m, C*H*$_2$ x4), 2.18 (3H, s, C(=O)C*H*$_3$), 2.67-2.75 (4H, m, C(=O)C*H*$_2$C*H*$_2$C(=O)), 2.90 (1H, dddd, *J* = 8.2, 8.2, 8.2, 8.2 Hz, C*H*C(=O)); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.0, 28.9, 30.0, 35.2, 36.9, 51.3, 207.4, 211.7; HRMS (ESI-TOF) calcd for $C_{10}H_{16}NaO_2$ $[M+Na]^+$ 191.1043, found 191.1044.



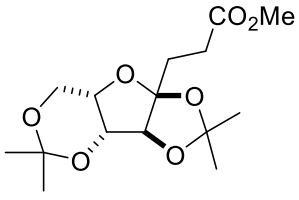
3-34

Methyl ester 3-34 [DK-6-023, DK-6-140]. According to the procedure of **3-33**, **3-34** (44 mg, 0.24 mmol) was synthesized from cyclopentane carbonylchliride (59 μL, 0.49 mmol) in 49% yield over 2 steps by using $(PhTe)_2$ (0.10 g, 0.24 mmol) and NaBH4 (28 mg, 0.73 mmol) in THF (1.2 mL) and MeOH (0.12 mL) for the first reaction, and methyl acrylate (88 μL, 1.0 mmol) and Et_3B (1.03 M in hexane, 1.4 mL, 1.5 mmol) in CH_2Cl_2 (9.8 mL) for the second reaction: colorless oil; IR (film) 2953, 2870, 1739, 1711, 1438, 1410, 1362, 1210, 1171, 1105, 1023 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.46-1.84 (8H, m, C*H*$_2$ x4), 2.25 (2H, t, *J* = 6.4 Hz, C*H*$_2$$CO_2Me$), 2.72 (2H, t, *J* = 6.4 Hz, C(=O)C*H*$_2$), 2.85 (1H, dddd, *J* = 8.2, 8.2, 8.2, 8.2 Hz, C*H*C(=O)), 3.61 (3H, s, CO_2C*H*$_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.0, 27.8, 28.8, 36.1, 51.3, 51.7, 173.4, 211.2; HRMS (ESI-TOF) calcd for $C_{10}H_{16}NaO_3$ $[M+Na]^+$ 207.0992, found 207.0996.



3-35

Ketone 3-35 [DK-5-112, DK-5-180]. According to the general procedure D, **3-35** (27 mg, 0.090 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol) and methyl vinyl ketone (18 μL, 0.22 mmol) in 82% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1): colorless oil; $[\alpha]_D^{24}$ = 0.035 (*c* 2.3, $CHCl_3$); IR (film) 2990, 2935, 1715, 1441, 1378, 1288, 1239, 1197, 1167, 1120, 1079 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (3H, s, acetonide), 1.36 (3H, s, acetonide), 1.40 (3H, s, acetonide), 1.44 (3H, s, acetonide), 2.12-2.24 (5H, m, C*H*$_2$$CH_2$C(=O)$CH_3$, C(=O)C*H*$_3$), 2.66-2.89 (2H, m, C*H*$_2$C(=O)CH_3), 3.95-4.02 (2H, m, H4, H5a), 4.05 (1H, dd, *J* = 13.7, 2.3 Hz, H5b), 4.18-4.24 (2H, m, H2, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.6, 26.3, 27.2, 28.9, 29.9, 31.7, 38.4, 60.3, 71.8, 73.7, 86.7, 97.3, 110.8, 114.7, 208.4; HRMS (ESI-TOF) calcd for $C_{15}H_{24}NaO_6$ $[M+Na]^+$ 323.1465, found 323.1459.

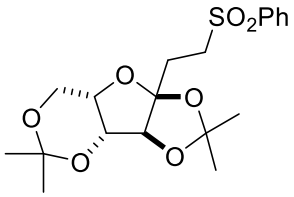


3-36

Methyl ester 3-36 [DK-5-137, DK-5-178]. According to the general procedure D, **3-36** (33 mg, 0.10 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol), methyl acrylate (19 μL, 0.22 mmol) and tris(trimethylsilyl)silane (0.10 mL, 0.33 mmol) in 91% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The

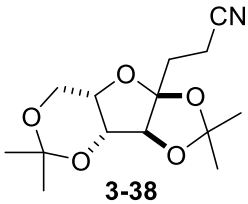
residue was purified by flash column chromatography on silica gel (hexane/EtOAc 4:1).

Colorless oil; $[\alpha]_D^{24}$ = 2.0 (*c* 3.4, $CHCl_3$); IR (film) 2990, 2938, 1739, 1440, 1375, 1239, 1199, 1170, 1122, 1079, 1022 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, acetonide), 1.35 (3H, s, acetonide), 1.39 (3H, s, acetonide), 1.44 (3H, s, acetonide), 2.22-2.31 (2H, m, C*H*$_2$CH$_2$CO$_2$CH$_3$), 2.54-2.72 (2H, m, C*H*$_2$CO$_2$CH$_3$), 3.64 (3H, s, CO$_2$C*H*$_3$), 3.95-4.07 (3H, m, H4, H5a, H5b), 4.21 (1H, d, *J* = 1.8 Hz, H3), 4.26 (1H, s, H2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 26.4, 27.2, 28.7, 28.9, 32.8, 51.5, 60.3, 71.9, 73.6, 86.5, 97.3, 110.9, 114.5, 174.0; HRMS (ESI-TOF) calcd for $C_{15}H_{24}NaO_7$ $[M+Na]^+$ 339.1414, found 339.1417.



3-37

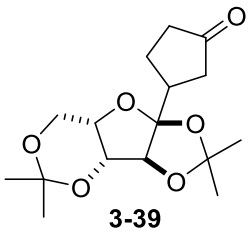
Phenyl sulfone 3-37 [DK-5-138, DK-5-181]. According to the general procedure D, **3-37** (40 mg, 0.10 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol), phenyl vinyl sulfone (36 mg, 0.22 mmol) and tris(trimethylsilyl)silane (0.10 mL, 0.33 mmol) in 91% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1): colorless oil; $[\alpha]_D^{24}$ = 4.4 (*c* 1.0, $CHCl_3$); IR (film) 3063, 2991, 2935, 1447, 1377, 1312, 1285, 1239, 1205, 1147, 1120, 1082, 1018 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.17 (3H, s, acetonide), 1.29 (3H, s, acetonide), 1.36 (3H, s, acetonide), 1.42 (3H, s, acetonide), 2.24-2.42 (2H, m, C*H*$_2$CH$_2$SO$_2$Ph), 3.32-3.53 (2H, m, C*H*$_2$SO$_2$Ph), 3.91 (1H, d, *J* = 13.3 Hz, H5a), 3.94-3.99 (1H, m, H4), 3.99 (1H, dd, *J* = 13.3, 2.3 Hz, H5b), 4.20 (1H, d, *J* = 1.8 Hz, H3), 4.22 (1H, s, H2), 7.50-7.66 (3H, m, aromatics), 7.87-7.97 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.6, 26.3, 27.1, 28.6, 30.8, 51.7, 60.1, 72.1, 73.3, 86.7, 97.3, 111.3, 113.4, 128.0, 129.2, 133.6, 138.9; HRMS (ESI-TOF) calcd for $C_{19}H_{26}NaO_7S$ $[M+Na]^+$ 421.1291, found 421.1288.



3-38

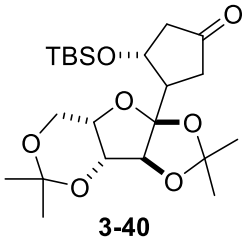
Cyanide 3-38 [DK-5-142, DK-5-179]. According to the general procedure D, **3-38** (22 mg, 0.078 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol), acrylonitrile (14 μL, 0.22 mmol) and tris(trimethylsilyl)silane (0.10 mL, 0.33 mmol) in 71% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 3:1): colorless oil; $[\alpha]_D^{24}$ = 0.94 (*c* 1.5, $CHCl_3$); IR (film) 2991, 2938, 2248, 1447, 1376, 1286, 1238, 1200, 1186, 1173, 1120, 1078, 1041, 1021 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, s, acetonide), 1.36 (3H, s, acetonide), 1.42 (3H, s, acetonide), 1.47 (3H, s, acetonide), 2.22-2.38 (2H, m, C*H*$_2$CH$_2$CN), 2.58-2.74 (2H, m, C*H*$_2$CN), 3.96-4.11 (3H, m, H4, H5a, H5b), 4.26 (1H, d, *J* = 1.8 Hz, H3), 4.29 (1H, s, H2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.4, 18.6, 26.4, 27.2, 28.9, 33.9, 60.2,

72.2, 73.4, 86.5, 97.5, 111.5, 113.3, 119.9; HRMS (ESI-TOF) calcd for $C_{14}H_{21}NNaO_5$ $[M+Na]^+$ 306.1312, found 306.1306.



3-39

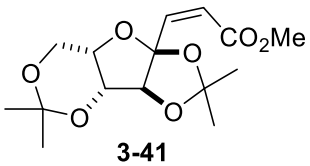
Ketone 3-39 [DK-5-113, DK-5-184]. According to the general procedure D, **3-39** (22 mg, 0.070 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol) and cyclopentenone (18 μL, 0.22 mmol) in 64% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1). Inseparable diastereomixture (1:1): colorless oil; IR (film) 2989, 2935, 1740, 1375, 1241, 1195, 1121, 1080, 1009 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32, 1.34, 1.35 (6H, s x3, acetonide), 1.40, 1.41 (3H, s x2, acetonide), 1.46, 1.48 (3H, s x2, acetonide), 2.04-2.76 (7H, m, cyclopentanone), 3.96-4.10 (3H, m, H4, H5a, H5b), 4.18-4.32 (2H, m, H3, H2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 18.8, 24.2 (2 peaks), 26.78, 26.81, 27.5, 27.6, 28.6, 28.8, 37.8, 38.1, 40.8, 40.9, 43.4, 44.0, 60.27, 60.34, 71.8, 71.9, 73.65, 73.67, 86.2, 86.5, 97.3, 97.5, 111.0, 111.2, 115.9, 116.2, 218.8, 219.1; HRMS (ESI-TOF) calcd for $C_{16}H_{24}NaO_6$ $[M+Na]^+$ 335.1465, found 335.1453.



3-40

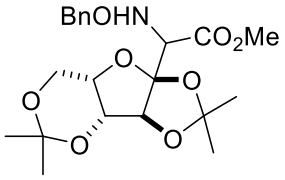
Ketone 3-40 [DK-5-187, DK-5-191]. According to the general procedure D, **3-40** (32 mg, 0.072 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol) and (*R*)-4-(*tert*-butyldimethylsilyloxy)-2-cyclopentene-1-one (46 mg, 0.22 mmol) in 65% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1): colorless oil; $[\alpha]_D^{25}$ = 50.1 (*c* 1.6, $CHCl_3$); IR (film) 2991, 2953, 2930, 2857, 1747, 1471, 1463, 1375, 1286, 1255, 1190, 1145, 1121, 1080, 1065, 1036, 1007 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.08 (3H, s, TBS), 0.11 (3H, s, TBS), 0.87 (9H, s, TBS), 1.31 (3H, s, acetonide), 1.36 (3H, s, acetonide), 1.42 (3H, s, acetonide), 1.44 (3H, s, acetonide), 2.05 (1H, d, *J* = 18.3, C(=O)CH_AH$_B$CH(OTBS)), 2.35 (1H, d, *J* = 18.3 Hz, C(=O)CH_AH$_B$CH), 2.64 (1H, ddd, *J* = 18.3, 9.2, 1.2 Hz, C(=O)CH$_A$$H_B$CH), 2.71 (1H, d, *J* = 9.2 Hz, C*H*CH(OTBS)), 2.86 (1H, ddd, *J* = 18.3, 5.2, 1.2 Hz, C(=O)CH$_A$$H_B$CH(OTBS)), 3.96 (1H, d, *J* = 13.2 Hz, H5a), 4.00-4.07 (2H, m, H4, H5b), 4.27 (1H, d, *J* = 2.3 Hz, H3), 4.35 (1H, s, H2), 4.86 (1H, d, *J* = 5.2 Hz, C*H*(OTBS)); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.8, -4.7, 17.9, 18.6, 25.7, 26.6, 27.5, 28.8, 38.1, 47.4, 52.0, 60.2, 70.8, 72.0, 73.6, 86.3, 97.3, 111.5, 115.3, 218.8; HRMS (ESI-TOF) calcd for

$C_{22}H_{38}NaO_7Si$ $[M+Na]^+$ 465.2279, found 465.2279.



3-41

Methyl ester 3-41 [DK-5-143, DK-5-182]. According to the general procedure D, **3-41** (24 mg, 0.076 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol), methyl propiolate (19 μL, 0.22 mmol) and tris(trimethylsilyl)silane (0.10 mL, 0.33 mmol) in 69% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1): colorless oil; $[\alpha]_D^{25}$ = 18.7 (*c* 1.8, $CHCl_3$); IR (film) 2991, 2939, 1734, 1660, 1437, 1380, 1229, 1205, 1172, 1119, 1073, 1005 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.35 (3H, s, acetonide), 1.36 (3H, s, acetonide), 1.41 (3H, s, acetonide), 1.48 (3H, s, acetonide), 3.72 (3H, s, CO_2CH_3), 4.03 (2H, d, *J* = 1.8 Hz, H5a, H5b), 4.13 (1H, q, *J* = 1.8 Hz, H4), 4.32 (1H, d, *J* = 2.3 Hz, H3), 4.49 (1H, s, H2), 5.93 (1H, d, *J* = 12.4 Hz, CH=C*H*CO_2CH_3), 6.04 (1H, d, *J* = 12.4 Hz, C*H*=$CHCO_2CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 25.8, 27.3, 28.8, 51.6, 60.2, 73.0, 73.2, 88.2, 97.3, 111.9, 112.8, 122.3, 135.0, 167.4; HRMS (ESI-TOF) calcd for $C_{15}H_{22}NaO_7$ $[M+Na]^+$ 337.1258, found 337.1269.

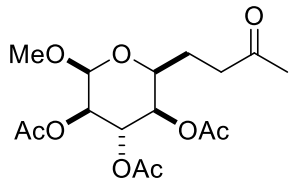


3-42

Hydroxyl amine 3-42 [DK-5-175, DK-5-176]. According to the general procedure D, **3-42** (30 mg, 0.071 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol) and methyl 2-(Benzyloxyimino)ethanate (42 mg, 0.22 mmol) in 65% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1). Inseparable diastereomixture (1:1): Colorless oil; IR (film) 3279, 2990, 2938, 2868, 1745, 1453, 1377, 1212, 1160, 1122, 1078, 1021 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.24, 1.29 (3H, s x2, acetonide), 1.35, 1.36 (3H, s x2, acetonide), 1.40, 1.41 (3H, s x2, acetonide), 1.43, 1.44 (3H, s x2, acetonide), 3.80, 3.81 (3H, s, OMe), 3.92-3.4.10 (4H, m, H5a, H5b, NHC*H*CO_2CH_3, H4), 4.20-4.29 (1H, m, H3), 4.53 (0.5H, s, H2a), 4.59 (0.5H, s, H2b), 4.69 (1H, s, NHOCH_2Ph), 4.74 (0.5H, d, *J* = 11.9 Hz, NHOCH_2Ph), 4.78 (0.5H, d, *J* = 11.9 Hz, NHOCH_2Ph), 7.23-7.42 (5H, m, Ph), N*H* missing; ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 18.9, 26.3, 27.3, 27.6, 28.5, 28.7, 52.2, 59.9, 60.1, 66.1, 68.8, 72.77, 72.84, 73.2, 76.1, 76.3, 85.6, 86.6, 97.3, 97.4, 111.9, 112.4, 113.1, 127.7, 128.21, 128.23, 128.6, 128.7, 137.7, 170.3, 171.3; HRMS (ESI-TOF) calcd for $C_{21}H_{29}NNaO_8$ $[M+Na]^+$ 446.1785, found 446.1803.

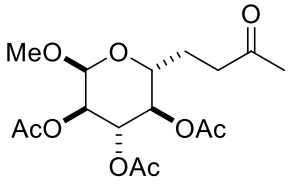
Glucose derivatives 3-43, 3-44 and 3-45 [DK-6-071, DK-6-101]. According to the general procedure D, **3-43** (10 mg, 0.028 mmol), **3-44** (11 mg, 0.031 mmol) and **3-45** (6.3 mg, 0.016 mmol) were synthesized from telluride **3-15** (50 mg, 0.096 mmol) and methyl vinyl ketone (16 μL, 0.19 mmol) in 32%, 29% and 17% yield by using Et_3B (1.03

M in hexane, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (4.8 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 1:1 to 1:2).



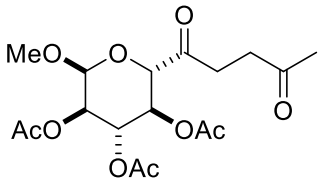
3-43

Compound 3-43. Colorless oil; $[\alpha]_D^{24}$ = 24.3 (*c* 0.52, $CHCl_3$); IR (film) 2961, 2936, 2851, 1745, 1714, 1433, 1371, 1252, 1223, 1159, 1121, 1045 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.75-1.85 (1H, m, CH_AH$_B$CH$_2$C(=O)CH$_3$), 1.90-2.02 (1H, m, CH$_A$$H_BCH_2$C(=O)CH$_3$), 2.12 (6H, s, Me x2), 2.13 (3H, s, Me), 2.16 (3H, s, Me), 2.64 (2H, dd, *J* = 7.3, 7.3 Hz, CH_2C(=O)CH$_3$), 3.54 (3H, s, OMe), 3.92 (1H, ddd, *J* = 10.8, 3.7, 1.8 Hz, H5), 4.64 (1H, d, *J* = 1.8 Hz, H1), 4.74 (1H, m, H4), 4.92 (1H, m, H2), 5.08 (1H, dd, *J* = 3.2, 3.2 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.7, 20.79, 20.85, 24.3, 30.0, 38.9, 57.0, 66.3, 67.3, 68.0, 72.1, 99.0, 168.2, 169.7, 169.8, 207.9; HRMS (ESI-TOF) calcd for $C_{16}H_{24}NaO_9$ $[M+Na]^+$ 383.1313, found 383.1307.



3-44

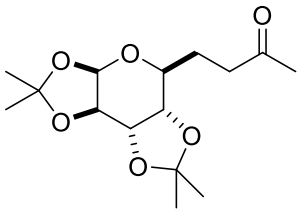
Compound 3-44. Colorless oil; $[\alpha]_D^{24}$ = 134.5 (*c* 0.54, $CHCl_3$); IR (film) 2925, 2849, 1750, 1715, 1435, 1370, 1225, 1168, 1126, 1040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.56-1.72 (1H, m, CH_AH$_B$CH$_2$C(=O)CH$_3$), 1.87-1.96 (1H, m, CH$_A$$H_BCH_2$C(=O)CH$_3$), 1.99 (3H, s, Me), 2.03 (3H, s, Me), 2.07 (3H, s, Me), 2.15 (3H, s, Me), 2.52 (1H, ddd, *J* = 17.8, 8.7, 6.4 Hz, CH_AH$_B$C(=O)CH$_3$), 2.64 (1H, ddd, *J* = 17.8, 9.2, 6.0 Hz, CH$_A$$H_B$C(=O)CH$_3$), 3.36 (3H, s, OMe), 3.78 (1H, td, *J* = 8.7, 2.8 Hz, H5), 4.80-4.89 (3H, m, H1, H2, H4), 5.42 (1H, dd, *J* = 10.8, 10.8 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.9, 20.72, 20.74, 24.8, 29.8, 38.7, 55.3, 67.4, 70.0, 71.1, 71.9, 96.5, 169.9, 170.0, 170.2, 207.8; HRMS (ESI-TOF) calcd for $C_{16}H_{24}NaO_9$ $[M+Na]^+$ 383.1313, found 383.1299.



3-45

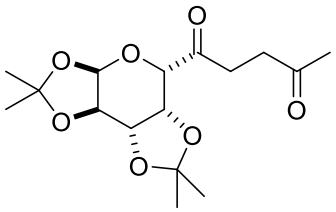
Compound 3-45. Colorless oil; $[\alpha]_D^{24}$ = 66.8 (*c* 0.32, $CHCl_3$); IR (film) 2920, 2850, 1752, 1716, 1369, 1223, 1167, 1128, 1045 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.00 (3H, s, Me), 2.01 (3H, s, Me), 2.08 (3H, s, Me), 2.18 (3H, s, Me), 2.62 (1H, dt, *J* = 18.4, 5.8 Hz, CH_AH$_B$CH$_2$C(=O)CH$_3$), 2.75-2.96 (3H, m, CH$_A$$H_BCH_2$C(=O)CH$_3$), 3.45 (3H, s, OMe), 4.18 (1H, d, *J* = 10.3 Hz, H5), 4.87 (1H, dd, *J* = 10.3, 3.5 Hz, H2), 5.03 (1H, d, *J* = 3.4 Hz, H1), 5.10 (1H, dd, *J* = 10.3, 10.3 Hz, H4), 5.50 (1H, dd, *J* = 10.3, 10.3 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.6, 20.67, 20.70, 29.8, 32.2, 36.5, 55.9, 69.0, 69.4, 70.6, 72.7, 96.9, 169.8, 169.9, 170.2, 204.5, 206.6; HRMS (ESI-TOF) calcd for $C_{17}H_{24}NaO_{10}$ $[M+Na]^+$ 411.1262, found 411.1253.

Galactose derivatives 3-46 and 3-47 [DK-4-058, DK-6-083]. According to the general procedure D, **3-46** (14 mg, 0.047 mmol) and **3-47** (10 mg, 0.10 mmol) were synthesized from telluride **3-17** (100 mg, 0.22 mmol) and methyl vinyl ketone (35 μL, 0.43 mmol) in 21% and 45% yield by using Et_3B (1.03 M in hexane, 0.63 mL, 0.65 mmol) in CH_2Cl_2 (11 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 4:1 to 2:1).



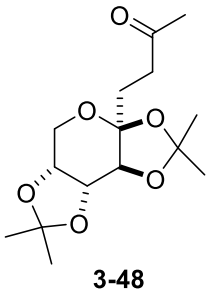
3-46

Compound 3-46. Colorless oil; $[\alpha]_D^{26}$ = -93.2 (*c* 0.70, $CHCl_3$); IR (film) 2987, 2935, 1716, 1371, 1238, 1223, 1167, 1140, 1096, 1065, 1042 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, s, acetonide), 1.37 (3H, s, acetonide), 1.44 (3H, s, acetonide), 1.50 (3H, s, acetonide), 1.62-1.73 (1H, m, CH_AH$_B$CH$_2$C(=O)CH$_3$), 2.01-2.12 (1H, m, CH$_A$$H_BCH_2$C(=O)CH$_3$), 2.13 (3H, s, C(=O)C$H_3$), 2.55 (1H, ddd, *J* = 17.9, 7.8, 7.8 Hz, CH_AH$_B$C(=O)CH$_3$), 2.67 (1H, ddd, *J* = 17.8, 8.2, 6.0 Hz, CH$_A$$H_B$C(=O)CH$_3$), 3.18 (1H, ddd, *J* = 9.1, 9.1, 3.6 Hz, H5), 3.85 (1H, dd, *J* = 9.6, 5.5 Hz, H4), 4.20 (1H, d, *J* = 1.8 Hz, H2), 4.49 (1H, d, *J* = 5.0 Hz, H3), 5.17 (1H, d, *J* = 2.3 Hz, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.7, 25.89, 25.93, 27.9 (2 peaks), 29.9, 38.9, 71.8, 73.7, 74.3, 75.8, 96.8, 108.7, 110.7, 208.2 ; HRMS (ESI-TOF) calcd for $C_{15}H_{24}NaO_6$ $[M+Na]^+$ 323.1465, found 323.1456.



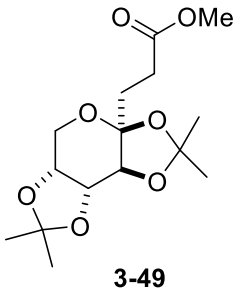
3-47

Compound 3-47. Colorless oil; $[\alpha]_D^{26}$ = -118.3 (*c* 1.2, $CHCl_3$); IR (film) 2988, 2935, 1715, 1380, 1256, 1212, 1169, 1068, 1005 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.29 (3H, s, acetonide), 1.32 (3H, s, acetonide), 1.43 (3H, s, acetonide), 1.50 (3H, s, acetonide), 2.18 (3H, s, C(=O)CH_3), 2.60-2.83 (3H, m, C(=O)CH_2CH_AH$_B$C(=O)), 2.98-3.10 (1H, m, C(=O)CH$_2$CH$_A$$H_B$C(=O)), 4.22 (1H, d, *J* = 2.3 Hz, H5), 4.34 (1H, dd, *J* = 5.0, 2.3 Hz, H2), 4.55 (1H, dd, *J* = 7.8, 2.3 Hz, H3), 4.62 (1H, dd, *J* = 7.8, 2.3 Hz, H4), 5.63 (1H, d, *J* = 5.0 Hz, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.2, 24.8, 25.8, 25.9, 29.9, 34.1, 36.3, 70.3, 70.5, 72.3, 73.6, 96.3, 108.9, 109.6, 207.1, 207.8; HRMS (ESI-TOF) calcd for $C_{16}H_{24}NaO_7$ $[M+Na]^+$ 351.1414, found 351.1405.



3-48

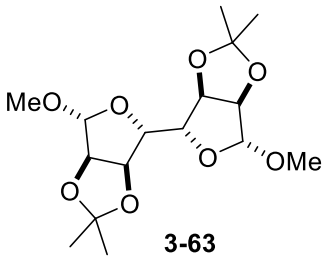
Fructose derivative 3-48 [DK-6-085]. According to the general procedure D, **3-48** (23 mg, 0.077 mmol) was synthesized from telluride **3-19** (50 mg, 0.11 mmol) and methyl vinyl ketone (18 μL, 0.22 mmol) in 70% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{24}$ = -9.8 (*c* 1.1, $CHCl_3$); IR (film) 2987, 2936, 1717, 1442, 1376, 1316, 1253, 1211, 1168, 1105, 1078, 1040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.33 (6H, s, acetonide x2), 1.48 (3H, s, acetonide), 1.49 (3H, s, acetonide), 1.97 (1H, ddd, *J* = 14.2, 10.1, 5.5 Hz, $CH_3C(=O)CH_2CH_AH_B$), 2.08-2.20 (4H, m, $CH_3C(=O)CH_2CH_AH_B$), 2.71 (1H, ddd, *J* = 18.8, 10.1, 5.5 Hz, $CH_3C(=O)CH_AH_B$), 2.83 (1H, ddd, *J* = 18.8, 10.1, 5.5 Hz, $CH_3C(=O)CH_AH_B$), 3.68 (1H, d, *J* = 13.3 Hz, H5a), 3.81 (1H, dd, *J* = 13.3, 1.8 Hz, H5b), 4.10 (1H, d, *J* = 2.7 Hz, H2), 4.19 (1H, dd, *J* = 8.2, 1.4 Hz, H4), 4.53 (1H, dd, *J* = 8.2, 2.3 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.9, 24.9, 25.8, 26.3, 29.9, 34.5, 37.6, 60.8, 70.4, 70.6, 73.8, 103.5, 107.5, 108.8, 208.5; HRMS (ESI-TOF) calcd for $C_{15}H_{24}NaO_6$ $[M+Na]^+$ 323.1465, found 323.1465.



3-49

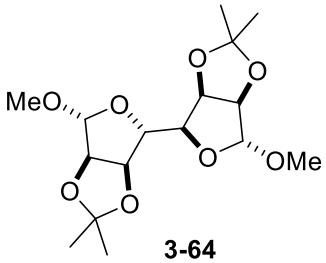
Fructose derivative 3-49 [DK-6-086, DK-6-088]. According to the general procedure D, **3-49** (26 mg, 0.082 mmol) was synthesized from telluride **3-19** (50 mg, 0.11 mmol), methyl acrylate (19 μL, 0.22 mmol) and tris(trimethylsilyl)silane (0.10 mL, 0.33 mmol) in 75% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1): colorless oil; $[\alpha]_D^{18}$ = -12.8 (*c* 1.1, $CHCl_3$); IR (film) 2987, 2938, 2904, 1739, 1442, 1378, 1317, 1253, 1211, 1170, 1105, 1075 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, acetonide), 1.33 (3H, s, acetonide), 1.47 (3H, s, acetonide), 1.49 (3H, s, acetonide), 2.04 (1H, ddd, *J* = 14.2, 10.1, 6.0 Hz, $CH_AH_BCH_2CO_2Me$), 2.18 (1H, ddd, *J* = 14.2, 10.1, 6.0 Hz, $CH_AH_BCH_2CO_2Me$), 2.56 (1H, ddd, *J* = 16.0, 10.1, 6.0 Hz, $CH_AH_BCO_2Me$), 2.65 (1H, ddd, *J* = 16.0, 10.1, 6.0 Hz, $CH_AH_BCO_2Me$), 3.65 (3H, s, CO_2Me), 3.68 (1H, d, *J* = 13.3 Hz, H5a), 3.80 (1H, dd, *J* = 13.3, 1.8 Hz, H5b), 4.11 (1H, d, *J* = 2.7 Hz, H2), 4.19 (1H, dd, *J* = 8.2, 1.4 Hz, H4), 4.53 (1H, dd, *J* = 8.2, 2.8 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.0, 24.9, 25.7, 26.3, 28.2, 35.8, 51.5, 60.9, 70.4, 70.6, 73.7, 103.3, 107.5, 108.9, 174.1; HRMS (ESI-TOF) calcd for $C_{15}H_{24}NaO_7$ $[M+Na]^+$ 339.1414, found 339.1424.

General procedure E: **Dimer 3-63 and 3-64 [DK-5-108].** To a solution of acyl telluride **3-9** (100 mg, 0.25 mmol) in CH_2Cl_2 (1.2 mL) were added Et_3B (1.03 mol/L hexane solution, 1.2 mL) at room temperature. The reaction mixture was stirred for 15 min at rt, and then the reaction mixture was concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1) to give **3-63** (16 mg, 0.046 mmol) in 37% yield and **3-64** (17 mg, 0.049 mmol) in 39% yield.



3-63

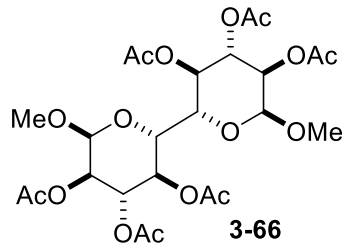
Dimer 3-63. Colorless oil; $[\alpha]_D^{24}$ = -53.8 (*c* 2.2, $CHCl_3$); IR (film) 2986, 2935, 2834, 1447, 1377, 1271, 1242, 1210, 1161, 1094, 1052 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30 (6H, s, acetonide), 1.47 (6H, s, acetonide), 3.40 (6H, s, OMe), 4.04 (2H, s, H4), 4.58 (2H, d, *J* = 6.0 Hz, H3), 4.60 (2H, d, *J* = 6.0 Hz, H2), 5.00 (2H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.7, 26.3, 54.7, 81.3, 85.1, 88.0, 108.9, 112.5; HRMS (ESI-TOF) calcd for $C_{16}H_{26}NaO_8$ $[M+Na]^+$ 369.1520, found 369.1531.



3-64

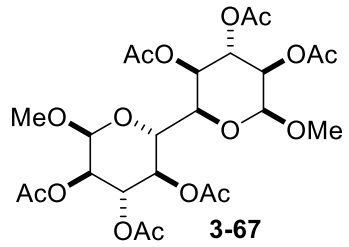
Dimer 3-64. Colorless solid; $[\alpha]_D^{24}$ = -71.4 (*c* 2.2, $CHCl_3$); m.p. 95-100 °C; IR (film) 2987, 2939, 2834, 1456, 1375, 1271, 1210, 1163, 1100, 1032 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, acetonide), 1.33 (3H, s, acetonide), 1.49 (6H, s, acetonide), 3.31 (3H, s, OMe-a), 3.36 (3H, s, OMe-b), 3.87 (1H, dd, *J* = 10.1, 3.2 Hz, H4a), 4.47 (1H, d, *J* = 10.1 Hz, H4b), 4.57 (1H, d, *J* = 6.0 Hz, H2a), 4.62 (1H, d, *J* = 6.0 Hz, H2b), 4.72 (1H, dd, *J* = 6.0, 3.7 Hz, H3a), 4.86 (1H, d, *J* = 6.0 Hz, H3b), 4.89 (1H, s, H1a), 5.00 (1H, s, H1b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.7, 25.0, 25.9, 26.5, 54.7, 55.3, 79.3, 79.9, 82.7, 84.2, 85.2, 85.4, 107.6, 109.6, 112.5, 112.8; HRMS (ESI-TOF) calcd for $C_{16}H_{26}NaO_8$ $[M+Na]^+$ 369.1520, found 369.1535.

Glucose derivatives 3-66, 3-67 and 3-68 [DK-6-073, DK-6-102]. According to the general procedure E, **3-66** (17 mg, 0.029 mmol), **3-67** (15 mg, 0.026 mmol) and **3-68** (5.9 mg, 0.010 mmol) were synthesized from telluride **3-15** (100 mg, 0.19 mmol) in 31%, 27% and 11% yield by using Et_3B (1.03 M in hexane, 0.93 mL, 0.96 mmol) in CH_2Cl_2 (0.96 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1 to 1:4).



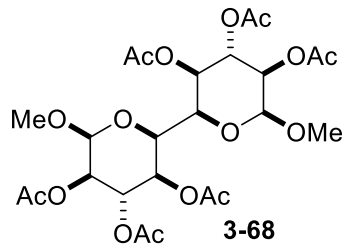
3-66

Dimer 3-66. Colorless oil; $[\alpha]_D^{25}$ = 114.3 (*c* 0.83, $CHCl_3$); IR (film) 3021, 2964, 2941, 2848, 1752, 1433, 1369, 1224, 1169, 1120, 1038 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.00 (6H, s, Me), 2.06 (12H, s, Me), 3.32 (6H, s, OMe), 3.89 (2H, d, *J* = 9.6 Hz, H5a, H5b), 4.90 (2H, dd, *J* = 10.5, 3.7 Hz, H2a, H2b), 4.98 (2H, d, *J* = 3.7 Hz, H1a, H1b), 5.29 (2H, dd, *J* = 10.0, 10.0 Hz, H4a, H4b), 5.43 (2H, dd, *J* = 10.5, 10.5 Hz, H3a, H3b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.67, 20.70, 20.73, 56.1, 65.4, 68.0, 70.2, 70.6, 97.2, 169.6, 170.0, 170.4; HRMS (ESI-TOF) calcd for $C_{24}H_{34}NaO_{16}$ $[M+Na]^+$ 601.1739, found 601.1759.



3-67

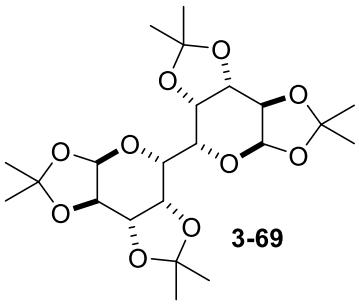
Dimer 3-67. Colorless oil; $[\alpha]_D^{25}$ = 95.5 (*c* 0.74, $CHCl_3$); IR (film) 3018, 2939, 2849, 1748, 1432, 1371, 1226, 1173, 1130, 1046 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.01 (3H, s, Me), 2.04 (3H, s, Me), 2.06 (3H, s, Me), 2.11 (6H, s, Me x2), 2.14 (3H, s, Me), 3.33 (3H, s, OMe), 3.46 (3H, s, OMe), 4.02-4.10 (2H, m, H5a, H5b), 4.55 (1H, d, *J* = 1.7 Hz, H1b), 4.80 (1H, dd, *J* = 10.3, 3.5 Hz, H2a), 4.85 (1H, d, *J* = 3.5 Hz, H1a), 4.95 (2H, m, H2b, H4b), 5.10 (1H, dd, *J* = 2.9, 2.9 Hz, H3b), 5.16 (1H, dd, *J* = 9.2, 9.2 Hz, H4a), 5.49 (1H, dd, *J* = 10.3, 10.3 Hz, H3a); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.66, 20.69, 20.80, 20.83, 20.9, 21.1, 55.7, 57.5, 64.9, 65.4 (2 peaks), 67.2, 69.5, 70.7, 72.0, 75.6, 96.6, 99.8, 168.1, 169.4, 169.5, 169.8, 170.2, 170.5; HRMS (ESI-TOF) calcd for $C_{24}H_{34}NaO_{16}$ $[M+Na]^+$ 601.1739, found 601.1717.



3-68

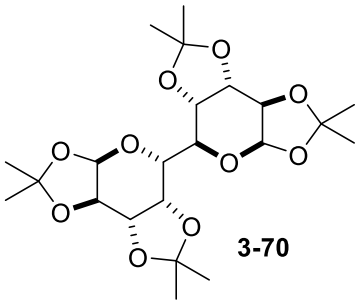
Dimer 3-68. Colorless oil; $[\alpha]_D^{25}$ = 28.8 (*c* 0.30, $CHCl_3$); IR (film) 3006, 2959, 2925, 2849, 1752, 1444, 1371, 1221, 1160, 1128, 1044 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.08 (6H, s, Me), 2.11 (6H, s, Me), 2.14 (6H, s, Me), 3.58 (6H, s, OMe), 4.22 (2H, s, H5a, H5b), 4.70-4.75 (4H, m, H1a, H1b, H4a, H4b), 5.01 (2H, dd, *J* = 1.8, 1.8 Hz, H2a, H2b), 5.09 (2H, dd, *J* = 3.2, 3.2 Hz, H3a, H3b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.6 (2 peaks), 20.9, 57.2, 65.0, 65.7, 67.5, 72.3, 98.9, 167.6, 169.2, 169.4; HRMS (ESI-TOF) calcd for $C_{24}H_{34}NaO_{16}$ $[M+Na]^+$ 601.1739, found 601.1711.

Galactose derivatives 3-69, 3-70 and 3-71 [DK-5-126, DK-6-089]. According to the general procedure E, **3-69** (11 mg, 0.024 mmol), **3-70** (22 mg, 0.048 mmol) and **3-71** (11 mg, 0.024 mmol) were synthesized from telluride **3-17** (100 mg, 0.22 mmol) in 22%, 44% and 22% yield by using Et_3B (1.03 M in hexane, 1.1 mL, 1.1 mmol) in CH_2Cl_2 (1.1 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1 to 4:1).



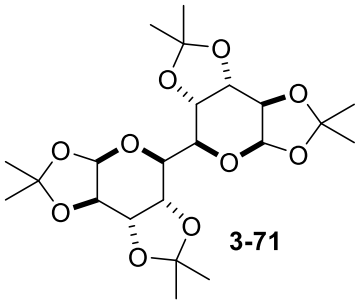
3-69

Dimer 3-69. Colorless solid; $[\alpha]_D^{26}$ = -158.5 (*c* 0.55, $CHCl_3$); m.p. 140-145 °C; IR (film) 2986, 2935, 2898, 1382, 1371, 1238, 1222, 1166, 1124, 1099, 1067, 1032 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.36 (6H, s, acetonide), 1.37 (6H, s, acetonide), 1.43 (6H, s, acetonide), 1.47 (6H, s, acetonide), 3.43 (2H, d, *J* = 9.6 Hz, H5a, H5b), 4.19-4.22 (2H, m, H2a, H2b), 4.44 (2H, dd, *J* = 9.6, 5.5 Hz, H4a, H4b), 4.55 (2H, dd, *J* = 5.5, 1.4 Hz, H3a, H3b), 5.29 (2H, d, *J* = 2.3 Hz, H1a, H1b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.9, 26.2, 27.9, 28.0, 68.2, 69.5, 74.3, 75.8, 97.2, 108.8, 111.1; HRMS (ESI-TOF) calcd for $C_{22}H_{34}NaO_{10}$ $[M+Na]^+$ 481.2044, found 481.2065.

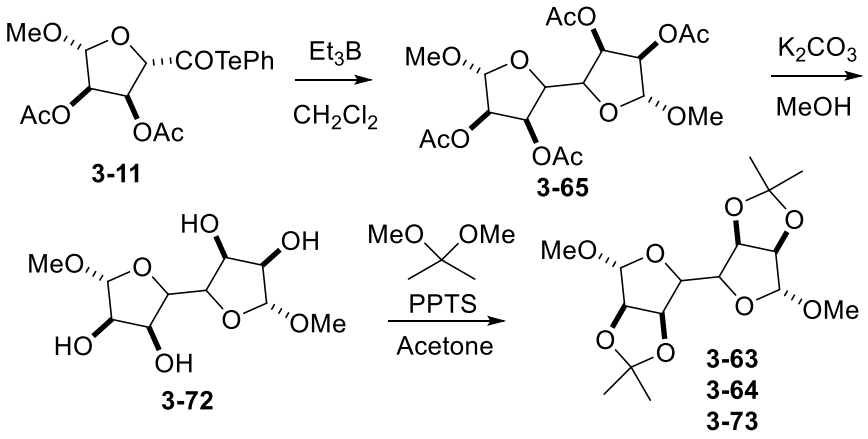


3-70

Dimer 3-70. Colorless solid; $[\alpha]_D^{26}$ = -72.1 (*c* 1.1, $CHCl_3$); m.p. 148-153 °C; IR (film) 2986, 2937, 2907, 1458, 1378, 1250, 1214, 1170, 1108, 1065, 1002 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (3H, s, acetonide), 1.33-1.38 (9H, m, acetonide), 1.44 (3H, s, acetonide), 1.45 (3H, s, acetonide), 1.50 (3H, s, acetonide), 1.53 (3H, s, acetonide), 3.85-3.92 (2H, m, H5a, H5b), 4.19 (1H, dd, *J* = 4.1, 1.4 Hz, H2a), 4.29 (1H, dd, *J* = 5.0, 2.3 Hz, H2b), 4.33 (1H, ddd, *J* = 6.9, 3.2, 1.8 Hz, H4a), 4.49 (1H, dd, *J* = 6.8, 1.4 Hz, H3a), 4.55 (1H, d, *J* = 8.2 Hz, H4b), 4.59 (1H, dd, *J* = 8.2, 2.3 Hz, H3b), 5.41 (1H, d, *J* = 4.1 Hz, H1a), 5.56 (1H, d, *J* = 5.0 Hz, H1b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.6, 24.9, 25.2, 25.3, 26.0, 26.1, 27.1, 27.3, 67.8, 70.2, 70.5, 70.6, 71.1, 71.5, 72.6, 74.6, 95.8, 96.4, 108.59, 108.64, 109.3, 109.4; HRMS (ESI-TOF) calcd for $C_{22}H_{34}NaO_{10}$ $[M+Na]^+$ 481.2044, found 481.2034.



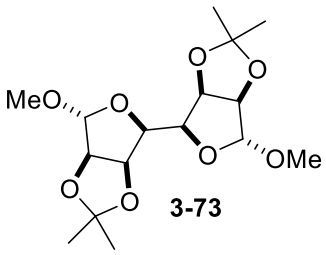
Dimer 3-71. Colorless oil; $[\alpha]_D^{26}$ = -78.4 (*c* 0.53, $CHCl_3$); IR (film) 2986, 2936, 2902, 2361, 1458, 1381, 1253, 1213, 1169, 1113, 1070 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (6H, s, acetonide), 1.33 (6H, s, acetonide), 1.44 (6H, s, acetonide), 1.53 (6H, s, acetonide), 3.96 (2H, s, H5a, H5b), 4.33 (2H, dd, *J* = 5.5, 2.8 Hz, H2a, H2b), 4.41 (2H, d, *J* = 7.8 Hz, H4a, H4b), 4.62 (2H, dd, *J* = 7.8, 2.8 Hz, H3a, H3b), 5.62 (2H, d, *J* = 5.5 Hz, H1a, H1b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.0, 25.1, 25.8, 26.1, 67.4, 70.0, 71.05, 71.12, 96.7, 108.6, 109.4; HRMS (ESI-TOF) calcd for $C_{22}H_{34}NaO_{10}$ $[M+Na]^+$ 481.2044, found 481.2064.



Dimer 3-73 [DK-6-074, DK-6-100]. According to the general procedure E, inseparable mixture **3-65** (19 mg, 0.044 mmol) was synthesized from telluride **3-11** (45 mg, 0.10 mmol) in 88% yield by using Et_3B (1.03 M in hexane, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (0.50 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 2:1).

To a solution of **3-65** (35 mg, 0.081 mmol) in methanol (0.81 mL) was added K_2CO_3 (5.6 mg, 0.040 mmol) and the reaction stirred at room temperature for 4 h. Reaction mixture was passed through a short pad of silica gel ($CHCl_3$/MeOH 4:1) to afford tetraol **3-72**.

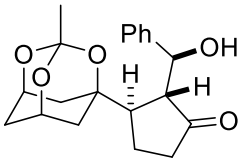
A mixture of the above tetraol **3-72** and PPTS (20 mg, 0.081 mmol) in a mixture of dimethoxypropane (1.6 mL) and acetone (1.6 mL) was stirred at room temperature for 23 h, and then concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 5:1) to afford dimer **3-63** (10 mg, 0.029 mmol), **3-64** (13 mg, 0.038 mmol) and **3-73** (3.3 mg, 0.0095 mmol) in 36%, 48% and 12% yield each.



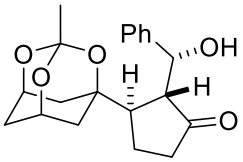
3-73

Dimer 3-73. Colorless solid; $[\alpha]_D^{24}$ = -49.8 (*c* 0.17, $CHCl_3$); m.p. 115-120 °C; IR (film) 2986, 2934, 2835, 1456, 1374, 1210, 1164, 1115, 1094, 1054, 1013 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (6H, s, acetonide), 1.45 (6H, s, acetonide), 3.38 (6H, s, OMe), 4.15 (2H, dd, *J* = 1.8, 1.8 Hz, H4), 4.56 (2H, d, *J* = 6.4 Hz, H2), 4.90 (2H, ddd, *J* = 5.5, 1.8, 1.8 Hz, H3), 4.98 (2H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 26.1, 55.0, 79.7, 79.9, 84.8, 107.5, 112.4; HRMS (ESI-TOF) calcd for $C_{16}H_{26}NaO_8$ $[M+Na]^+$ 369.1520, found 369.1521.

6-3. 第四章の実験項



4-14



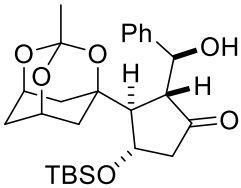
4-15

General procedure F: **Compounds 4-14 and 4-15 [DK-2-190].** Et_3B (1.03 M in hexane, 0.28 mL, 0.29 mmol) was added to a solution of O,Te-acetal **2-8** (35 mg, 0.097 mmol), cyclopentenone (24 µL, 0.29 mmol) and benzaldehyde (30 µL, 0.30 mmol) in CH_2Cl_2 (0.97 mL) at 0 °C. The reaction mixture was stirred at 0 °C for 15 min under air, and was then directly subjected to flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1) to afford **4-14** (27 mg, 0.078 mmol) and **4-15** (3.0 mg, 8.7 µmol) in 80% and 9% yields, respectively.

4-14: colorless solid; m.p. 140-143 °C; IR (film) 3421, 2957, 1736, 1451, 1395, 1325, 1299, 1128, 1050 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.49-15.8 (1H, m, C(=O)$CH_2C\mathit{H}_A\mathrm{H}_B$), 1.56 (1H, dd, *J* = 12.8, 1.4 Hz, $COC\mathit{H}_{ax}\mathrm{H}_{eq}CHO$), 1.57 (3H, s, $C\mathit{H}_3$), 1.59 (1H, dt, *J* = 12.8, 1.4 Hz, $COC\mathit{H}_{ax}\mathrm{H}_{eq}CHO$), 1.65 (1H, dt, *J* = 12.8 Hz, 1.4 Hz, $COC\mathit{H}_{ax}\mathrm{H}_{eq}CHO$), 1.73-1.89 (2H, m, C(=O)$C\mathit{H}_A\mathrm{H}_B$, C(=O)$CH_2CH_A\mathit{H}_B$), 2.07-2.19 (2H, m, $COCH_{ax}\mathit{H}_{eq}CHO$, C(=O)CHC*H*), 2.26-2.40 (2H, m, $COCH_{ax}\mathit{H}_{eq}CHO$, C(=O)$CH_A\mathit{H}_B$), 2.57 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, $COCH_{ax}\mathit{H}_{eq}CHO$), 2.87 (1H, dd, *J* = 8.2, 3.2 Hz, C(=O)C*H*CH), 4.44 (1H, m, $CH_2C\mathit{H}OCH_2$), 4.48 (1H, m, $CH_2C\mathit{H}OCH_2$), 4.77 (1H, d, *J* = 8.7 Hz, O*H*), 5.30 (1H, m, C*H*OH), 7.20-7.34 (5H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.7, 25.9, 31.2, 32.2, 36.3, 38.6, 46.6, 56.5, 67.8, 67.9, 72.9, 75.0, 110.4, 126.7, 127.3, 128.1, 141.6, 217.4; HRMS (ESI-TOF) calcd for $C_{20}H_{24}NaO_5$ $[M+Na]^+$ 367.1516, found 367.1523.

4-15: colorless solid; m.p. 141-144 °C; IR (film) 3443, 2954, 2929, 1733, 1450, 1395, 1324, 1298, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30 (1H, d, *J* = 13.7 Hz,

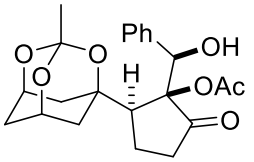
$COC\mathit{H}_{ax}\mathrm{H}_{eq}CHO$), 1.39 (1H, d, *J* = 13.7 Hz, $COC\mathit{H}_{ax}\mathrm{H}_{eq}CHO$), 1.40 (3H, s, $C\mathit{H}_3$), 1.50 (1H, d, *J* = 13.3 Hz, $COC\mathit{H}_{ax}\mathrm{H}_{eq}CHO$), 1.74-1.92 (2H, m, C(=O)$CH_2C\mathit{H}_2$), 1.93-2.10 (2H, m, $COCH_{ax}\mathit{H}_{eq}CHO$, C(=O)$C\mathit{H}_A\mathrm{H}_B$), 2.16 (1H, dt, *J* = 7.8, 7.3 Hz, C(=O)CHC*H*), 2.20 (1H, m, $COCH_{ax}\mathit{H}_{eq}CHO$), 2.32 (1H, dt, *J* = 18.3, 7.3 Hz, C(=O)$CH_A\mathit{H}_B$), 2.48 (1H, m, $COCH_{ax}\mathit{H}_{eq}CHO$), 2.72 (1H, t, *J* = 6.0 Hz, C(=O)C*H*CH), 3.69 (1H, d, *J* = 6.9 Hz, O*H*), 4.33 (1H, m, $CH_2C\mathit{H}OCH_2$), 4.39 (1H, m, $CH_2C\mathit{H}OCH_2$), 5.26 (1H, m, C*H*OH), 7.22-7.38 (5H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.9, 26.0, 32.3, 32.8, 35.4, 39.1, 46.2, 54.4, 67.8, 67.9, 74.3, 74.7, 110.2, 126.6, 127.6, 128.3, 142.0, 221.8; HRMS (ESI-TOF) calcd for $C_{20}H_{24}NaO_5$ $[M+Na]^+$ 367.1516, found 367.1518.



4-16

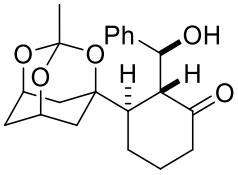
Compound 4-16 [DK-2-192]. According to the general procedure F, **4-16** (41 mg, 0.086 mmol) was synthesized from O,Te-acetal **2-8** (36 mg, 0.10 mmol) 4-(*tert*-butyldimethylsilyloxy)-2-cyclopentenone (63 mg, 0.30 mmol), and

benzaldehyde (30 μL, 0.30 mmol) in 86% yield by using Et_3B (1.03 M in hexane, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (0.99 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 4:1 to 3:1): colorless solid; m.p. 128-132 °C; IR (film) 3464, 2953, 2857, 1729, 1394, 1325, 1299, 1254, 1127, 1057 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.10 (3H, s, C*H*$_3$ of TBS), 0.13 (3H, s, C*H*$_3$ of TBS), 0.65 (1H, dd, *J* = 13.3, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.93 (9H, s, *t*-Bu of TBS), 1.30 (1H, dd, *J* = 12.8, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.34 (3H, s, C*H*$_3$), 1.40 (1H, dt, *J* = 13.3, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.84 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 1.88 (1H, br s, C(=O)CHC*H*), 2.05 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.31 (1H, d, *J* = 17.8 Hz, C(=O)C*H*$_A$H$_B$CH(OTBS)), 2.40 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, CHOCH$_{ax}$*H*$_{eq}$CHO), 2.57 (1H, dd, *J* = 9.2, 1.8 Hz, C(=O)C*H*CH), 2.72 (1H, ddd, *J* = 17.3, 4.6, 0.9 Hz, C(=O)CH$_A$*H*$_B$CH(OTBS)), 4.22 (1H, m, CH$_2$C*H*OCH$_2$), 4.31 (1H, m, CH$_2$C*H*OCH$_2$), 4.63 (1H, d, *J* = 4.6 Hz, C*H*(OTBS)), 4.74 (1H, s, O*H*), 4.92 (1H, d, *J* = 9.2 Hz, C*H*OH), 7.24-7.30 (1H, m, aromatic), 7.31-7.40 (4H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ -4.7, -4.6, 17.9, 25.8, 25.9, 32.0, 33.7, 34.6, 49.6, 53.0, 57.4, 67.6, 67.7, 69.2, 72.8, 75.9, 110.2, 127.4, 128.0, 128.3, 141.1, 220.7; HRMS (ESI-TOF) calcd for $C_{26}H_{38}NaO_6Si$ $[M+Na]^+$ 497.2330, found 497.2323.



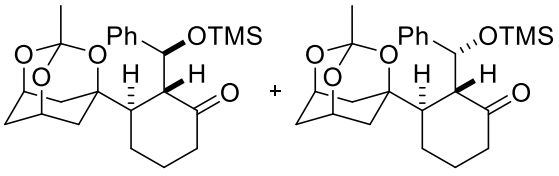
4-17

General procedure G: **Compound 4-17 [DK-3-066]**. Et_3B (1.03 M in hexane, 0.43 mL, 0.44 mmol) was added to a solution of O,Te-acetal **2-8** (53 mg, 0.15 mmol), 2-acetoxy-2-cyclopentenone (41 mg, 0.29 mmol), and benzaldehyde (75 μL, 0.74 mmol) in CH_2Cl_2 (1.5 mL) at 0 °C. The reaction mixture was stirred under air at 0 °C for 15 min, and at room temperature for 1 h. Then the reaction mixture was directly subjected to flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1) to afford **4-17** (52 mg, 0.13 mmol) in 87% yield: colorless solid; m.p. 164-166 °C; IR (film) 3422, 2956, 1745, 1396, 1370, 1326, 1296, 1237, 1128 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 0.88 (1H, d, *J* = 11.9 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.91 (1H, d, *J* = 12.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.07 (1H, dd, *J* = 12.8, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.20 (1H, dt, *J* = 11.4, 8.2 Hz, C(=O)CH$_2$C*H*$_A$H$_B$), 1.46 (1H, ddd, *J* = 17.8, 11.9, 9.2 Hz, C(=O)C*H*$_A$H$_B$CH$_2$), 1.63 (3H, s, C*H*$_3$), 1.67 (3H, s, Ac), 2.07-2.31 (4H, m, COCH$_{ax}$*H*$_{eq}$CHO x3, C(=O)CH$_2$CH$_A$*H*$_B$), 2.37 (1H, dd, *J* = 12.4, 7.8 Hz, C(=O)CC*H*), 2.51 (1H, dd, *J* = 17.8, 7.8 Hz, C(=O)CH$_A$*H*$_B$CH$_2$), 3.94-4.07 (3H, m, O*H*, CH$_2$C*H*OCH$_2$ x2), 5.29 (1H, d, *J* = 3.2 Hz, C*H*OH), 7.06 (1H, t, *J* = 7.3 Hz, aromatic), 7.13-7.19 (2H, m, aromatic), 7.49 (2H, d, *J* = 7.8 Hz, aromatic); ^{13}C NMR (100 MHz, $CO(CD_3)_2$) δ 20.8, 21.1, 26.8, 33.1, 36.4, 36.5, 38.9, 50.1, 68.85, 68.90, 74.9, 77.3, 83.9, 111.1, 128.0, 128.2, 129.4, 140.8, 169.5, 212.0; HRMS (ESI-TOF) calcd for $C_{22}H_{26}NaO_7$ $[M+Na]^+$ 425.1571, found 425.1563.



4-18

Compound 4-18 [DK-3-109]. According to the general procedure F, **4-18** (9.2 mg, 0.026 mmol) and **2-19** (16 mg, 0.063 mmol) were synthesized from O,Te-acetal **2-8** (35 mg, 0.097 mmol), cyclohexanone (19 µL, 0.20 mmol), and benzaldehyde (50 µL, 0.49 mmol) in 27% and 65% yields, respectively, by using Et_3B (1.03 M in hexane, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.98 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless solid; m.p. 128-130 °C; IR (film) 3427, 2952, 1706, 1449, 1394, 1322, 1301, 1129 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 0.66 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 0.70 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 0.73 (1H, dt, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.20-1.46 (4H, m, C(=O)CH_2CH_2CH_2), 1.57 (3H, s, CH_3), 1.67-1.75 (1H, m, C(=O)CHCH), 1.79 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, $COCH_{ax}H_{eq}$CHO), 1.93 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz,, $COCH_{ax}H_{eq}$CHO), 2.15 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, $COCH_{ax}H_{eq}$CHO), 2.22-2.37 (2H, m, C(=O)C$H_2$$CH_2$), 2.87 (1H, d, *J* = 4.1 Hz, OH), 3.03 (1H, d, *J* = 8.7 Hz, C(=O)CHCH), 3.89-3.97 (2H, m, CH_2CHOCH_2 x2), 4.86 (1H, dd, *J* = 8.7, 4.1 Hz, CHOH), 7.08 (1H, tt, *J* = 7.3, 1.4 Hz, aromatic), 7.13 (2H, m, aromatic), 7.25 (2H, dd, *J* = 7.3, 1.4 Hz, aromatic); ^{13}C NMR (100 MHz, C_6D_6) δ 21.5, 22.2, 26.4, 32.4, 33.6, 35.1, 39.3, 45.6, 56.8, 67.87, 67.91, 74.9, 75.5, 110.7, 127.4, 128.57, 128.59, 143.1, 211.0; HRMS (ESI-TOF) calcd for $C_{21}H_{26}NaO_5$ $[M+Na]^+$ 381.1672, found 381.1676.



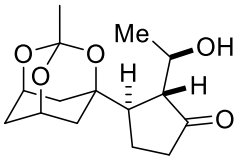
4-19 **4-20**

Compounds 4-19 and 4-20 [DK-4-181]. Et_3B (1.03 M in hexane, 0.26 mL, 0.27 mmol) was added to a solution of O,Te-acetal **2-8** (32 mg, 0.089 mmol), cyclohexenone (17 µL, 0.18 mmol) and benzaldehyde (45 µL, 0.43 mmol) in CH_2Cl_2 (0.89 mL) at 0 °C. The reaction mixture was stirred at 0 °C for 15 min under air, and then TMS-imidazole (0.13 mL, 0.89 mmol) and 4-dimethylaminopyridine (DMAP, 3.3 mg, 0.027 mmol) were successively added. The reaction mixture was stirred at 0 °C for 3 h, and then saturated aqueous $NaHCO_3$ (10 mL) was added. The resultant mixture was extracted with EtOAc (10 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 3:1) to afford **4-19** (17.2 mg, 0.039 mmol) and **4-20** (11 mg, 0.026 mmol) in 44% and 29% yields, respectively.

4-19 : colorless oil; IR (film) 2953, 1710, 1449, 1394, 1322, 1297, 1251, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ -0.01 (9H, s, CH_3 of TMS), 0.77 (1H, d, *J* = 12.8 Hz, COC$H_{ax}$$H_{eq}$CHO), 0.83 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO) 0.97 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.27-1.54 (2H, m, C(=O)$CH_2$$CH_2CH_2$), 1.60 (3H, s, C$H_3$), 1.67-1.85 (4H, m, C(=O)CH_2CH_2, $COCH_{ax}H_{eq}$CHO, C(=O)CHCH), 1.99 (1H, m, $COCH_{ax}H_{eq}$CHO),

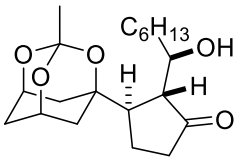
2.17 (1H, m, $COCH_{ax}H_{eq}CHO$), 2.34 (1H, dt, *J* = 16.0, 6.0 Hz, $C(=O)CH_AH_B$), 2.51 (1H, dt, *J* = 16.0, 7.8 Hz, $C(=O)CH_AH_B$), 3.05 (1H, d, *J* = 7.3 Hz, C(=O)C*H*CH), 3.92 (1H, m, CH_2CHOCH_2), 3.98 (1H, m, CH_2CHOCH_2), 5.03 (1H, d, *J* = 7.3 Hz, C*H*(OTMS)Ph), 7.04 (1H, dd, *J* = 7.3, 7.3 Hz, aromatic), 7.12 (2H, dd, *J* = 7.3, 7.3 Hz, aromatic), 7.26 (2H, d, *J* = 7.3 Hz, aromatic); ^{13}C NMR (100 MHz, $CO(CD_3)_2$) δ 0.26, 22.0, 23.4, 26.6, 33.0, 34.3, 36.0, 40.0, 48.0, 58.4, 68.8, 68.9, 76.0, 78.1, 110.8, 128.0, 128.6, 129.0, 144.0, 210.5; HRMS (ESI-TOF) calcd for $C_{24}H_{34}NaO_5Si$ $[M+Na]^+$ 453.2068, found 453.2058.

4-20 : colorless oil; IR (film) 2953, 1703, 1448, 1394, 1322, 1297, 1253, 1129, 1064 cm^{-1}; ^{1}H NMR (400 MHz, C_6D_6) δ 0.01 (9H, s, C*H*$_3$ of TMS), 0.42 (1H, d, *J* = 13.3 Hz, $COCH_{ax}H_{eq}CHO$), 0.71 (1H, d, *J* = 12.8 Hz, $COCH_{ax}H_{eq}CHO$), 0.74 (1H, d, *J* = 12.8 Hz, $COCH_{ax}H_{eq}CHO$), 1.53 (3H, s, C*H*$_3$), 1.64-1.84 (3H, m, $C(=O)CH_2CH_AH_BCH_AH_B$, $COCH_{ax}H_{eq}CHO$), 1.90-2.10 (4H, m, C(=O)CHC*H*, $C(=O)CH_2CH_AH_BCH_AH_B$, $COCH_{ax}H_{eq}CHO$), 2.14 (1H, m, $COCH_{ax}H_{eq}CHO$), 2.35 (1H, ddd, *J* = 16.5, 10.1, 7.4 Hz, $C(=O)CH_AH_B$), 2.54 (1H, dt, *J* = 16.5, 4.6 Hz, $C(=O)CH_AH_B$), 2.94 (1H, d, *J* = 3.7 Hz, C(=O)C*H*CH), 3.88 (1H, m, CH_2CHOCH_2), 3.96 (1H, m, CH_2CHOCH_2), 5.35 (1H, d, *J* = 3.7 Hz, C*H*(OTMS)Ph), 7.01 (1H, t, *J* = 7.3, 7.3 Hz, aromatic), 7.10 (2H, t, *J* = 7.3, 7.3 Hz, aromatic), 7.18 (2H, d, *J* = 7.3 Hz, aromatic); ^{13}C NMR (100 MHz, $CO(CD_3)_2$) δ 0.11, 22.68, 22.74, 26.5, 33.0, 35.0, 35.2, 41.6, 43.6, 57.7, 68.86, 68.93, 75.9, 77.4, 110.8, 127.5, 128.3, 128.9, 143.8, 211.2; HRMS (ESI-TOF) calcd for $C_{24}H_{34}NaO_5Si$ $[M+Na]^+$ 453.2068, found 453.2059.



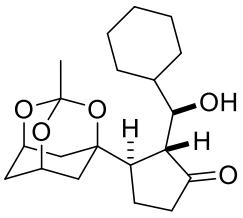
4-22

Compound 4-22 [DK-2-196]. According to the general procedure F, **4-22** (34 mg, 0.12 mmol) was synthesized from O,Te-acetal **2-8** (51 mg, 0.14 mmol), cyclopentenone (36 μL, 0.43 mmol), and acetaldehyde (80 μL, 1.4 mmol) in 86% yield by using Et_3B (1.03 M in hexane, 0.41 mL, 0.42 mmol) in CH_2Cl_2 (1.4 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:2): colorless solid; m.p. 132-135 °C; IR (film) 3453, 2960, 1735, 1396, 1325, 1299, 1127 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.12 (3H, d, *J* = 6.4 Hz, CH(OH)C*H*$_3$), 1.44 (3H, s, C*H*$_3$), 1.49-1.64 (3H, m, $COCH_{ax}H_{eq}CHO$ x2, $C(=O)CH_2CH_AH_B$), 1.80 (1H, d, *J* = 12.8 Hz, $COCH_{ax}H_{eq}CHO$), 1.95-2.06 (1H, m, $C(=O)CH_2CH_AH_B$), 2.15 (1H, td, *J* = 18.8, 10.6 Hz, $C(=O)CH_AH_B$), 2.13 (1H, m, $COCH_{ax}H_{eq}CHO$), 2.27-2.46 (3H, m, $C(=O)CH_AH_B$, C(=O)CHC*H*, $COCH_{ax}H_{eq}CHO$), 2.50-2.61 (2H, m, $COCH_{ax}H_{eq}CHO$, C(=O)C*H*CH), 3.56 (1H, br s, O*H*), 4.24 (1H, m, C*H*OH), 4.46 (2H, m, CH_2CHOCH_2 x2); ^{13}C NMR (100 MHz, CDCl3) δ 19.9, 20.7, 26.0, 31.0, 32.3, 36.6, 38.0, 47.2, 56.2, 66.8, 67.7, 67.9, 74.8, 110.3, 217.2; HRMS (ESI-TOF) calcd for $C_{15}H_{22}NaO_5$ $[M+Na]^+$ 305.1359, found 305.1368.



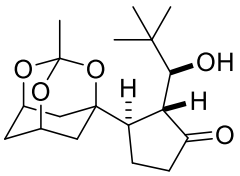
4-23

Compound 4-23 [DK-2-195]. According to the general procedure F, **4-23** (30 mg, 0.085 mmol) was synthesized from O,Te-acetal **2-8** (38 mg, 0.11 mmol), cyclopentenone (26 μL, 0.31 mmol), and heptanal (44 μL, 0.31 mmol) in 77% yield by using Et_3B (1.03 M in hexane, 0.30 mL, 0.31 mmol) in CH_2Cl_2 (1.0 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless oil; IR (film) 3462, 2930, 2857, 1737, 1453, 1396, 1324, 1299, 1128 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.86 (3H, t, *J* = 6.9 Hz, CH_2CH_3), 1.20-1.34 (8H, m, CH_2 of *n*-hexyl x4), 1.43 (3H, s, CH_3), 1.44-1.62 (5H, m, CH_2 of *n*-hexyl, $COCH_{ax}H_{eq}CHO$ x2, $C(=O)CH_2CH_AH_B$), 1.79 (1H, d, *J* = 11.4 Hz, $COCH_{ax}H_{eq}CHO$), 1.95-2.05 (1H, m, $C(=O)CH_2CH_AH_B$), 2.10-2.28 (2H, m, $C(=O)CH_AH_B$, $COCH_{ax}H_{eq}CHO$), 2.30-2.43 (3H, m, $C(=O)CH_AH_B$, C(=O)CHCH, $COCH_{ax}H_{eq}CHO$), 2.54 (2H, m, $COCH_{ax}H_{eq}CHO$, C(=O)CHCH), 3.14 (1H, br s, OH), 3.99 (1H, m, CHOH), 4.46 (2H, m, CH_2CHOCH_2 x2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 20.7, 22.6, 25.9, 26.3, 29.2, 31.2, 31.7, 32.3, 34.6, 36.7, 38.3, 47.7, 55.6, 67.8, 68.0, 71.2, 74.8, 110.3, 217.4; HRMS (ESI-TOF) calcd for $C_{20}H_{32}NaO_5$ $[M+Na]^+$ 375.2142, found 375.2144.



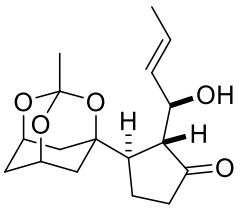
4-24

Compound 4-24 [DK-2-194]. According to the general procedure F, **4-24** (28 mg, 0.080 mmol) was synthesized from O,Te-acetal **2-8** (29 mg, 0.081 mmol), cyclopentenone (20 μL, 0.24 mmol), and cyclohexanecarboxaldehyde (29 μL, 0.24 mmol) in 99% yield by using Et_3B (1.03 M in hexane, 0.24 mL, 0.25 mmol) in CH_2Cl_2 (0.81 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless solid; m.p. 128-130 °C; IR (film) 3465, 2926, 2850, 1734, 1448, 1395, 1324, 1299, 1128 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 0.77 (1H, dd, *J* = 12.8, 1.4 Hz, $COCH_{ax}H_{eq}CHO$), 0.84 (1H, dt, *J* = 13.3, 1.8 Hz, $COCH_{ax}H_{eq}CHO$), 0.98 (1H, dd, *J* = 13.3, 1.8 Hz, $COCH_{ax}H_{eq}CHO$), 1.01-1.46 (7H, m, $C(=O)CH_2CH_2$, CH of cyclohexyl x5), 1.57-1.78 (4H, m, CH of cyclohexyl x4), 1.61 (3H, s, CH_3), 1.86 (1H, m, $COCH_{ax}H_{eq}CHO$), 1.91-2.03 (4H, m, $C(=O)CH_AH_B$, $COCH_{ax}H_{eq}CHO$, CH of cyclohexyl x2), 2.12-2.21 (2H, m, C(=O)CHCH, $C(=O)CH_AH_B$), 2.25 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, $COCH_{ax}H_{eq}CHO$), 2.49-2.58 (2H, m, C(=O)CHCH, OH), 3.78 (1H, m, CHOH), 4.03 (2H, m, CH_2CHOCH_2 x2); ^{13}C NMR (100 MHz, C_6D_6) δ 20.6, 26.3, 26.4, 26.5, 26.8, 29.9, 30.5, 32.0, 32.4, 36.4, 38.7, 41.5, 48.5, 52.1, 67.7, 67.9, 74.8, 77.1, 110.8, 216.7; HRMS (ESI-TOF) calcd for $C_{20}H_{30}NaO_5$ $[M+Na]^+$ 373.1985, found 373.1993.



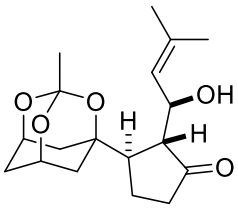
4-25

Compound 4-25 [DK-4-172]. According to the general procedure F, **4-25** (24 mg, 0.074 mmol) was synthesized from O,Te-acetal **2-8** (36 mg, 0.10 mmol), cyclopentenone (17 μL, 0.20 mmol), and trimethylacetaldehyde (54 μL, 0.50 mmol) in 74% yield by using Et_3B (1.03 M in hexane, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (0.99 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless oil; IR (film) 3480, 2955, 2874, 1736, 1396, 1366, 1323, 1299, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.98 (9H, s, *t*-Bu), 1.40 (3H, s, C*H*$_3$) 1.50-1.66 (3H, m, COC*H*$_{ax}$H$_{eq}$CHO x2, C(=O)CH$_2$C*H*$_A$H$_B$), 1.73 (1H, dd, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 2.03 (1H, dt, *J* = 13.2, 8.7 Hz, C(=O)CH$_2$CH$_A$*H*$_B$), 2.20 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.23-2.33 (4H, m, C(=O)C*H*$_2$, COCH$_{ax}$*H*$_{eq}$CHO, O*H*), 2.40 (1H, q, *J* = 8.7 Hz, C(=O)CHC*H*), 2.52 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, CHOCH$_{ax}$*H*$_{eq}$CHO), 2.60 (1H, d, *J* = 8.7 Hz, C(=O)C*H*CH), 3.75 (1H, s, C*H*OH), 4.42 (2H, m, CH$_2$C*H*OCH$_2$ x2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 25.9, 26.9, 31.7, 32.5, 36.5, 36.6, 38.6, 50.8, 51.3, 67.8, 68.0, 74.9, 81.0, 110.1, 218.6; HRMS (ESI-TOF) calcd for $C_{18}H_{28}NaO_5$ $[M+Na]^+$
347.1829, found 347.1840.



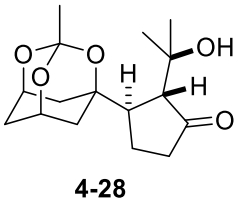
4-26

Compound 4-26 [DK-3-026]. According to the general procedure F, **4-26** (18 mg, 0.058 mmol) was synthesized from O,Te-acetal **2-8** (51 mg, 0.14 mmol), cyclopentenone (24 μL, 0.29 mmol), and *E*-2-butenal (35 μL, 0.42 mmol) in 41% yield by using Et_3B (1.03 M in hexane, 0.41 mL, 0.42 mmol) in CH_2Cl_2 (1.4 mL) under air. The crude was purified by flash column chromatography on silica gel (15 g, hexane/EtOAc 2:1 to 1:1 to 1:2): colorless solid; m.p. 118-121 °C; IR (film) 3437, 2955, 1737, 1396, 1324, 1299, 1128 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.46 (3H, s, C*H*$_3$), 1.50-1.62 (1H, m, C(=O)CH$_2$C*H*$_A$H$_B$), 1.57 (1H, dd, *J* = 11.0, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.60 (1H, dt, *J* = 11.0, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO,), 1.67 (3H, dd, *J* = 6.0, 1.4 Hz, CH=CHC*H*$_3$), 1.78 (1H, dd, *J* = 12.6, 1.2 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.98 (1H, m, C(=O)CH$_2$CH$_A$*H*$_B$), 2.05-2.15 (1H, ddd, *J* = 16.5, 9.2, 7.8 Hz, C(=O)C*H*$_A$H$_B$), 2.22 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.30-2.42 (3H, m, C(=O)CHC*H*, C(=O)CH$_A$*H*$_B$, COCH$_{ax}$*H*$_{eq}$CHO), 2.55 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.60 (1H, dd, *J* = 8.2, 3.2 Hz, C(=O)C*H*CH), 3.86 (1H, br s, O*H*), 4.45 (2H, m, CH$_2$C*H*OCH$_2$ x2), 4.53 (1H, m, C*H*OH), 5.40 (1H, ddd, *J* = 12.4, 5.5, 1.4 Hz, CH(OH)C*H*=CH), 5.63 (1H, dqd, *J* = 12.4, 6.0, 1.0 Hz, CH(OH)CH=C*H*); ^{13}C NMR (125 MHz, $CDCl_3$) δ 17.8, 20.8, 25.9, 31.1, 32.2, 36.5, 38.2, 47.3, 55.7, 67.8, 67.9, 71.7, 74.9, 110.3, 128.0, 130.4, 216.9; HRMS (ESI-TOF) calcd for $C_{17}H_{24}NaO_5$ $[M+Na]^+$ 331.1516, found 331.1522.



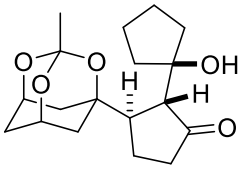
4-27

Compound 4-27 [DK-3-020]. According to the general procedure F, **4-27** (32 mg, 0.099 mmol) was synthesized from O,Te-acetal **2-8** (45 mg, 0.13 mmol), cyclopentenone (21 μL, 0.25 mmol), and 3-methyl-2-butenal (60 μL, 0.62 mmol) in 76% yield by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (1.2 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless oil; IR (film) 3450, 2958, 1737, 1447, 1395, 1324, 1299, 1240, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.45 (3H, s, CH_3), 1.55-1.63 (1H, m, C(=O)CH_2C$H_A$$H_B$), 1.58 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.60 (1H, dt, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.67 (3H, d, *J* = 0.9 Hz, CH=CCH_3), 1.71 (3H, d, *J* = 0.9 Hz, CH=CCH_3), 1.77 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.93-2.04 (1H, m, C(=O)$CH_2$$CH_A$$H_B$), 2.10 (1H, m, C(=O)C$H_A$$H_B$), 2.25 (1H, m, COC$H_{ax}$$H_{eq}$CHO), 2.27-2.45 (3H, m, C(=O)$CH_A$$H_B$, C(=O)CHC*H*, COC$H_{ax}$$H_{eq}$CHO), 2.50-2.61 (2H, m, C(=O)C*H*CH, COC$H_{ax}$$H_{eq}$CHO), 3.48 (1H, br s, O*H*), 4.46 (2H, m, CH_2C*H*OCH_2 x2), 4.79 (1H, m, C*H*OH), 5.30 (1H, d, *J* = 9.6 Hz, C*H*=C); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.3, 20.9, 26.0, 26.1, 31.4, 32.3, 36.4, 38.3, 47.5, 55.5, 67.6, 67.8, 68.0, 74.9, 110.3, 124.4, 135.9, 217.0; HRMS (ESI-TOF) calcd for $C_{18}H_{26}NaO_5$ $[M+Na]^+$ 345.1672, found 345.1687.



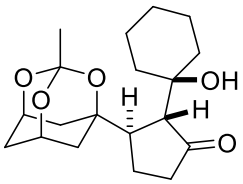
4-28

Compound 4-28 [DK-3-016]. According to the general procedure A, **4-28** (19 mg, 0.064 mmol) was synthesized from O,Te-acetal **2-8** (36 mg, 0.10 mmol), cyclopentenone (17 μL, 0.20 mmol), and acetone (36 μL, 0.49 mmol) in 64% yield by using Et_3B (1.03 M in hexane, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (0.99 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless oil; IR (film) 3457, 2962, 1730, 1395, 1325, 1299, 1130 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.19 (3H, s, CH_3), 1.29 (3H, s, CH_3), 1.42 (3H, s, CH_3), 1.56 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.58 (1H, dt, *J* = 13.3, 1.8 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.69 (1H, dd, *J* = 12.8, 1.4 Hz, COC$H_{ax}$$H_{eq}$CHO), 1.86-2.08 (2H, m, C(=O)CH_2CH_2), 2.18-2.40 (5H, m, C(=O)CH_2, COC$H_{ax}$$H_{eq}$CHO x2, C(=O)CHC*H*), 2.45 (1H, d, *J* = 4.6 Hz, C(=O)C*H*), 2.54 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, CHOC$H_{ax}$$H_{eq}$CHO), 4.45 (2H, m, CH_2C*H*OCH_2 x2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 25.9, 27.3, 27.9, 32.3, 32.6, 35.6, 38.9, 47.8, 57.3, 67.7, 67.9, 72.1, 74.7, 110.3, 221.2; HRMS (ESI-TOF) calcd for $C_{16}H_{24}NaO_5$ $[M+Na]^+$ 319.1516, found 319.1525.



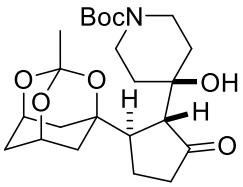
4-29

Compound 4-29 [DK-3-008]. According to the general procedure F, **4-29** (6.3 mg, 0.021 mmol) was synthesized from O,Te-acetal **2-8** (34 mg, 0.094 mmol), cyclopentenone (16 μL, 0.19 mmol), and cyclopentanone (42 μL, 0.47 mmol) in 22% yield by using Et_3B (1.03 M in hexane, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.96 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless oil; IR (film) 3478, 2955, 2873, 1730, 1395, 1324, 1298, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.42 (3H, s, CH_3), 1.50-1.71 (8H, m, COC$H_{ax}H_{eq}$CHO x3, C*H* of cyclopentyl x5), 1.74-1.85 (3H, m, C*H* of cyclopentyl x3), 1.89-2.10 (2H, m, C(=O)CH_2CH_2), 2.16-2.35 (5H, m, COC$H_{ax}H_{eq}$CHO x2, C(=O)CHC*H*, C(=O)CH_2), 2.53 (1H, m, COC$H_{ax}H_{eq}$CHO), 2.59 (1H, d, *J* = 4.1 Hz, C(=O)C*H*), 3.22 (1H, s, O*H*), 4.44 (2H, m, CH_2CHOCH_2 x2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.6, 23.2, 23.5, 26.0, 32.3, 32.9, 35.5, 37.6, 37.8, 38.8, 48.2, 54.7, 67.8, 67.9, 74.6, 83.4, 110.3, 221.5; HRMS (ESI-TOF) calcd for $C_{18}H_{26}NaO_5$ $[M+Na]^+$ 345.1672, found 345.1686.



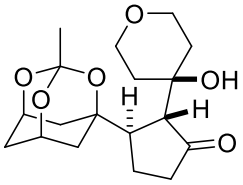
4-30

Compound 4-30 [DK-2-191]. According to the general procedure F, **4-30** (29 mg, 0.086 mmol) was synthesized from O,Te-acetal **2-8** (36 mg, 0.10 mmol), cyclopentenone (17 μL, 0.20 mmol), and cyclohexanone (51 μL, 0.49 mmol) in 86% yield by using Et_3B (1.03 M in hexane, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (0.99 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1): colorless oil; IR (film) 3479, 2933, 1726, 1448, 1395, 1323, 1297, 1130 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 0.76 (1H, dd, *J* = 12.8, 1.8 Hz, COC$H_{ax}H_{eq}$CHO), 0.79-0.86 (2H, m, COC$H_{ax}H_{eq}$CHO x2), 1.09 (1H, ddt, *J* = 12.8, 12.8, 3.6 Hz, C*H* of cyclohexyl), 1.27-1.40 (2H, m), 1.43-1.66 (7H, m), 1.60 (3H, s, CH_3), 1.74-1.99 (6H, m, COC$H_{ax}H_{eq}$CHO x2), 2.05 (1H, dt, *J* = 9.2, 3.6 Hz, C(=O)CHC*H*), 2.24 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, COC$H_{ax}H_{eq}$CHO), 2.27 (1H, d, *J* = 3.6 Hz, C(=O)C*H*), 3.39 (1H, s, O*H*), 4.01 (2H, m, CH_2CHOCH_2 x2); ^{13}C NMR (100 MHz, C_6D_6) δ 20.6, 22.0, 22.1, 26.1, 26.4, 32.3, 33.4, 34.8, 35.5, 36.0, 39.0, 47.2, 57.3, 67.7, 67.8, 73.5, 74.7, 110.8, 221.3; HRMS (ESI-TOF) calcd for $C_{19}H_{28}NaO_5$ $[M+Na]^+$ 359.1829, found 359.1830.



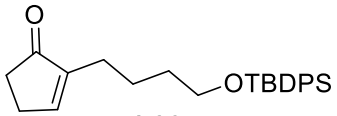
4-31

Compound 4-31 [DK-2-167]. According to the general procedure F, **4-31** (39 mg, 0.089 mmol) was synthesized from O,Te-acetal **2-8** (35 mg, 0.097 mmol), cyclopentenone (24 μL, 0.29 mmol), and 1-(*tert*-butoxycarbonyl)-4-piperidone (58 mg, 0.29 mmol) in 92% yield by using Et_3B (1.03 M in hexane, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.50 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:2): colorless solid; m.p. 150-153 °C; IR (film) 3447, 2960, 2929, 1729, 1683, 1427, 1395, 1365, 1324, 1299, 1279, 1248, 1148, 1131 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30 (1H, d, *J* = 14.0 Hz, C*H* of piperidyl), 1.41 (3H, s, CH_3), 1.45 (9H, s, *t*-Bu), 1.48-1.60 (4H, m, COC$H_{ax}H_{eq}$CHO x2, C*H* of piperidyl x2), 1.71 (1H, d, *J* = 12.8 Hz, COC$H_{ax}H_{eq}$CHO), 1.78-2.01 (3H, m, C(=O)CH_2CH_2, C*H* of piperidyl), 2.14-2.40 (5H, m, C(=O)CH_2, C(=O)CHC*H*, COC$H_{ax}H_{eq}$CHO x2), 2.48 (1H, d, *J* = 4.1 Hz, C(=O)C*H*), 2.53 (1H, m, CHOC$H_{ax}H_{eq}$CHO), 2.98-3.23 (2H, m, C*H* of pyperidyl x2), 3.77-4.03 (3H, m, C*H* of pyperidyl x2, O*H*), 4.41-4.47 (2H, m, CH_2C*H*OCH_2 x2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.6, 25.9, 28.4, 32.3, 32.4, 34.6 (br), 35.8, 38.9 (br), 39.1, 39.6 (br), 46.9, 56.4 (br), 67.7, 67.9, 71.6 (br), 74.6, 79.4, 110.3, 154.8, 221.0 222.0; HRMS (ESI-TOF) calcd for $C_{23}H_{35}NNaO_7$ $[M+Na]^+$ 460.2306, found 460.2309.



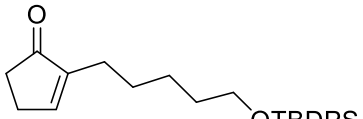
4-32

Compound 4-32 [DK-2-166]. According to the general procedure F, **4-32** (27 mg, 0.080 mmol) was synthesized from O,Te-acetal **2-8** (32 mg, 0.089 mmol), cyclopentenone (22 μL, 0.26 mmol), and 4-Oxotetrahydropyran (24 μL, 0.26 mmol) in 90% yield by using Et_3B (1.03 M in hexane, 0.26 mL, 0.26 mmol) in CH_2Cl_2 (0.88 mL) under air. The crude was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:3 to 1:4): colorless solid; m.p. 145-147 °C; IR (film) 3441, 3007, 2955, 1728, 1395, 1324, 1299, 1242, 1148, 1131 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 0.67 (1H, dd, *J* = 12.8, 0.9 Hz, COC$H_{ax}H_{eq}$CHO), 0.78-0.85 (2H, m, COC$H_{ax}H_{eq}$CHO x2), 1.05 (1H, dd, *J* = 13.3, 2.3 Hz, C(OH)C$H_A$$H_B$$CH_2$), 1.27-1.47 (4H, m, C(=O)C$H_A$$H_B$, C(=O)$CH_2CH_2$, C(OH)C$H_A$$H_B$$CH_2$), 1.56 (3H, s, C$H_3$), 1.64-2.02 (6H, m, C(OH)$CH_A$$H_B$ x2, COC$H_{ax}H_{eq}$CHO x2, C(=O)$CH_A$$H_B$, C(=O)CHC*H*), 2.24 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, CHOC$H_{ax}H_{eq}$CHO), 2.28 (1H, d, *J* = 3.6 Hz, C(=O)C*H*), 3.70 (1H, s, O*H*), 3.79 (2H, td, *J* = 11.4, 4.1 Hz, C$H_A$$H_BOCH_A$$H_B$), 3.90-4.04 (4H, m, $CH_A$$H_BOCH_A$$H_B$, CH_2C*H*OCH_2 x2); ^{13}C NMR (100 MHz, C_6D_6) δ 20.6, 26.4, 32.3, 32.9, 35.2, 35.7, 36.4, 39.1, 46.9, 56.7, 63.6, 63.7, 67.6, 67.9, 71.3, 74.5, 110.8, 220.8; HRMS (ESI-TOF) calcd for $C_{18}H_{26}NaO_6$ $[M+Na]^+$ 361.1622, found 361.1606.



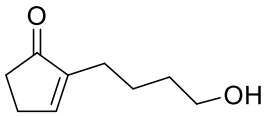
4-36

Compound 4-36 [DK-3-118]. 9-Borabicyclo[3.3.1]nonane (9-BBN, 0.5 M THF solution, 8.5 mL, 4.3 mmol) was added to a solution of 4-[(*tert*-butyldiphenylsilyl)oxy]-1-butene[11] (**4-33**) (1.20 g, 3.86 mmol) in THF (8.5 mL) at 0 °C, and resultant mixture was stirred at 60°C for 4 h. To an additional solution of 2-iodo-2-cyclopentene-1-one (**4-35**)[12] (800 mg, 3.85 mmol) and [1,1′-bis(diphenylphosphino)ferrocene]dichloropalladium(II) [$PdCl_2$(dppf), 281 mg, 0.344 mmol] in DMF (8.5 mL), the above reaction mixture and 3 M aqueous K_3PO_4 (3.2 mL, 9.6 mmol) were successively added at room temperature. After being stirred at 60°C for 2 h, the reaction mixture was diluted with H_2O (20 mL) at room temperature. The resultant mixture was extracted with EtOAc (20 mL x3), and the combined organic layers were washed with H_2O (30 mL) and brine (30 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (40 g, hexane/EtOAc 4/1) to afford enone **4-36** (961 mg, 2.45 mmol) in 64% yield: colorless oil; IR (film) 3069, 3048, 2931, 2859, 1704, 1631, 1589, 1468, 1428, 1388, 1361, 1301, 1254, 1192, 1108, 1002 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu), 1.53-1.62 (4H, m, C*H*$_2$C*H*$_2$CH$_2$OTBDPS), 2.16 (2H, m, C(=O)CC*H*$_2$), 2.39 (2H, m, C(=O)C*H*$_2$), 2.55 (2H, ddd, *J* = 9.2, 4.6, 1.7 Hz, C(=O)CH$_2$C*H*$_2$), 3.67 (2H, t, *J* = 6.3 Hz, C*H*$_2$OTBDPS), 7.27 (1H, m, C(=O)CC*H*), 7.35-7.44 (6H, m, aromatic), 7.66 (4H, dd, *J* = 8.0, 1.8 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.2, 23.9, 24.5, 26.4, 26.8 (3C), 32.2, 34.6, 63.6, 127.6 (4C), 129.5 (2C), 134.0 (2C), 135.5 (4C), 146.3, 157.3, 210.0; HRMS (ESI-TOF) calcd for $C_{25}H_{32}NaO_2Si$ $[M+Na]^+$ 415.2064, found 415.2072.



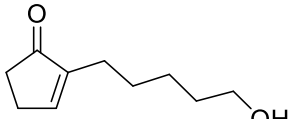
4-37

Compound 4-37 [DK-5-129]. According to the synthetic protocol of **4-36**, enone **4-37** (635 mg, 1.56 mmol) was synthesized from **4-35** (800 mg, 3.85 mmol) and 5-[(*tert*-butyldiphenylsilyl)oxy]-1-penten **4-34**[13] (1.25 g, 3.85 mmol) in 41% yield by using 9-BBN (8.5 mL of a 0.5 M solution in THF, 4.3 mmol), THF (8.5 mL), $PdCl_2$(dppf) (281 mg, 0.344 mmol) and DMF (8.5 mL). The residue was purified by flash column chromatography on silica gel (30 g, hexane/EtOAc 10/1): colorless oil; IR (film) 3065, 3048, 2931, 2859, 2361, 1703, 1631, 1466, 1432, 1386, 1355, 1297, 1251, 1193, 1106, 1002 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu), 1.34-1.52 (4H, m, C*H*$_2$C*H*$_2$CH$_2$CH$_2$OTBDPS), 1.58 (2H, dt, *J* = 14.6, 6.9 Hz, C*H*$_2$CH$_2$OTBDPS), 2.17 (2H, m, C(=O)CC*H*$_2$), 2.39 (2H, m, C(=O)C*H*$_2$), 2.54 (2H, ddd, *J* = 9.2, 4.6, 2.3 Hz, C(=O)CH$_2$C*H*$_2$), 3.66 (2H, t, *J* = 6.4 Hz, C*H*$_2$OTBDPS), 7.27 (1H, m, C(=O)CC*H*), 7.35-7.45 (6H, m, aromatic), 7.67 (4H, dd, *J* = 8.2, 1.8 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 24.7, 25.6, 26.4, 26.8 (3C), 27.4, 32.3, 34.6, 63.8, 127.5 (4C), 129.5 (2C), 134.1 (2C), 135.5 (4C), 146.3, 157.3, 210.0; HRMS (ESI-TOF) calcd for $C_{26}H_{34}NaO_2Si$ $[M+Na]^+$ 429.2220, found 429.2222.



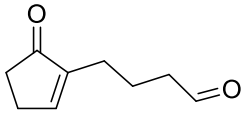
4-38

Compound 4-38 [DK-5-121]. Tetra-*n*-butylammonium fluoride (TBAF, 1 M in THF, 1.1 mL, 1.1 mmol) was added to a solution of enone **4-36** (144 mg, 0.367 mmol) and AcOH (0.11 mL, 1.8 mmol) in THF (1.8 mL) at room temperature. The reaction mixture was stirred at room temperature for 22 h, and then saturated aqueous NH_4Cl (10 mL) was added. The resultant mixture was extracted with $CHCl_3$ (10 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1/4) to afford alcohol **4-38** (50.9 mg, 0.330 mmol) in 90% yield: colorless oil; IR (film) 3413, 2933, 2865, 1688, 1629, 1441, 1355, 1254, 1204, 1056, 1004 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.47-1.58 (4H, m, C*H*$_2$C*H*$_2$CH$_2$OH), 2.15 (2H, m, C(=O)CC*H*$_2$), 2.35 (2H, m, C(=O)C*H*$_2$), 2.42-2.58 (3H, m, C(=O)CH$_2$C*H*$_2$, O*H*), 3.60 (2H, t, *J* = 6.3 Hz, C*H*$_2$OH), 7.30 (1H, m, C(=O)CC*H*); ^{13}C NMR (125 MHz, $CDCl_3$) δ 23.9, 24.3, 26.4, 32.2, 34.5, 62.2, 146.0, 157.9, 210.3; HRMS (ESI-TOF) calcd for $C_9H_{14}NaO_2$ $[M+Na]^+$ 177.0886, found 177.0879.



4-39

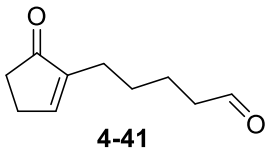
Compound 4-39 [DK-5-131]. According to the synthetic protocol of **4-38**, alcohol **4-39** (242 mg, 1.44 mmol) was synthesized from enone **4-37** (635 mg, 1.56 mmol) in 92% yield by using TBAF (1 M in THF, 4.7 mL, 4.7 mmol), AcOH (0.45 mL, 7.8 mmol) and THF (7.8 mL). The residue was purified by flash column chromatography on silica gel (30 g, hexane/EtOAc 1/4): colorless oil; IR (film) 3402, 2930, 2861, 1691, 1630, 1441, 1407, 1350, 1301, 1249, 1203, 1148, 1053, 1004 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.29 (2H, m, C*H*$_2$CH$_2$CH$_2$OH), 1.38-1.56 (4H, m, C*H*$_2$CH$_2$C*H*$_2$CH$_2$OH), 2.11 (2H, m, C(=O)CC*H*$_2$), 2.26-2.36 (3H, m, C(=O)C*H*$_2$, OH), 2.50 (2H, ddd, *J* = 9.2, 4.6, 2.3 Hz, C(=O)CH$_2$C*H*$_2$), 3.55 (2H, t, *J* = 6.9 Hz, C*H*$_2$OH), 7.26 (1H, m, C(=O)CC*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.6, 25.4, 26.3, 27.4, 32.3, 34.5, 62.4, 146.1, 157.7, 210.2; HRMS (ESI-TOF) calcd for $C_{10}H_{16}NaO_2$ $[M+Na]^+$ 191.1043, found 191.1033.



4-40

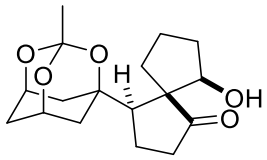
Compound 4-40 [DK-3-121]. (Diacetoxyiodo)benzene [$PhI(OAc)_2$, 467 mg, 1.45 mmol] and 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO, 19.0 mg, 0.122 mmol) were successively added to a solution of alcohol **4-38** (187 mg, 1.21 mmol) in CH_2Cl_2 (2.4 mL) at room temperature. The reaction mixture was stirred at room temperature for 18 h, and was then concentrated. The residue was purified by flash column chromatography on

silica gel (15 g, hexane/EtOAc 1/1) to afford aldehyde **4-40** (147 mg, 0.966 mmol) in 80% yield: colorless oil; IR (film) 2926, 2846, 2727, 1696, 1631, 1442, 1403, 1357, 1297, 1254, 1202, 1169, 1088, 1051, 1002 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.79 (2H, dt, *J* = 15.1, 7.8 Hz, C*H*$_2$CH$_2$CHO), 2.17 (2H, m, C(=O)CC*H*$_2$), 2.37 (2H, m, C(=O)C*H*$_2$), 2.43 (2H, td, *J* = 7.3, 1.8 Hz, C*H*$_2$CHO), 2.55 (2H, ddd, *J* = 9.2, 4.6, 2.3 Hz, C(=O)CH$_2$C*H*$_2$), 7.33 (1H, m, C(=O)CC*H*), 9.73 (1H, t, *J* = 1.4 Hz, C*H*O); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.2, 24.1, 26.4, 34.4, 43.3, 145.3, 158.1, 202.0, 209.7; HRMS (ESI-TOF) calcd for $C_9H_{12}NaO_2$ $[M+Na]^+$ 175.0730, found 175.0723.



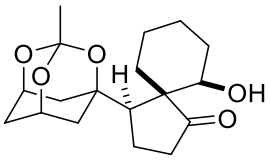
4-41

Compound 4-41 [DK-5-134]. According to the synthetic protocol of **4-40**, aldehyde **4-41** (188 mg, 1.13 mmol) was synthesized from alcohol **4-39** (203 mg, 1.21 mmol) in 93% yield by using $PhI(OAc)_2$ (467 g, 1.45 mmol), TEMPO (19.0 mg, 0.122 mmol) and CH_2Cl_2 (6.1 mL). The residue was purified by flash column chromatography on silica gel (15 g, hexane/EtOAc 1/1): colorless oil; IR (film) 2929, 2862, 2725, 1721, 1697, 1631, 1442, 1407, 1390, 1351, 1298, 1252, 1200, 1160, 1092, 1045, 1003 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.50 (2H, m, C*H*$_2$CH$_2$CH$_2$CHO), 1.62 (2H, dt, *J* = 15.6, 7.8 Hz, C*H*$_2$CH$_2$CHO), 2.18 (2H, m, C(=O)CC*H*$_2$), 2.37 (2H, m, C(=O)C*H*$_2$), 2.44 (2H, td, *J* = 7.3, 1.8 Hz, C*H*$_2$CHO), 2.54 (2H, ddd, *J* = 9.2, 4.6, 1.8 Hz, C(=O)CH$_2$C*H*$_2$), 7.30 (1H, m, C(=O)CC*H*), 9.74 (1H, t, *J* = 1.8 Hz, C*H*O); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 24.4, 26.3, 27.1, 34.4, 43.4, 145.6, 157.6, 202.3, 209.8; HRMS (ESI-TOF) calcd for $C_{10}H_{14}NaO_2$ $[M+Na]^+$ 189.0886, found 189.0885.



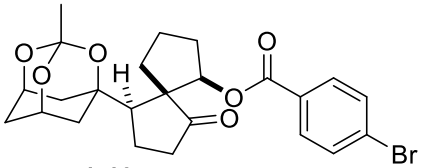
4-42

Compound 4-42 [DK-3-123, DK-5-132]. According to the general procedure A, **4-42** (16.1 mg, 0.0522 mmol) was synthesized from O,Te-acetal **2-8** (18.8 mg, 0.0522 mmol) and 4-(5-oxocyclopent-1-en-1-yl)butanal (**4-40**) (16.0 mg, 0.105 mmol) in 100% yield by using Et_3B (1.03 M hexane solution, 0.15 mL, 0.16 mmol) in CH_2Cl_2 (0.52 mL). The reaction mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1/3): colorless oil; IR (film) 3449, 3003, 2952, 2872, 2360, 2340, 1728, 1447, 1395, 1324, 1298, 1226, 1150, 1129, 1073 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.41 (3H, s, CC*H*$_3$), 1.52 (1H, dd, *J* = 12.8, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.60 (1H, dt, *J* = 12.8, 1.8 Hz, CHOC*H*$_{ax}$H$_{eq}$CHO), 1.64-1.95 (6H, m, COC*H*$_{ax}$H$_{eq}$CHO, C(=O)CH$_2$C*H*$_A$H$_B$, CH(OH)C*H*$_A$H$_B$, CH(OH)CH$_2$C*H*$_2$, CH(OH)CH$_2$CH$_2$C*H*$_A$H$_B$), 1.99-2.26 (3H, m, C(=O)C*H*$_A$H$_B$, C(=O)CH$_2$CH$_A$*H*$_B$, CH(OH)CH$_A$*H*$_B$), 2.26-2.40 (4H, m, C(=O)CH$_A$*H*$_B$, C(=O)CC*H*, CH(OH)CH$_2$CH$_2$CH$_A$*H*$_B$, COCH$_{ax}$*H*$_{eq}$CHO), 2.42-2.57 (2H, m, COCH$_{ax}$*H*$_{eq}$CHO, CHOCH$_{ax}$*H*$_{eq}$CHO), 4.28 (1H, t, *J* = 7.3 Hz, C*H*(OH)), 4.38-4.46 (2H, m, CH$_2$C*H*OCH$_2$ x2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.8, 21.4, 26.1, 28.9, 32.5, 33.9, 34.6, 37.4, 37.5, 51.2, 62.6, 68.0, 68.2, 74.6, 81.0, 110.0, 222.2; HRMS (ESI-TOF) calcd for $C_{17}H_{24}NaO_5$ $[M+Na]^+$ 331.1516, found 331.1517.



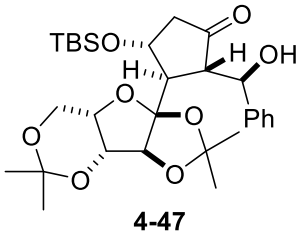
4-43

Compound 4-43 [DK-5-136]. According to the general procedure A, **4-43** (30.1 mg, 0.0934 mmol) was synthesized from O,Te-acetal **2-8** (36.0 mg, 0.100 mmol) and **4-41** (33.0 mg, 0.199 mmol) in 93% yield by using Et_3B (1.03 M hexane solution, 0.29 mL, 0.30 mmol) in CH_2Cl_2 (1.0 mL). The reaction mixture was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1/3): colorless prism; m.p. 173-174 °C; IR (film) 3449, 2948, 2863, 1727, 1448, 1394, 1394, 1324, 1298, 1154, 1129, 1041 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.26 (1H, m, $CH(OH)CH_2CH_AH_B$), 1.40-1.52 (2H, m, $CH(OH)CH_2CH_2CH_2$), 1.43 (3H, s, CCH_3), 1.54-1.64 (2H, m, $COCH_{ax}H_{eq}CHO$ x2), 1.69 (1H, m, $CH(OH)CH_2CH_2CH_2CH_AH_B$), 1.73-1.86 (3H, m, $CHOCH_{ax}H_{eq}CHO$, $CH(OH)CH_AH_B$, $CH(OH)CH_2CH_AH_B$), 1.86-2.02 (3H, m, $C(=O)CH_2CH_2$, $CH(OH)CH_2CH_2CH_2CH_AH_B$), 2.02-2.24 (2H, m, $C(=O)CH_AH_B$, $CH(OH)CH_AH_B$), 2.37 (1H, m, $C(=O)CH_AH_B$), 2.45-2.62 (3H, m, $COCH_{ax}H_{eq}CHO$ x2, $CHOCH_{ax}H_{eq}CHO$), 2.67 (1H, dd, J = 11.0, 8.2 Hz, $C(=O)CH_2CH_2CH$), 3.99 (1H, dd, J = 11.4, 5.5 Hz, $CH(OH)$), 4.41 (2H, m, J = 1.8 Hz, CH_2CHOCH_2 x2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.5, 20.3, 24.3, 26.1, 27.0, 31.3, 32.5, 35.2, 37.6, 37.9, 48.6, 57.5, 68.2, 68.3, 72.7, 74.9, 110.1, 220.8; HRMS (ESI-TOF) calcd for $C_{18}H_{26}NaO_5$ $[M+Na]^+$ 345.1672, found 345.1664.



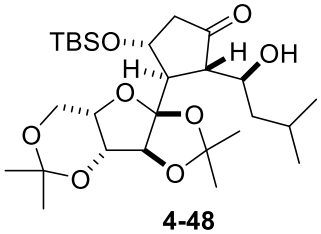
4-46

Compound 4-46 [DK-5-139]. *p*-Bromobenzoyl chloride (*p*-Br-BzCl, 22.0 mg, 0.100 mmol) was added to a solution of alcohol **4-42** (27.5 mg, 0.0892 mmol) and 4-dimethylaminopyridine (DMAP, 13.0 mg, 0.106 mmol) in CH_2Cl_2 (0.89 mL) at room temperature. The reaction mixture was stirred at room temperature for 12h, and then was directly subjected to flash column chromatography on silica gel (10 g, hexane/EtOAc 1/1) to afford *p*-bromobenzoyl ester **4-46** (35.7 mg, 0.0727 mmol) in 82% yield: colorless prism; m.p. 158.0-159.0 °C; IR (film) 2952, 2360, 2341, 1732, 1716, 1590, 1395, 1272, 1128, 1011 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, s, CCH_3), 1.49 (1H, dd, J = 12.8, 1.4 Hz, $COCH_{ax}H_{eq}CHO$), 1.60 (1H, m, $CHOCH_{ax}H_{eq}CHO$), 1.80-2.11 (7H, m,), 2.17-2.36 (5H, m,), 2.37-2.48 (2H, m,), 2.53 (1H, m, $CHOCH_{ax}H_{eq}CHO$), 4.38-4.46 (2H, m, CH_2CHOCH_2 x2), 5.45 (1H, dd, J = 6.4, 6.0 Hz, $CHOC(=O)Ar$), 7.56 (2H, m, aromatic), 7.79 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.7, 21.5, 26.0, 28.6, 30.0, 32.5, 34.3, 37.0, 37.6, 51.4, 61.1, 67.9, 68.2, 74.6, 83.0, 110.0, 128.2, 129.0, 131.0, 131.8, 165.5, 218.3; HRMS (ESI-TOF) calcd for $C_{24}H_{27}BrNaO_6$ $[M+Na]^+$ 513.0883, found 513.0891.



4-47

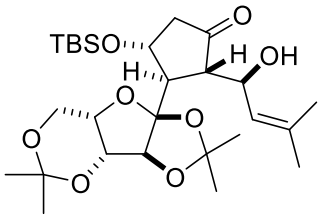
Compound 4-47 [DK-5-188, DK-6-094]. *General procedure H*: To a solution of acyl telluride **3-13** (50 mg, 0.11 mmol) in CH_2Cl_2 (1.1 mL) were added (*R*)-4-(*tert*-butyldimethylsilyloxy)-2-cyclopentene-1-one (46 mg, 0.22 mmol), benzaldehyde (33 μL, 0.33 mmol) and Et_3B (1.03 mol/L hexane solution, 0.32 mL, 0.33 mmol) at room temperature. Then, the reaction mixture was stirred for 15 min at rt, and then the reaction mixture was concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 4:1) to give **4-47** (53 mg, 0.097 mmol) in 88% yield: Colorless oil; $[\alpha]_D^{24}$ = -17.1 (*c* 2.7, $CHCl_3$); IR (film) 3484, 2992, 2953, 2931, 2898, 2858, 1724, 1457, 1377, 1250, 1192, 1119, 1065, 1011 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.10 (3H, s, TBS), 0.12 (3H, s, TBS), 0.52 (3H, s, acetonide), 0.94 (9H, s, TBS), 1.26 (3H, s, acetonide), 1.30 (3H, s, acetonide), 1.38 (3H, s, acetonide), 2.21-2.31 (2H, m, C(=O)CHC*H*, C(=O)CH_AH_B), 2.89 (1H, d, *J* = 9.6 Hz, C(=O)C*H*CH), 3.24 (1H, ddd, *J* = 18.8, 5.5, 1.4 Hz, C(=O)CH_AH_B), 3.86 (1H, m, H4), 3.90 (1H, d, *J* = 13.7 Hz, H5a), 4.00 (1H, dd, *J* = 13.7, 2.3 Hz, H5b), 4.14 (1H, d, *J* = 2.3 Hz, H3), 4.18 (1H, s, H2), 4.62 (1H, s, OH), 4.79 (1H, d, *J* = 5.0 Hz, C*H*(OTBS)), 4.92 (1H, d, *J* = 9.6 Hz, PhC*H*(OH)), 7.21-7.34 (3H, m, aromatics), 7.39-7.45 (2H, m, aromatics); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.5, 17.9, 18.4, 24.7, 25.8, 26.8, 28.9, 48.9, 54.6, 55.8, 59.8, 70.3, 71.4, 73.8, 75.7, 86.1, 97.3, 110.5, 114.4, 127.9, 128.1, 128.2, 141.0, 222.6; HRMS (ESI-TOF) calcd for $C_{29}H_{44}NaO_8Si$ $[M+Na]^+$ 571.2698, found 571.2689.



4-48

Compound 4-48 [DK-5-198, DK-6-095]. According to the general procedure H, **4-48** (49 mg, 0.093 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol), (*R*)-4-(*tert*-butyldimethylsilyloxy)-2-cyclopentene-1-one (46 mg, 0.22 mmol) and Isovaleraldehyde (35 μL, 0.33 mmol) in 85% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (1.1 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{24}$ = 9.7 (*c* 2.4, $CHCl_3$); IR (film) 3501, 2990, 2954, 2932, 2860, 1726, 1469, 1377, 1252, 1192, 1121, 1067, 1009 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.09 (3H, s, TBS), 0.13 (3H, s, TBS), 0.87 (9H, s, TBS), 0.90 (6H, t, *J* = 6.4 Hz, CH(CH_3)$_2$), 1.25-1.62 (14H, m, CH_2CH(CH_3)$_2$, acetonide x4), 1.81-2.11 (1H, m, C*H*(CH_3)$_2$), 2.16 (1H, d, *J* = 18.3 Hz, C(=O)CH_AH_B), 2.44 (1H, d, *J* = 5.5 Hz, C(=O)C*H*), 2.56 (1H, s, C(=O)CHC*H*), 2.97 (1H, dd, *J* = 18.3, 5.5 Hz, C(=O)CH_AH_B), 3.90-4.17 (5H, m, H4, H5a, H5b, C*H*O*H*), 4.29 (1H,

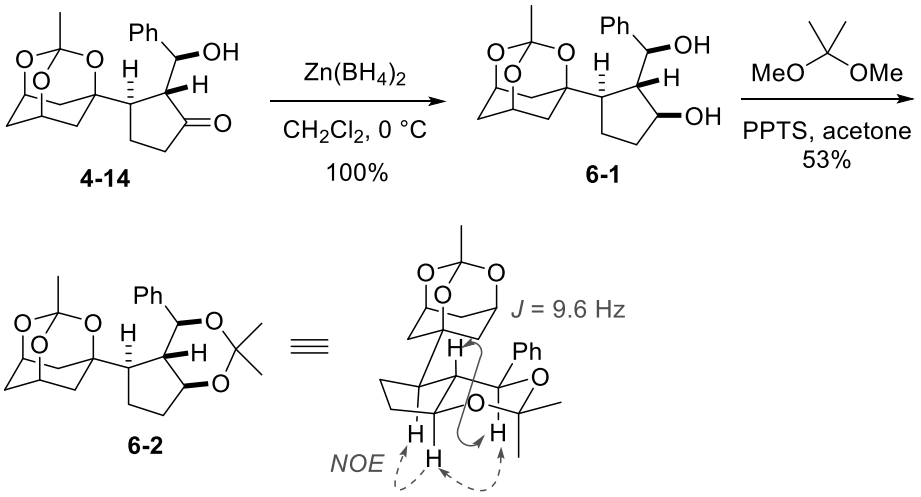
d, *J* = 2.3 Hz, H3), 4.39 (1H, s, H2), 4.85 (1H, d, *J* = 5.5 Hz, C*H*(OTBS)); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.80, -4.77, 17.8, 18.5, 21.7, 23.6, 24.3, 25.7, 26.7, 27.5, 28.9, 43.9, 49.0, 53.9, 57.0, 60.1, 70.1, 71.2, 72.0, 73.6, 86.1, 97.3, 111.4, 115.3, 220.5; HRMS (ESI-TOF) calcd for $C_{27}H_{48}NaO_8Si$ [M+Na]$^+$ 551.3011, found 551.3020.



Compound 4-49 [DK-5-199, DK-6-096]. According to the general procedure H, **4-49** (46 mg, 0.087 mmol) was synthesized from telluride **3-13** (50 mg, 0.11 mmol), (*R*)-4-(*tert*-butyldimethylsilyloxy)-2-cyclopentene-1-one (18 μL, 0.22 mmol) and 3-Methyl-2-butenal (31 μL, 0.33 mmol) in 79% yield by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (1.1 mL). The residue was purified by flash column chromatography on silica gel (hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{24}$ = 0.73 (*c* 2.3, $CHCl_3$); IR (film) 3487, 2990, 2952, 2931, 2858, 1725, 1453, 1377, 1453, 1377, 1249, 1193, 1120, 1065, 1007 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.07 (3H, s, TBS), 0.08 (3H, s, TBS), 0.87 (9H, s, TBS), 1.29 (3H, s, acetonide), 1.33 (3H, s, acetonide), 1.42 (3H, s, acetonide), 1.45 (3H, s, acetonide), 1.71 (3H, s, CH=C(C*H*$_3$)$_2$), 1.72 (3H, s, CH=C(C*H*$_3$)$_2$), 2.19 (1H, d, *J* = 18.3 Hz, C(=O)C*H*$_A$H$_B$), 2.43 (1H, s, C(=O)CHC*H*), 2.57 (1H, d, *J* = 8.2 Hz, C(=O)C*H*), 3.09 (1H, dd, *J* = 17.8, 5.5 Hz, C(=O)CH$_A$*H*$_B$), 3.93 (1H, d, *J* = 13.7 Hz, H5a), 3.96 (1H, m, H4), 4.04 (1H, dd, *J* = 13.8, 2.3 Hz, H5b), 4.14 (1H, s, OH), 4.25 (1H, d, *J* = 2.3 Hz, H3), 4.40 (1H, s, H2), 4.63 (1H, dd, *J* = 8.7, 8.7 Hz, C*H*OH), 4.81 (1H, d, *J* = 5.5 Hz, C*H*(OTBS)), 5.25 (1H, d, *J* = 8.7 Hz, C*H*=C(CH$_3$)$_2$); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.6, 17.9, 18.5, 18.6, 25.8, 25.9, 26.5, 27.4, 28.9, 49.1, 53.5, 56.4, 60.0, 69.6, 70.3, 71.6, 73.9, 86.4, 97.3, 111.1, 115.0, 125.5, 136.6, 222.2; HRMS (ESI-TOF) calcd for $C_{27}H_{46}NaO_8Si$ [M+Na]$^+$ 549.2854, found 549.2835.

6-4. 構造決定

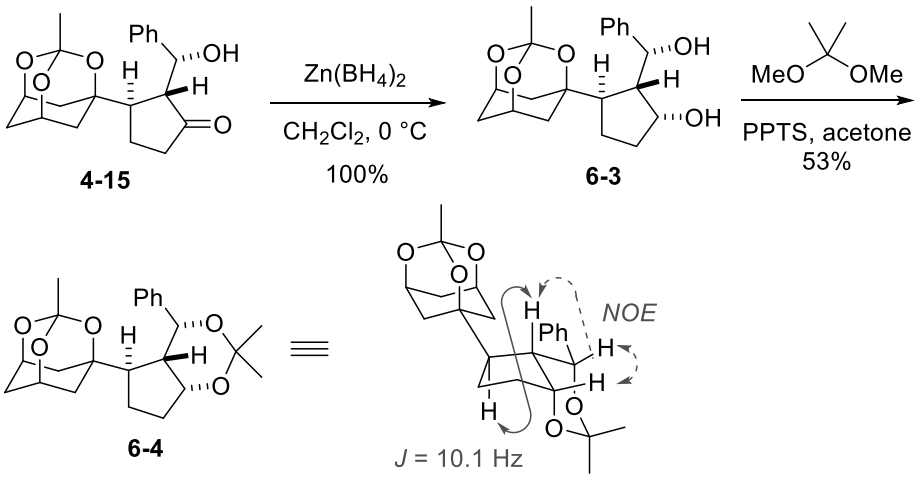
6-4-1. stereochemistry of **4-14**



Acetonide 6-2 [DK-4-167]. *General procedure I*: A suspension of $ZnCl_2$[14] (54 mg, 0.40 mmol) and $NaBH_4$ (30 mg, 0.79 mmol) in THF (1.6 mL) was stirred at room temperature for 2 h. A solution of compound **4-14** (27.4 mg, 0.0796 mmol) in CH_2Cl_2 (4.0 mL) was added to the suspension at 0 °C. The reaction mixture was stirred at 0 °C for 1 h, and then saturated aqueous NH_4Cl (10 mL) was added. The resultant mixture was extracted with EtOAc (10 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:2) to afford alcohol **6-1** (27.5 mg, 0.0793 mmol) in 100% yield.

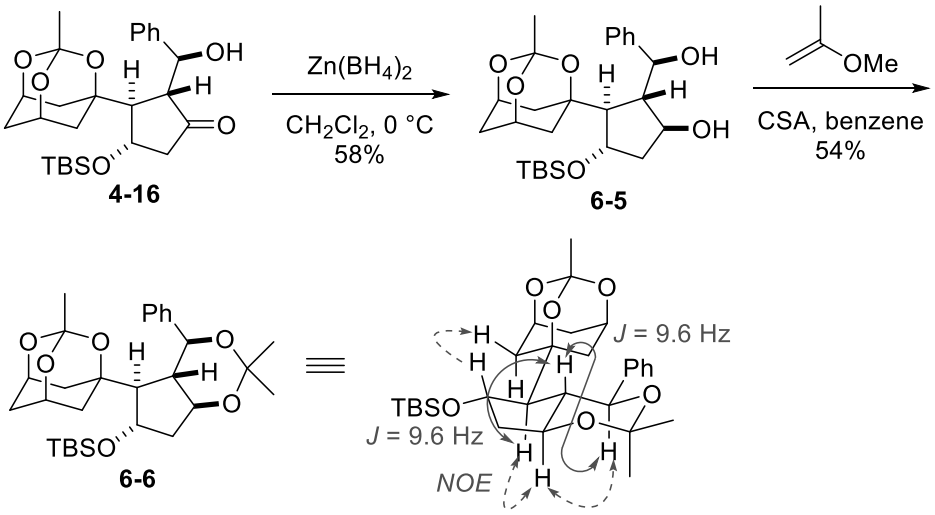
A mixture of **6-1** (27.5 mg, 0.0793 mmol) and pyridinium *p*-toluenesulfonate (PPTS, 4.0 mg, 0.016 mmol) in a mixture of dimethoxypropane (1.6 mL) and acetone (1.6 mL) was stirred at room temperature for 2 h, and was concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 4:1) to afford acetonide **6-2** (16 mg, 0.041 mmol) in 53% yield: ^{1}H NMR (400 MHz, C_6D_6) δ 0.12 (1H, dd, *J* = 12.8, 1.8 Hz, COCH_{ax}H$_{eq}$CHO), 0.66 (1H, dt, *J* = 12.8, 1.4 Hz, COCH_{ax}H$_{eq}$CHO), 0.68 (1H, dd, *J* = 12.8, 1.4 Hz, COCH_{ax}H$_{eq}$CHO), 1.40 (3H, s, CH_3), 1.40-1.69 (5H, m, COCH$_{ax}H_{eq}$CHO, CHOC*H*CHOPh, CHOCHC*H*, CHOCH_AH$_B$CH_AH$_B$), 1.50 (3H, s, CH_3), 1.62 (3H, s, CH_3), 1.73 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, COCH$_{ax}H_{eq}$CHO), 1.77-1.92 (2H, m, CHOCH$_A H_B$CH$_A H_B$), 2.11 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, COCH$_{ax}H_{eq}$CHO), 3.61 (1H, m, CH$_2$C*H*OCHCH), 3.81 (1H, m, CH$_2$C*H*OCH$_2$), 3.94 (1H, m, CH$_2$C*H*OCH$_2$), 4.51 (1H, d, *J* = 9.6 Hz, CHC*H*OPh), 6.98-7.09 (3H, m, aromatic), 7.22-7.28 (2H, d, *J* = 6.9 Hz, aromatic); ^{13}C NMR (100 MHz, C_6D_6) δ 20.3, 21.0, 26.6, 29.6, 30.5, 32.4, 32.8, 35.8, 46.5, 48.4, 67.81, 67.84, 74.4, 76.9, 81.2, 99.8, 110.7, 128.2, 128.4, 128.6, 142.5; HRMS (ESI-TOF) calcd for $C_{23}H_{30}NaO_5$ $[M+Na]^+$ 409.1985, found 409.1989.

6-4-2. stereochemistry of **4-15**



Acetonide 6-4 [DK-5-009]. According to the general procedure I, **6-4** (7.4 mg, 0.019 mmol) was synthesized from **4-15** (9.3 mg, 0.027 mmol) in 73% yield over 2 steps by using $ZnCl_2$ (18 mg, 0.13 mmol) and $NaBH_4$ (9.8 mg, 0.26 mmol) in THF (0.52 mL) and CH_2Cl_2 (1.3 mL) for the first reaction, and PPTS (2.0 mg) in dimethoxypropane (0.52 mL) and acetone (0.52 mL) for the second reaction: ^{1}H NMR (400 MHz, C_6D_6) δ -0.17 (1H, d, *J* = 12.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.58 (1H, d, *J* = 13.3 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.70 (1H, d, *J* = 12.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.36 (3H, s, C*H*$_3$), 1.53-1.65 (2H, m, COCH$_{ax}$*H*$_{eq}$CHO, CHOC*H*$_A$H$_B$CH$_2$), 1.55 (3H, s, C*H*$_3$), 1.57 (3H, s, C*H*$_3$), 1.78-1.94 (3H, m, CHOCH$_A$*H*$_B$CH$_2$, CHOC*H*CH, COCH$_{ax}$*H*$_{eq}$CHO), 2.00 (1H, dd, *J* = 12.8, 6.9 Hz, CHOCH$_2$C*H*$_A$H$_B$), 2.09-2.17 (1H, m, CHOCH$_{ax}$*H*$_{eq}$CHO), 2.12 (1H, dd, *J* = 10.1, 6.4 Hz, CHOCHC*H*), 2.28 (1H, tdd, *J* = 12.8, 10.1, 6.4 Hz, CHOCH$_2$CH$_A$*H*$_B$), 3.87-3.95 (2H, m, CH$_2$C*H*OCH$_2$ x2), 4.31 (1H, t, *J* = 3.2 Hz, CH$_2$C*H*OCHCH), 5.08 (1H, d, *J* = 3.7 Hz, CHC*H*OPh), 7.01 (1H, t, *J* = 7.3 Hz, aromatic), 7.09 (2H, t. *J* = 7.3 Hz, aromatic), 7.25 (2H, d, *J* = 7.3 Hz, aromatic); ^{13}C NMR (100 MHz, C_6D_6) δ 19.9, 26.4, 30.4, 33.1, 34.4, 35.1, 35.8, 43.8, 44.7 69.4, 69.5, 72.9, 76.0, 76.4, 99.0, 111.3, 128.5, 128.8, 129.1, 143.5; HRMS (ESI-TOF) calcd for $C_{23}H_{30}NaO_5$ $[M+Na]^+$ 409.1985, found. 409.1979.

6-4-3. stereochemistry of **4-16**

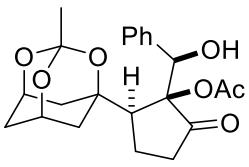


Acetonide 6-6 [DK-4-137]. A suspension of $ZnCl_2$ (58 mg, 0.43 mmol) and $NaBH_4$ (32 mg, 0.85 mmol) in THF (1.7

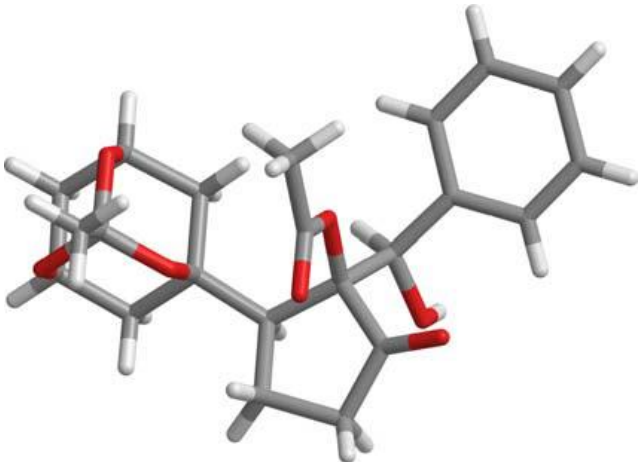
mL) was stirred at room temperature for 2 h. A solution of compound **4-16** (41 mg, 0.086 mmol) in CH_2Cl_2 (4.3 mL) was added to the suspension at 0 °C. The reaction mixture was stirred at 0 °C for 1 h, and then saturated aqueous NH_4Cl (10 mL) was added. The resultant mixture was extracted with EtOAc (10 mL x2), and the combined organic layers were washed with brine (20 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:1) to afford alcohol **6-5** (24 mg, 0.050 mmol) in 58% yield.

A mixture of **6-5** (24 mg, 0.050 mmol), 2-methoxypropene (48 µL, 0.51 mmol) and (+)-CSA (1.2 mg, 5.1 µmol) in benzene (1.6 mL) was stirred at room temperature for 30 min, and was then concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 10:1 to 5:1) to afford acetonide **6-6** (14 mg, 0.027 mmol) in 54% yield: ^{1}H NMR (400 MHz, C_6D_6) δ -0.05 (1H, d, *J* = 13.3 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.14 (3H, s, C*H*$_3$ of TBS), 0.21 (3H, s, C*H*$_3$ of TBS), 0.60 (1H, d, *J* = 11.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.65 (1H, d, *J* = 13.3 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.04 (9H, s, *t*-Bu of TBS), 1.37 (1H, d, *J* = 12.4 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 1.46-1.71 (3H, m, COCH$_{ax}$*H*$_{eq}$CHO, CHOC*H*$_A$H$_B$CHOTBS, CHOC*H*CH), 1.48 (3H, s, C*H*$_3$), 1.60 (3H, s, C*H*$_3$), 1.67 (3H, s, C*H*$_3$), 1.88 (1H, d, *J* = 9.6 Hz, CHOCHC*H*), 2.08 (1H, d, *J* = 12.4 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 2.15 (1H, dd, *J* = 11.9, 5.5 Hz, CHOCH$_A$*H*$_B$CH$_2$), 3.76 (1H, m, CH$_2$C*H*OCH$_2$), 3.87 (1H, m, CH$_2$C*H*OCH$_2$), 4.51-4.62 (2H, m, CH$_2$C*H*OCHCHOPh, CH$_2$C*H*(OTBS)), 4.78 (1H, d, *J* = 9.6 Hz, CHC*H*OPh), 6.94-7.04 (3H, m, aromatic), 7.13-7.24 (2H, m, aromatic); ^{13}C NMR (100 MHz,C_6D_6) δ -4.5, -4.1, 18.1, 20.3, 26.2, 26.8, 30.5, 32.1, 32.8, 36.5, 41.2, 49.3, 58.3, 67.8, 67.9, 70.3, 74.0, 75.2, 81.4, 100.4, 110.8, 128.1, 128.2, 128.4, 142.5; HRMS (ESI-TOF) calcd for $C_{29}H_{44}NaO_6Si$ $[M+Na]^+$ 539.2799, found 539.2777.

6-4-4. stereochemistry of **4-17**

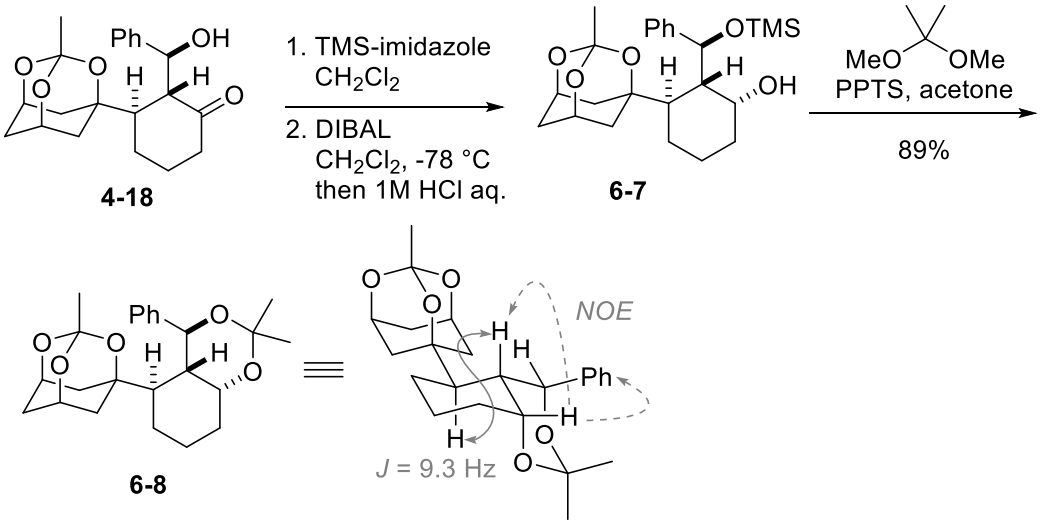


4-17



X-ray crystallographic analysis of compound **4-17**: CCDC 952270[15)]

6-4-5. stereochemistry of **4-18**

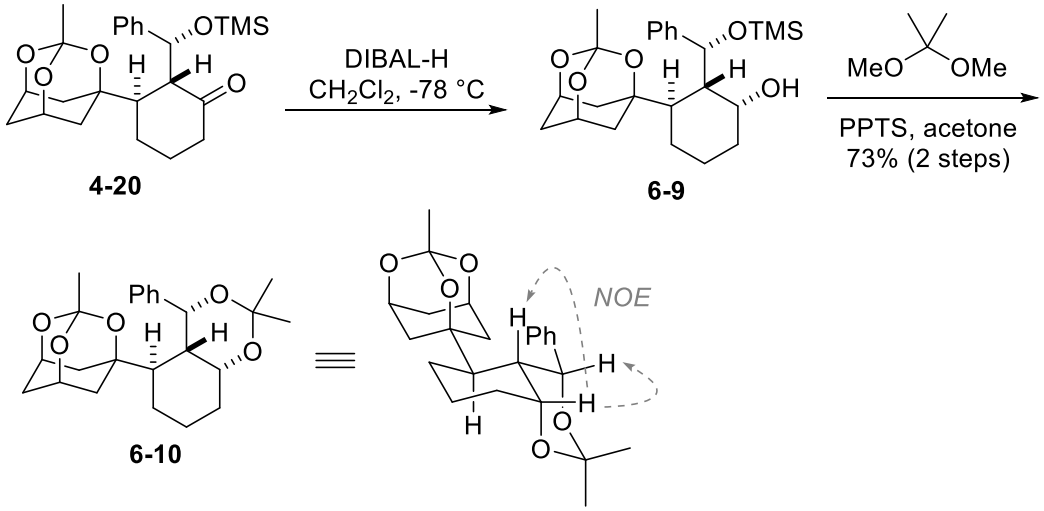


Acetonide 6-8 [DK-4-187]. *General procedure J*: A mixture of **4-18** (9.0 mg, 0.025 mmol), TMS-imidazole (18 µL, 0.13 mmol) and DMAP (1.5 mg, 0.013 mmol) in CH_2Cl_2 (0.50 mL) was stirred at 0°C for 3 h, and then saturated aqueous $NaHCO_3$ (10 mL) was added. The resultant mixture was extracted with EtOAc (5 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 2:1) to afford **4-19** (10 mg, 0.023 mmol) in 92% yield.

DIBAL-H (1.5 M in toluene, 58 µL, 0.087 mmol) was added to a solution of **4-19** (31 mg, 0.072 mmol) in CH_2Cl_2 (1.4 mL) at -78 °C. The reaction mixture was stirred at -78 °C for 1 h, and DIBAL-H (58 µL, 0.087 mmol) was added. The reaction mixture was stirred at -78 °C for 15 min, and then 1M HCl (10 mL) was added. After 1h, the resultant mixture was extracted with EtOAc (10 mL x2). The combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 2:1 to 1:1) to afford the alcohol **6-7** (23 mg, 0.063 mmol) in 74% yield.

A solution of the above alcohol **6-7** (23 mg, 0.053 mmol) and PPTS (3.3 mg, 0.013 mmol) in a mixture of dimethoxypropane (1.3 mL) and acetone (1.3 mL) was stirred at room temperature for 5 h, and was then concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5:1) to afford acetonide **6-8** (23 mg, 0.057 mmol) in 89% yield: ^{1}H NMR (400 MHz, C_6D_6) δ 0.71-0.75 (1H, m, $CHOCH_2CH_2C\mathit{H}_A\mathit{H}_B$), 0.75 (2H, dd, J = 12.8, 1.8 Hz, $COC\mathit{H}_{ax}\mathit{H}_{eq}CHO$ x2), 0.96 (1H, dd, J = 12.8, 1.8 Hz, $COC\mathit{H}_{ax}\mathit{H}_{eq}CHO$), 1.42-1.51 (3H, m, $CHOC\mathit{H}_A\mathit{H}_BC\mathit{H}_A\mathit{H}_BCH_A\mathit{H}_B$), 1.48 (3H, s, $C\mathit{H}_3$), 1.58 (3H, s, $C\mathit{H}_3$), 1.62 (3H, s, $C\mathit{H}_3$), 1.73-1.92 (3H, m, $CHOCH_2CH_A\mathit{H}_B$, $COCH_{ax}\mathit{H}_{eq}CHO$ x2), 1.96-2.08 (2H, m, $CHOCHC\mathit{H}$, $CHOCH_A\mathit{H}_BCH_2$), 2.14 (1H, ddd, J = 9.2, 5.5, 3.6 Hz, $CHOC\mathit{H}CHOPh$), 2.18 (1H, m, $COCH_{ax}\mathit{H}_{eq}CHO$), 3.93 (1H, m, $CH_2C\mathit{H}OCH_2$), 3.99 (1H, m, $CH_2C\mathit{H}OCH_2$), 4.54 (1H, dt, J = 3.6, 3.6 Hz, $CH_2C\mathit{H}OCHCH$), 5.58 (1H, d, J = 5.5 Hz, $CHC\mathit{H}OPh$), 7.11-7.18 (1H, m, aromatic), 7.27 (2H, t, J = 7.3 Hz, aromatic), 7.63 (2H, dd, J = 7.3, 1.4 Hz, aromatic); ^{13}C NMR (100 MHz, $CO(CD_3)_2$) δ 21.1, 24.9, 25.5, 26.8, 28.5, 30.9, 33.2, 33.4, 37.5, 43.9, 46.0, 67.1, 68.9, 69.1, 74.4, 77.4, 100.3, 110.8, 128.1, 128.9, 129.1, 144.5; HRMS (ESI-TOF) calcd for $C_{24}H_{32}NaO_5$ $[M+Na]^+$ 423.2142, found 423.2130.

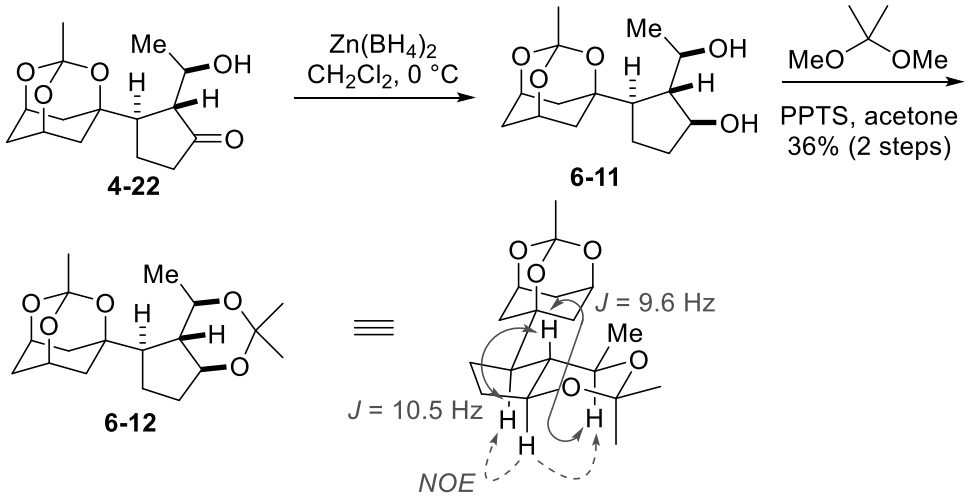
6-4-6. stereochemistry of **4-20**



Acetonide 6-10 [DK-4-191]. DIBAL-H (1.5 M in toluene, 72 µL, 0.11 mmol) was added to a solution of **4-20** (19 mg, 0.044 mmol) in CH_2Cl_2 (0.86 mL) at -78 °C. The reaction mixture was stirred at -78 °C for 1 h, and then 1M HCl (10 mL) was added. After 1 h, the resultant mixture was extracted with EtOAc (10 mL x2). The combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated to afford the crude alcohol **6-9**, which was used in the next reaction without further purification.

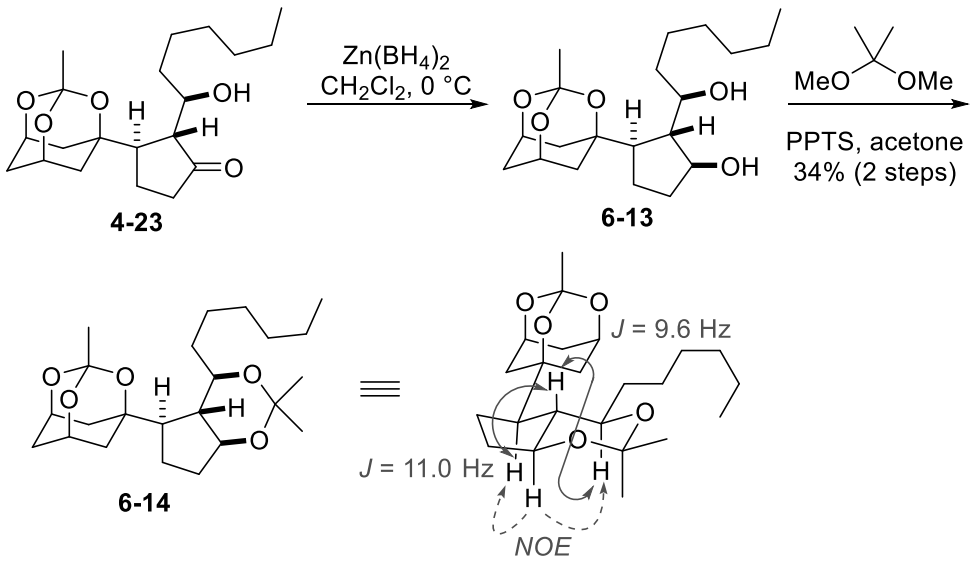
A solution of the above crude alcohol **6-9** and PPTS (2.0 mg, 8.0 µmol) in dimethoxypropane (0.82 mL) and acetone (0.82 mL) was stirred at room temperature for 12 h, and was then concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 5:1) to afford alcohol **6-10** (13 mg, 0.032 mmol) in 73% yield over 2 steps: ^{1}H NMR (400 MHz, C_6D_6) δ -0.06 (1H, dd, *J* = 13.3, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.65 (1H, dd, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.67 (1H, dt, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.39 (3H, s, C*H*$_3$), 1.56 (3H, s, C*H*$_3$), 1.56-1.63 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 1.62 (3H, s, C*H*$_3$), 1.69-1.90 (6H, m, CHOC*H*CHOPh, CHOCHC*H*, CHOC*H*$_A$H$_B$C*H*$_A$H$_B$C*H*$_2$), 1.93-2.17 (3H, m, CHOCH$_A$*H*$_B$CH$_2$, COCH$_{ax}$*H*$_{eq}$CHO x2), 2.43-2.59 (1H, m, CHOCH$_2$CH$_A$*H*$_B$CH$_2$), 3.90 (1H, m, CH$_2$C*H*OCH$_2$), 3.95 (1H, m, CH$_2$C*H*OCH$_2$), 4.18 (1H, m, CH$_2$C*H*OCH), 5.08 (1H, d, *J* = 2.7 Hz, CHC*H*OPh), 7.05 (1H, tt, *J* = 7.3, 1.4 Hz, aromatic), 7.11-7.18 (2H, m, aromatic), 7.24 (2H, dd, *J* = 7.3, 1.4 Hz, aromatic); ^{13}C NMR (100 MHz, C_6D_6) δ 16.6, 19.4, 19.5, 26.1, 26.8, 30.8, 32.3, 34.7, 34.9, 35.5, 38.0, 68.0, 68.1, 69.4, 74.5, 75.3, 98.7, 110.6, 127.2, 127.9, 128.0, 141.2; HRMS (ESI-TOF) calcd for $C_{24}H_{32}NaO_5$ $[M+Na]^+$ 423.2142, found 423.2131.

6-4-7. stereochemistry of **4-22**



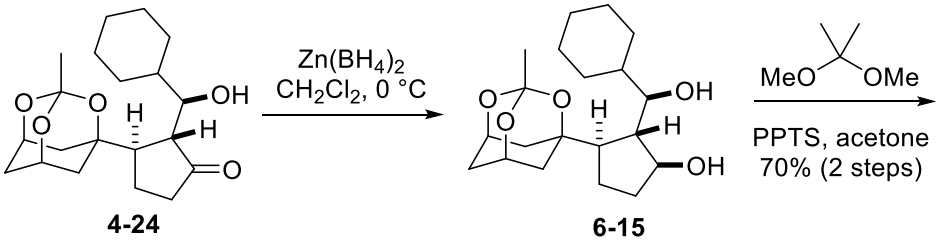
Acetonide 6-12 [DK-4-078]. According to the general procedure I, **6-12** (21 mg, 0.065 mmol) was synthesized from **4-22** (51 mg, 0.18 mmol) in 36% yield over 2 steps by using $ZnCl_2$ (83 mg, 0.61 mmol) and $NaBH_4$ (45 mg, 1.2 mmol) in THF (1.2 mL) and CH_2Cl_2 (2.4 mL) for the first reaction, and PPTS (2.2 mg) in dimethoxypropane (1.8 mL) and acetone (1.8 mL) for the second reaction: ^{1}H NMR (400 MHz, C_6D_6) δ 0.78-0.87 (1H, m, CHOCH$_2$C*H*$_A$H$_B$), 0.80 (1H, d, *J* = 11.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.83 (1H, d, *J* = 11.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.99 (1H, dd, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.16 (1H, ddd, *J* = 18.3, 13.7, 9.2 Hz, CHOCH$_2$CH$_A$*H*$_B$), 1.27-1.49 (3H, m, CHOC*H*C*H*CH$_2$, CHOC*H*$_A$H$_B$CH$_2$), 1.39 (3H, s, C*H*$_3$), 1.63 (3H, s, C*H*$_3$), 1.64 (3H, s, C*H*$_3$), 1.69 (3H, d, *J* = 6.4 Hz, CHOC*H*$_3$), 1.72-1.82 (2H, m, COCH$_{ax}$*H*$_{eq}$CHO, CHOCH$_A$*H*$_B$CH$_2$), 2.00 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.26 (1H, dtt, *J* = 13.3, 1.8, 1.8 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 3.47 (1H, td, *J* = 10.5, 6.9 Hz, CH$_2$C*H*OCHCH), 3.86 (1H, dq, *J* = 9.6, 6.4 Hz, C*H*OCH$_3$), 4.04 (2H, m, CH$_2$C*H*OCH$_2$ x2); ^{13}C NMR (100 MHz, C_6D_6) δ 20.4, 20.6, 22.9, 26.4, 28.2, 30.6, 31.0, 32.6, 37.1, 47.6, 48.6, 67.95, 68.04, 73.7, 74.1, 75.6, 98.9, 110.6; HRMS (ESI-TOF) calcd for $C_{18}H_{28}NaO_5$ $[M+Na]^+$ 347.1829, found 347.1840.

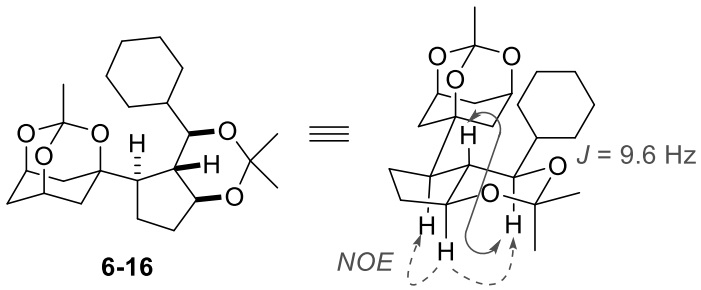
6-4-8. stereochemistry of **4-23**



Acetonide 6-14 [DK-3-098]. According to the general procedure I, **6-14** (9.1 mg, 0.023 mmol) was synthesized from **4-23** (24 mg, 0.68 mmol) in 34% yield over 2 steps by using $ZnCl_2$ (68 mg, 0.50 mmol) and $NaBH_4$ (38 mg, 1.0 mmol) in THF (1.0 mL) and CH_2Cl_2 (1.4 mL) for the first reaction, and PPTS (0.8 mg, 3 μmol) in dimethoxypropane (0.58 mL) and acetone (0.58 mL) for the second reaction: ^{1}H NMR (400 MHz, C_6D_6) δ 0.82 (1H, d, *J* = 12.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.84 (1H, dt, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.89 (3H, t, *J* = 7.3 Hz, CH$_2$C*H*$_3$), 0.80-0.90 (1H, m, CHOCH$_2$C*H*$_A$H$_B$), 1.01 (1H, dd, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.19 (1H, ddd, *J* = 18.3, 13.3, 8.7 Hz, CHOCH$_2$CH$_A$*H*$_B$), 1.27-1.62 (10H, m, C*H*$_2$ of *n*-hexyl x4, CHOC*H*C*H*CH$_2$), 1.42 (3H, s, C*H*$_3$), 1.65 (3H, s, C*H*$_3$), 1.71 (3H, s, C*H*$_3$), 1.72-1.94 (4H, m, C*H*$_2$ of *n*-hexyl, COCH$_{ax}$*H*$_{eq}$CHO, CHOC*H*$_A$H$_B$CH$_2$), 2.04 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.26 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 2.58 (1H, m, CHOCH$_A$*H*$_B$CH$_2$), 3.51 (1H, td, *J* = 11.0, 6.9 Hz, C*H*OCH$_2$CH$_2$), 3.75 (1H, td, *J* = 9.6, 2.3 Hz, C*H*O-hexyl), 4.05 (2H, m, CH$_2$C*H*OCH$_2$ x2); ^{13}C NMR (100 MHz, C_6D_6) δ 14.4, 20.3, 20.8, 23.2, 26.48, 26.52, 28.3, 30.1, 30.6, 30.9, 32.61, 32.64, 35.7, 37.2, 47.2, 47.5, 68.0, 68.1, 74.2, 76.0, 77.6, 99.0, 110.6; HRMS (ESI-TOF) calcd for $C_{23}H_{38}NaO_5$ [M+Na]$^+$ 417.2611, found 417.2595.

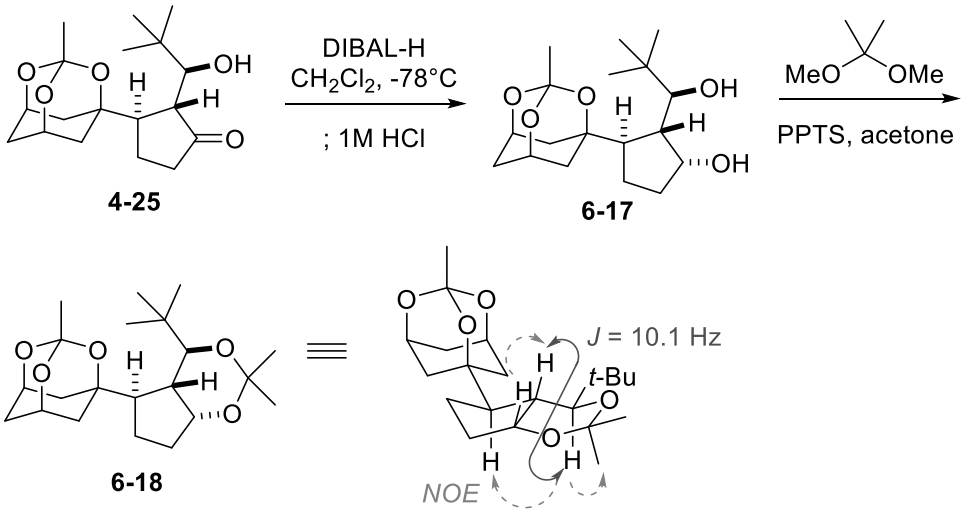
6-4-9. stereochemistry of **4-24**





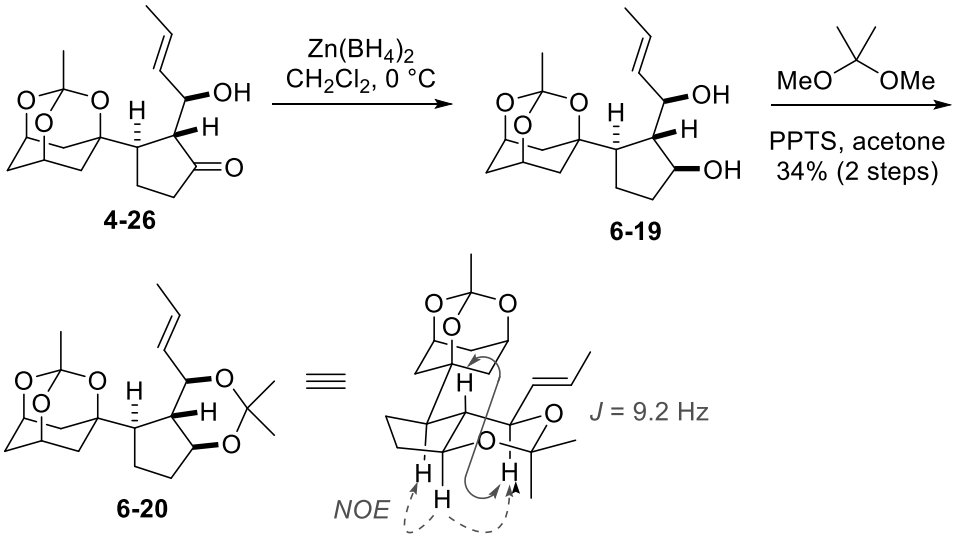
Acetonide 6-16 [DK-4-084]. According to the general procedure I, **6-16** (11 mg, 0.028 mmol) was synthesized from **4-24** (14 mg, 0.040 mmol) in 70% yield over 2 steps by using $ZnCl_2$ (27 mg, 0.20 mmol) and $NaBH_4$ (16 mg, 0.40 mmol) in THF (0.81 mL) and CH_2Cl_2 (2.0 mL) for the first reaction, and PPTS (0.7 mg, 3 μmol) in dimethoxypropane (0.60 mL) and acetone (0.60 mL) for the second reaction: ^{1}H NMR (400 MHz, C_6D_6) δ 0.77-0.91 (3H, m, COC*H*$_{ax}$H$_{eq}$CHO x2, C*H* of cyclohexyl), 1.02 (1H, dd, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.11-1.23 (1H, m, C*H* of cyclohexyl), 1.23-2.00 (15H, m, C*H* of cyclohexyl x 9, CH$_2$CHOC*H*C*H*CH$_2$, CHOC*H*$_A$H$_B$C*H*$_2$, COCH$_{ax}$*H*$_{eq}$CHO), 1.38 (3H, s, C*H*$_3$), 1.61 (3H, s, C*H*$_3$), 1.74 (3H, s, C*H*$_3$), 2.04 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.27 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 2.61 (1H, m, CHOCH$_A$*H*$_B$CH$_2$), 3.47 (1H, m, CH$_2$C*H*OCHCH), 3.60 (1H, d, *J* = 8.7 Hz, CHC*H*O-cyclohexyl), 4.03-4.08 (2H, m, CH$_2$C*H*OCH$_2$ x2); ^{13}C NMR (125 MHz, C_6D_6) δ 20.0, 20.7, 26.4, 26.7, 27.0, 27.3, 27.6, 27.9, 30.4, 30.9, 31.7, 32.6, 37.2, 39.1, 43.2, 48.3, 68.0, 68.1, 74.3, 75.9, 81.4, 98.7, 110.6; HRMS (ESI-TOF) calcd for $C_{23}H_{36}NaO_5$ [M+Na]$^+$ 415.2455, found 415.2474.

6-4-10. stereochemistry of **4-25**



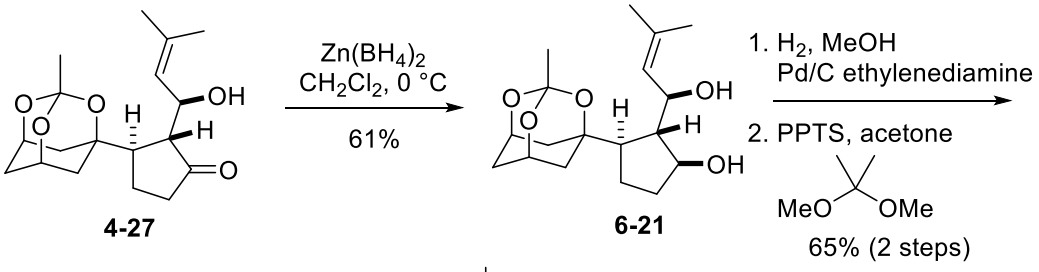
Acetonide 6-18 [DK-5-010]. According to the general procedure J, **6-18** (15 mg, 0.041 mmol) was synthesized from **4-25** (20 mg, 0.062 mmol) in 66% yield over 3 steps by using TMS-imidazole (44 mL, 0.030 mmol) and DMAP (2.2 mg, 0.018 mmol) in CH_2Cl_2 (1.2 mL) for the first reaction, and DIBAL-H (1.5 M in toluene, 72 µL, 0.11 mmol) in CH_2Cl_2 (2.2 mL) for the second reaction, and PPTS (2.2 mg, 8.6 µmol) in dimethoxypropane (0.86 mL) and acetone (0.86 mL) for the third reaction: ^{1}H NMR (400 MHz, C_6D_6) δ 0.70 (1H, dd, *J* = 12.4, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.85 (1H, dt, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.07 (1H, dd, *J* = 12.8, 6.8 Hz, CHOCH$_2$C*H*$_A$H$_B$), 1.19-1.35 (2H, m, CHOC*H*$_A$H$_B$, COC*H*$_{ax}$H$_{eq}$CHO), 1.24 (9H, s, *t*-Bu), 1.38 (3H, s, C*H*$_3$), 1.43 (3H, s, C*H*$_3$), 1.66 (3H, s, C*H*$_3$), 1.64-1.79 (3H, m, CHOCH$_A$*H*$_B$, COCH$_{ax}$*H*$_{eq}$CHO x2), 1.84-1.97 (1H, m, CHOCH$_2$CH$_A$*H*$_B$), 1.99 (1H, dd, *J* = 9.2, 2.8 Hz, CHOCHC*H*), 2.27 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 2.74 (1H, ddd, *J* = 10.1, 5.9, 2.7 Hz, CHOC*H*CH), 3.24 (1H, d, *J* = 10.1 Hz, CHC*H*O*t*Bu), 3.99-4.07 (2H, m, CH$_2$C*H*OCH$_2$ x2), 4.19 (1H, t, *J* = 6.0 Hz, CH$_2$C*H*OCH); ^{13}C NMR (100 MHz, C_6D_6) δ 24.4, 24.6, 25.9, 26.5, 27.2, 32.5, 33.2 (two peaks), 34.9, 38.3, 44.3, 52.0, 67.8, 68.2, 74.7, 75.1, 78.5, 99.0, 110.5; HRMS (DART-TOF) calcd for $C_{21}H_{35}O_5$ $[M+H]^+$ 367.2479, found 367.2464.

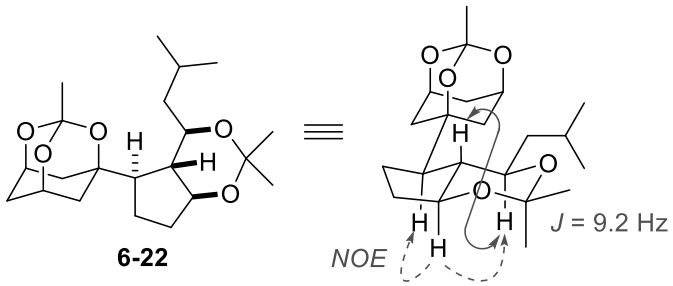
6-4-11. stereochemistry of **4-26**



Acetonide 6-20 [DK-4-102]. According to the general procedure I, **6-20** (7.6 mg, 0.022 mmol) was synthesized from **4-26** (20 mg, 0.065 mmol) in 34% yield over 2 steps by using $ZnCl_2$ (43 mg, 0.32 mmol) and $NaBH_4$ (24 mg, 0.64 mmol) in THF (1.3 mL) and CH_2Cl_2 (3.2 mL) for the first reaction, and PPTS (1.6 mg, 6.4 μmol) in dimethoxypropane (1.3 mL) and acetone (1.3 mL) for the second reaction: ^{1}H NMR (400 MHz, C_6D_6) δ 0.85 (1H, d, *J* = 12.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.94 (1H, d, *J* = 12.3 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.95 (1H, m, COC*H*$_{ax}$H$_{eq}$CHO), 1.06-1.16 (1H, m, CHOCH$_2$C*H*$_A$H$_B$), 1.18-1.30 (1H, m, CHOCH$_2$CH$_A$*H*$_B$), 1.39 (3H, s, C*H*$_3$), 1.39-1.48 (2H, m, CHOC*H*$_A$H$_B$CH$_2$, CHOC*H*CH), 1.53 (1H, td, *J* = 10.5, 7.3 Hz, CHOCHC*H*), 1.65 (3H, s, C*H*$_3$), 1.69 (3H, s, C*H*$_3$), 1.71 (3H, d, *J* = 6.4 Hz, CH=CHC*H*$_3$), 1.73-1.87 (2H, m, CHOCH$_A$*H*$_B$CH$_2$, COCH$_{ax}$*H*$_{eq}$CHO), 2.05 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.27 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 3.51 (1H, td, *J* = 11.0, 6.9 Hz, CH$_2$C*H*OCHCH), 4.06 (2H, m, CH$_2$C*H*OCH$_2$ x2), 4.19 (1H, dd, *J* = 9.2, 6.0 Hz, C*H*OCH=CH), 5.85 (1H, dq, *J* = 15.1, 6.4 Hz, CHOCH=C*H*CH$_3$), 6.13 (1H, ddd, *J* = 15.1, 6.0, 1.1 Hz, CHOC*H*=CHCH$_3$); ^{13}C NMR (125 MHz, C_6D_6) δ 18.0, 20.4, 20.8, 26.7, 28.5, 30.5, 32.7, 33.0, 36.1, 47.2, 47.9, 68.0 (two peaks), 74.3, 75.9, 77.4, 99.3, 110.7, 125.0, 133.6; HRMS (ESI-TOF) calcd for $C_{20}H_{30}NaO_5$ $[M+Na]^+$ 373.1985, found 373.1991.

6-4-12. stereochemistry of **4-27**



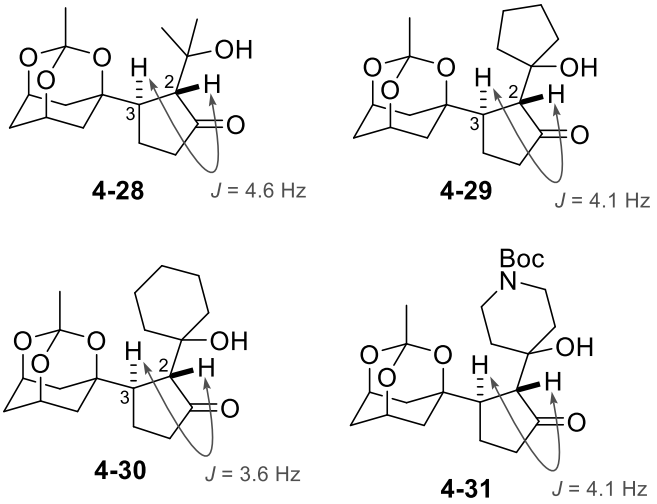


Acetonide 6-22 [DK-4-148]. A suspension of $ZnCl_2$ (39 mg, 0.29 mmol) and $NaBH_4$ (21 mg, 0.56 mmol) in THF (1.1 mL) was stirred at room temperature for 2 h. Then a solution of compound **4-27** (18 mg, 0.056 mmol) in CH_2Cl_2 (2.9 mL) was added to the suspension at 0 °C. The reaction mixture was stirred at 0 °C for 1 h, and saturated aqueous NH_4Cl (10 mL) was added. The resultant mixture was extracted with EtOAc (10 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 1:4) to afford diol **6-21** (11 mg, 0.034 mmol) in 61% yield.

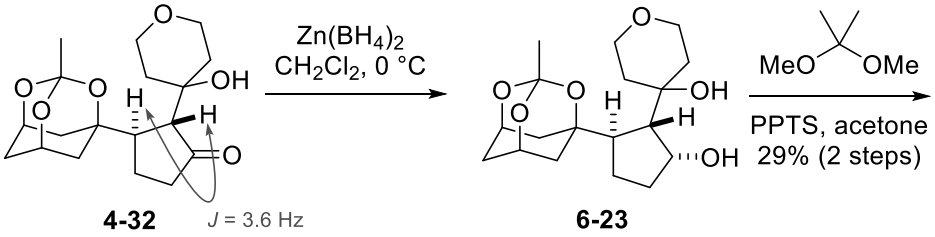
A suspension of **6-21** (11 mg, 0.034 mmol) and Pd/C ethylenediamine (Pd 3.5-6.5 %, 2.0 mg) in MeOH (1.8 mL) was exposed to H_2 atmosphere. The reaction mixture was stirred at room temperature for 12 h, and was then filtered through a pad of Celite. The filtrate was concentrated. The crude diol was used in the next reaction without further purification.

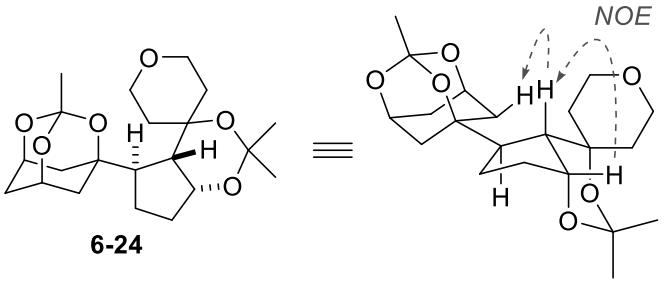
A mixture of the above crude alcohol and PPTS (1.8 mg, 7.2 μmol) in a mixture of dimethoxypropane (0.70 mL) and acetone (0.70 mL) was stirred at room temperature for 5 h, and then concentrated. The residue was purified by flash column chromatography on silica gel (10 g, hexane/EtOAc 4:1) to afford acetonide **6-22** (8.1 mg, 0.022 mmol) in 65% yield over 2 steps: ^{1}H NMR (400 MHz, C_6D_6) δ 0.79 (1H, dd, *J* = 11.0, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.82 (1H, d, *J* = 11.9 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.80-0.89 (1H, m, CHOCH$_2$C*H*$_A$H$_B$), 1.00 (1H, dd, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.07 (3H, d, *J* = 6.9 Hz, CHC*H*$_3$), 1.08 (3H, d, *J* = 6.9 Hz, CHC*H*$_3$), 1.12-1.24 (1H, m, CHOCH$_2$CH$_A$*H*$_B$), 1.32-1.51 (3H, m, CHOC*H*C*H*CH$_2$, CHOC*H*$_A$H$_B$CH$_2$,), 1.42 (3H, s, C*H*$_3$), 1.63 (3H, s, C*H*$_3$), 1.65-1.82 (3H, m, COCH$_{ax}$*H*$_{eq}$CHO, C*H*$_A$H$_B$CH(CH$_3$)$_2$, CHOCH$_A$*H*$_B$CH$_2$), 1.67 (3H, s, C*H*$_3$), 2.02 (1H, m, COCH$_{ax}$*H*$_{eq}$CHO), 2.14-2.30 (2H, m, C*H*(CH$_3$)$_2$, COCH$_{ax}$*H*$_{eq}$CHO), 2.39 (1H, ddd, *J* = 13.7, 10.5, 2.3 Hz, CH$_A$*H*$_B$CH(CH$_3$)$_2$), 3.52 (1H, td, *J* = 11.0, 11.0, 7.3 Hz, CH$_2$C*H*OCH), 3.85 (1H, td, *J* = 9.2, 2.3 Hz, CH$_2$C*H*OCH$_2$CH(CH$_3$)$_2$), 4.04 (2H, m, CH$_2$C*H*OCH$_2$ x2); ^{13}C NMR (100 MHz, C_6D_6) δ 20.2, 20.8, 21.4, 24.3, 24.6, 26.6, 28.3, 30.5, 30.9, 32.6, 37.2, 44.5, 47.4, 47.7, 67.9, 68.1, 74.2, 75.2, 76.0, 98.9, 110.6; HRMS (ESI-TOF) calcd for $C_{21}H_{34}NaO_5$ [M+Na]$^+$ 389.2298, found 389.2304.

6-4-13. stereochemistry of **4-28**, **4-29**, **4-30 and 4-31**



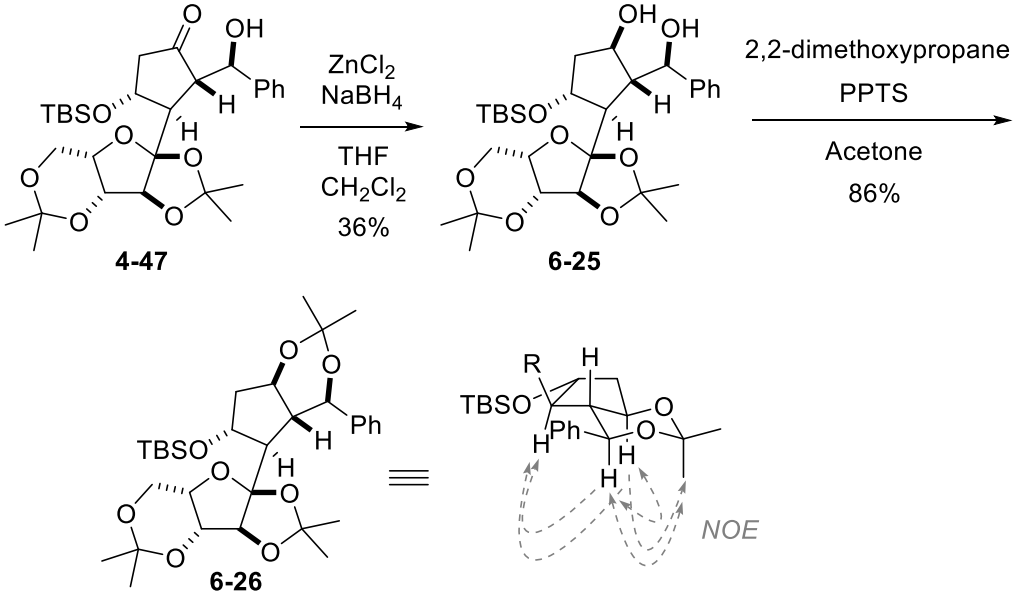
6-4-14. stereochemistry of **4-32**





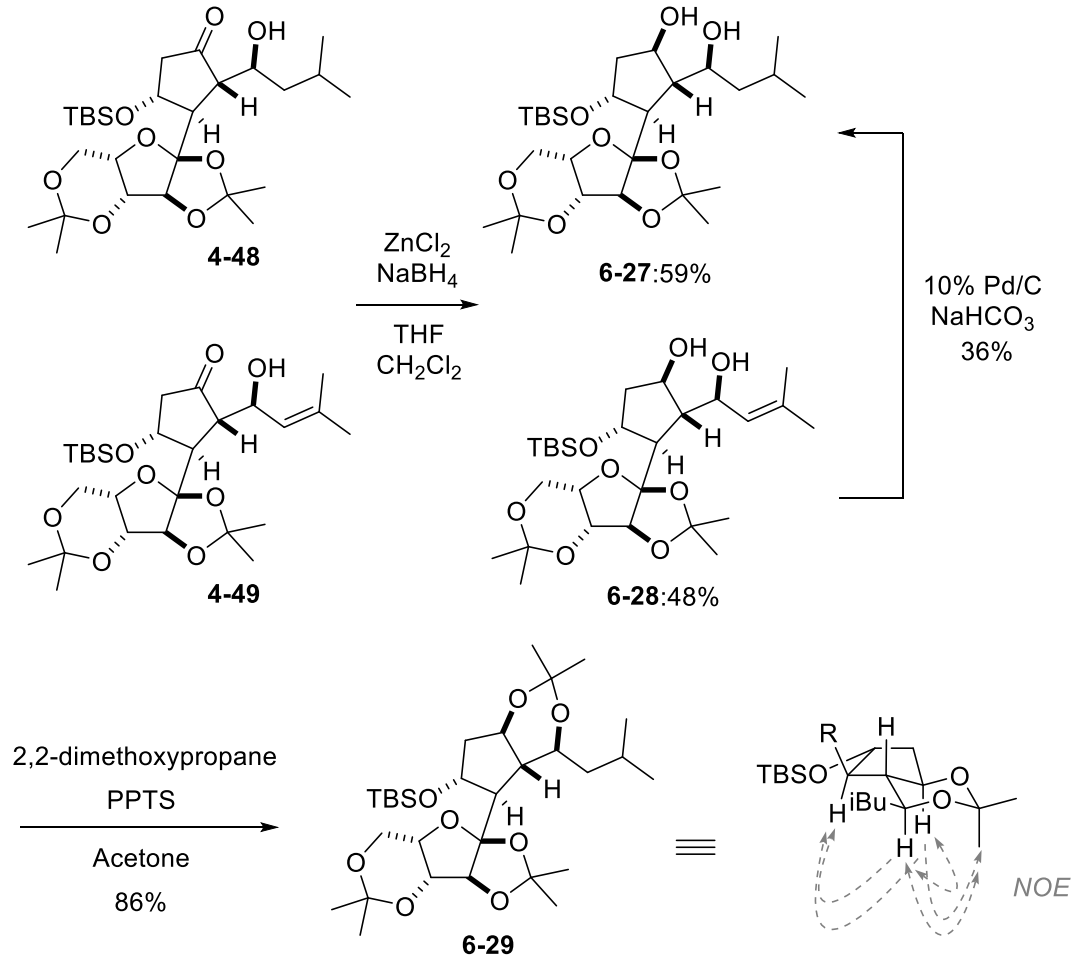
Acetonide 6-24 [DK-4-154]. According to the general procedure I, **6-24** (8.6 mg, 0.023 mmol) was synthesized from **4-32** (27 mg, 0.080 mmol) in 29% yield over 2 steps by using $ZnCl_2$ (54 mg, 0.40 mmol) and $NaBH_4$ (30 mg, 0.79 mmol) in THF (1.6 mL) and CH_2Cl_2 (4.0 mL) for the first reaction, and PPTS (1.8 mg, 7.0 μmol) in dimethoxypropane (0.7 mL) and acetone (0.7 mL) for the second reaction: ^{1}H NMR (400 MHz, C_6D_6) δ 0.80 (1H, dd, *J* = 12.4, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.89 (1H, dt, *J* = 12.8, 1.8 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 0.90 (1H, dd, *J* = 12.4, 1.4 Hz, COC*H*$_{ax}$H$_{eq}$CHO), 1.19-1.38 (2H, m, $CH_2OCH_2CH_AH_B$, $CHOCH_2CH_AH_B$), 1.40-1.55 (1H, m, $CHOCH_2CH_AH_B$), 1.42 (6H, s, C*H*$_3$ x2), 1.63 (3H, s, C*H*$_3$), 1.67-1.81 (3H, m, $CH_2OCH_2CH_ACH_B$, CHOCHC*H*, CHOC*H*$_A$H$_B$CH$_2$), 1.81-1.97 (3H, m, CHOCH$_A$*H*$_B$CH$_2$, COCH$_{ax}$*H*$_{eq}$CHO x2), 2.00-2.11 (2H, m, $CH_2OCH_2CH_ACH_B$ x2), 2.23 (1H, dd, *J* = 8.7, 3.2 Hz, CHOC*H*CH), 2.30 (1H, dtt, *J* = 12.8, 1.8, 1.8 Hz, COCH$_{ax}$*H*$_{eq}$CHO), 3.80-3.92 (2H, m, $CH_2OCH_2CH_2$), 3.99 (1H, ddd, *J* = 14.7, 11.9, 2.3 Hz, CH$_2$OC*H*$_A$H$_B$CH$_2$), 4.04-4.13 (3H, m, CH$_2$OCH$_A$*H*$_B$CH$_2$, CH$_2$C*H*OCH$_2$ x2), 4.30 (1H, td, *J* = 8.2, 5.5 Hz, CH$_2$C*H*OCHCH); ^{13}C NMR (100 MHz, C_6D_6) δ 23.2, 26.5, 26.6, 30.8, 32.6, 33.4, 33.5, 34.1, 36.4, 39.1, 47.7, 51.3, 63.8, 63.9, 67.9, 68.0, 73.2, 73.6, 74.1, 99.2, 110.7; HRMS (ESI-TOF) calcd for $C_{21}H_{32}NaO_6$ $[M+Na]^+$ 403.2091, found 403.2093.

6-4-15. stereochemistry of **4-47**



Acetonide 6-26 [DK-6-004]. According to the general procedure I, **6-26** (11 mg, 0.019 mmol) was synthesized from **4-47** (58 mg, 0.11 mmol) in 31% yield over 2 steps by using $ZnCl_2$ (72 mg, 0.53 mmol) and $NaBH_4$ (40 mg, 1.1 mmol) in THF (2.1 mL) and CH_2Cl_2 (5.3 mL) for the first reaction, and PPTS (2.8 mg, 11 μmol) in dimethoxypropane (0.44 mL) and acetone (0.44 mL) for the second reaction: Colorless solid; $[\alpha]_D^{25}$ = -21.9 (*c* 1.1, CH_2Cl_2); m.p. 170-175 °C; IR (film) 2990, 2935, 2890, 2857, 1461, 1380, 1259, 1244, 1197, 1186, 1171, 1123, 1067, 1037, 1010 cm^{-1}; ^1H NMR (400 MHz, Acetone-D_6) δ 0.08 (3H, s, TBS), 0.10 (3H, s, TBS), 0.82-1.05 (15H, s, TBS, acetonide x2), 1.29-1.40 (9H, m, acetonide x3), 1.51 (3H, s, acetonide), 1.61-1.73 (1H, m, C$H_A$$H_B$CH(OTBS)), 1.78 (1H, dd, *J* = 11.4, 5.5 Hz, CH$_A$$H_B$CH(OTBS)), 2.09 (1H, s, C*H*CH(OTBS)), 2.74-2.82 (2H, m, C*H*CHCH(OTBS), H2), 3.68 (1H, s, H3), 3.83 (1H, d, *J* = 13.3 Hz, H5a), 3.88-4.10 (2H, m, H4, H5b), 4.28 (1H, m, C*H*OCH_2), 4.57 (1H, d, *J* = 4.6 Hz, C*H*(OTBS), 4.83 (1H, d, *J* = 9.6 Hz, PhC*H*O), 7.16-7.28 (3H, m, aromatics), 7.28-7.36 (2H, m, aromatics); HRMS (ESI-TOF) calcd for $C_{32}H_{50}NaO_8Si$ $[M+Na]^+$ 613.3167, found 613.3176.

6-4-16. stereochemistry of **4-48** and **4-49**



Acetonide 6-29 [DK-6-108]. According to the general procedure I, **6-29** (11 mg, 0.019 mmol) was synthesized from **4-48** (42 mg, 0.079 mmol) in 51% yield over 2 steps by using $ZnCl_2$ (54 mg, 0.39 mmol) and $NaBH_4$ (30 mg, 0.79 mmol) in THF (0.79 mL) and CH_2Cl_2 (1.6 mL) for the first reaction, and PPTS (2.4 mg, 9.5 μmol) in dimethoxypropane (0.95 mL) and acetone (0.95 mL) for the second reaction: Colorless oil; $[\alpha]_D^{19}$ = -38.9 (*c* 1.1, MeOH); IR (film) 2989, 2952, 2934, 2862, 1466, 1378, 1255, 1190, 1123, 1065, 1005 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.11 (3H, s, TBS), 0.21 (3H, s, TBS), 0.97 (3H, s, asetonide), 1.00 (9H, s, TBS), 1.03-1.09 (6H, m, $CH(CH_3)_2$), 1.33 (3H, s, acetonide), 1.39 (3H, s, acetonide), 1.44 (3H, s, acetonide), 1.46 (3H, s, acetonide), 1.63 (3H, s, acetonide), 1.87 (1H, ddd, *J* = 13.7, 10.0, 3.6 Hz, $CH_AH_BCH(CH_3)_2$), 2.02-2.38 (6H, m, $CH_2CH(OTBS)CHCH$, $CH_ACH(CH_3)_2$), 3.42 (1H, dd, *J* = 13.7, 2.8 Hz, H5a), 3.60 (1H, s, H4), 3.79 (1H, d, *J* = 13.7 Hz, H5b), 4.00 (1H, d, *J* = 2.3 Hz, H3), 4.11 (1H, td, *J* = 10.1, 1.8 Hz, $CHOCH_2CH(CH_3)_2$), 4.28-4.40 (1H, m, $CHOCH_2CH(OTBS)$), 4.83 (1H, m, CH(OTBS)), 4.86 (1H, s, H2); ^{13}C NMR (100 MHz, C_6D_6) δ -4.1, -3.6, 18.0, 18.6, 20.3, 22.0, 24.6, 24.7, 26.1, 26.7, 27.6, 29.3, 30.6, 40.5, 44.1, 47.8, 57.8, 59.8, 71.1, 71.9, 74.2, 74.9, 75.9, 86.3, 97.3, 99.4, 110.0, 115.6; HRMS (ESI-TOF) calcd for $C_{30}H_{54}NaO_8Si$ $[M+Na]^+$ 593.3480, found 593.3468.

6-5. 参考文献

1) Durán-Galván, M.; Connell, B. T. *Tetrahedron* **2011**, *67*, 7901

2) Ikeda, K.; Achiwa, K.; Sekiya, M. *Chem. Pharm. Bull.* **1989**, *37*, 1179.

3) MacNevin, C. J.; Moore, R. L.; Liotta, D. C. *J. Org. Chem.* **2008**, *73*, 1264.

4) Donohoe, T. J.; Fishlock, L. P.; Procopiou, P. A. *Org. Lett*, **2008**, *10*, 285.

5) MacNevin, C. J.; Moore, R. L.; Liotta, D. C. *J. Org. Chem.* **2008**, *73*, 1264.

6) (a) Hampton, A.; Perini, F.; Harper, A. J. *Carbohydrate Research*, **1974**, *37*, 359-367. (b) Huang, L.; Teumelsan, N.; Huang, X. *Chem. Eur. J*. **2006**, *12*, 5246-5252.

7) (a) Timoshchuk, V. A. *Zhurnal Organicheskoi Khimii*, **1992**, 28, 1512-1519. (b) Cai, L.; Li, Q.; Ren, B.; Yang, Z.-J.; Zhang, L.-R.; Zhang, L.-H.Tetrahedron, **2007**, *63*, 8135-8144.

8) Polakova, M.; Pitt, N.; Tosin, M.; Murphy, P. V. *Angew. Chem. Int. Ed*. **2004**, *43*, 2518-2521.

9) Miyabe, H.; Shibata, R.; Sangawa, M.; Ushiro, C.; Naito, T. *Tetrahedron*, **1998,** *54*, 11431-11444.

10) Barton, D. H. R.; Garcia, B.; Togo, H.; Zard, S. *Tetrahedron Lett*. **1986**, *27*, 1327-1330.

11) Nemoto, H.; Shiraki, M.; Fukumoto, K. *Tetrahedron Lett*., **1995**, *36*, 8799.

12) Pandey, G.; Balakrishnan, M. *J. Org. Chem.* **2008**, *73*, 8128.

13) Dakin, L. A.; Langille, N. F.; Panek, J. S. M. A. *J. Org. Chem.* **2002**, *67*, 6812.

14) (a) Nakata, T.; Oishi, T. *Tetrahedron Lett.* **1980**, *21*, 1641. (b) Kathawala, F. G.; Prager, B.; Prasad, K.; Repic, O.; Shapiro, M. J.; Stabler, R. S.; Widler, L. *Helv. Chim. Acta*, **1986**, *69*, 803.

15) The crystal of **4-17** was obtained as an enantiopure form. The drawn structure of **4-17** was antipodal to the obtained X-ray structure.

スペクトルデータ

スペクトルデータ

single_pulse

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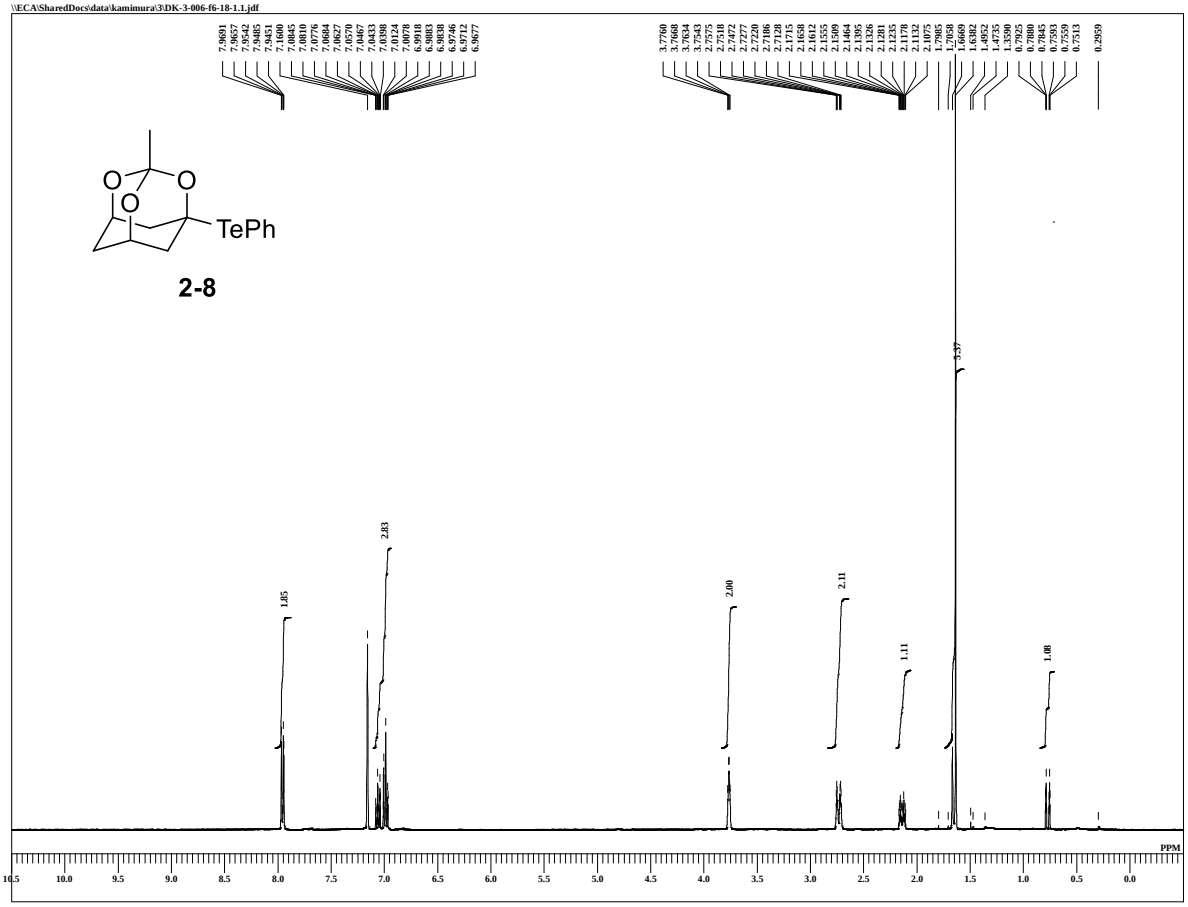


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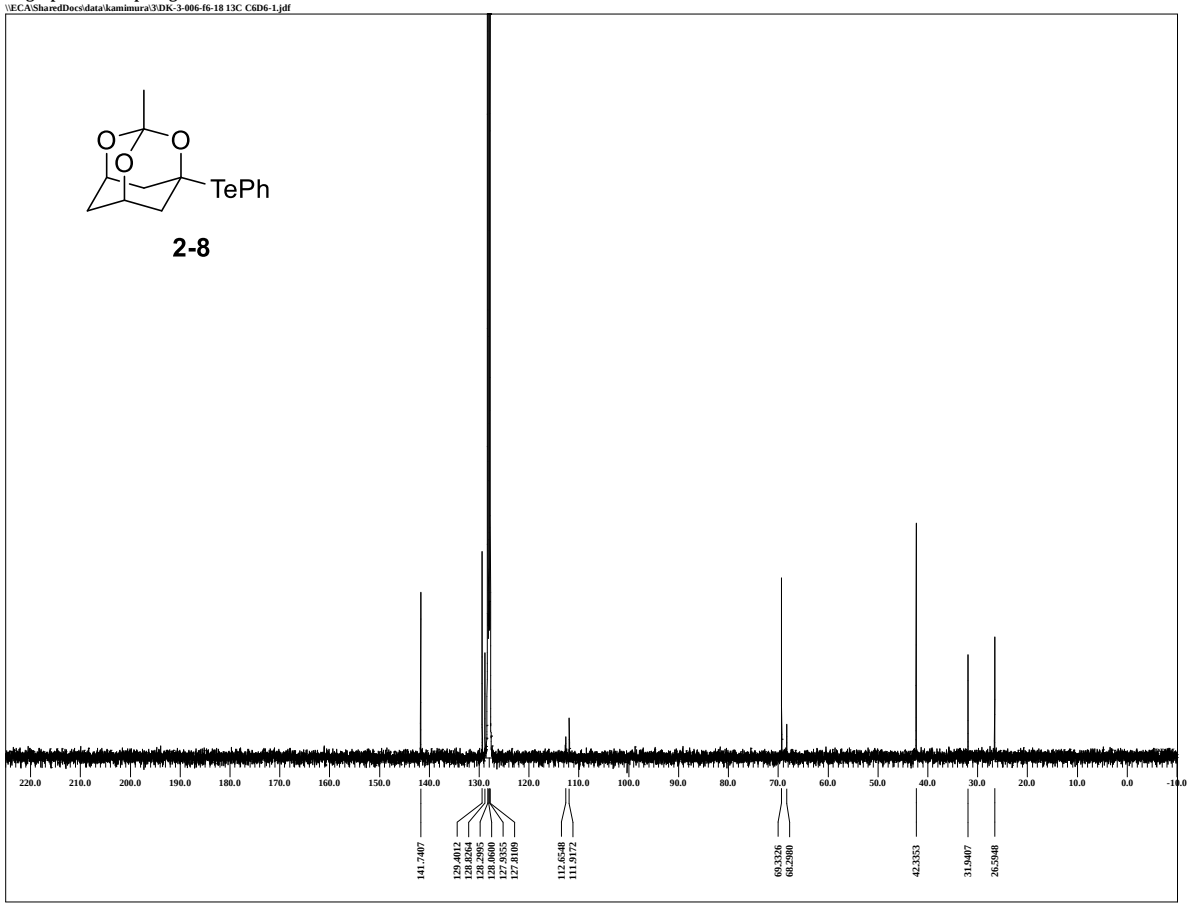


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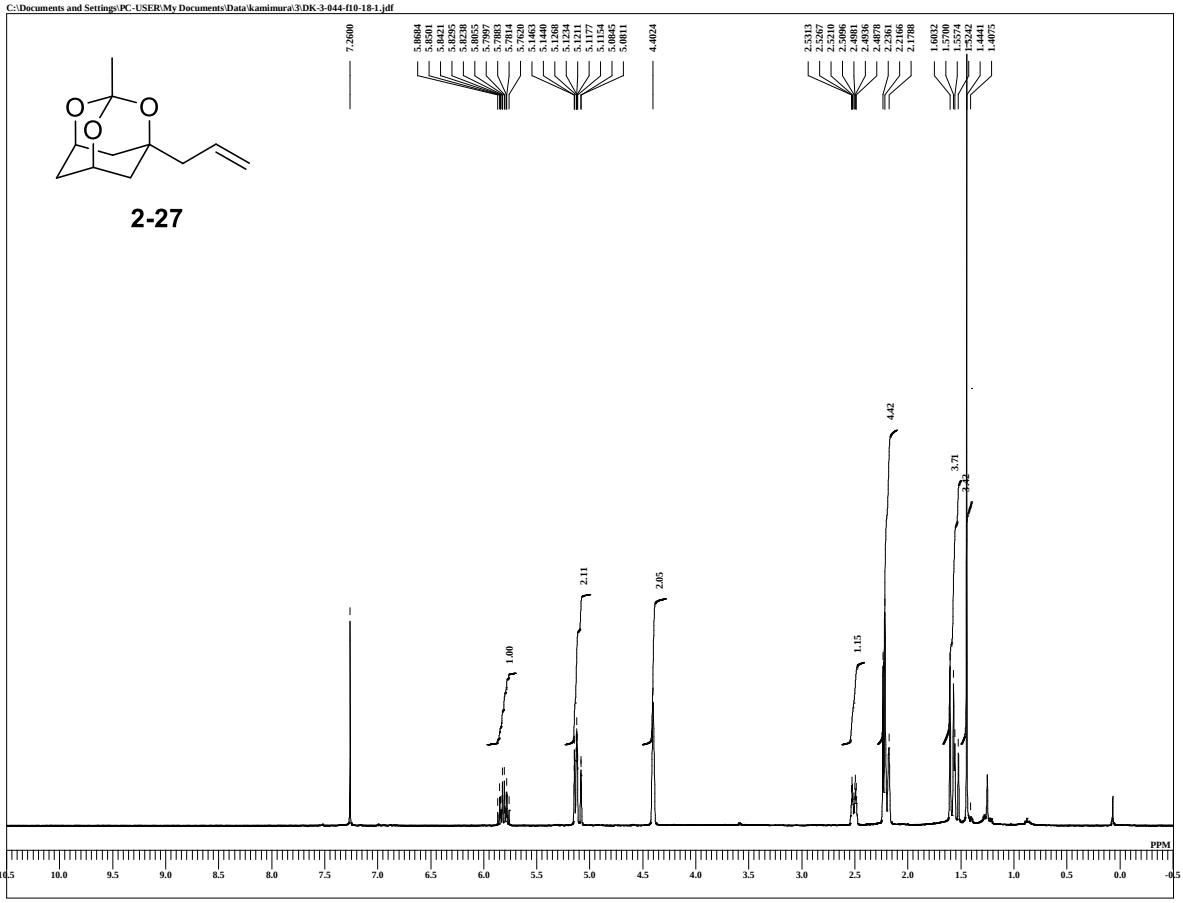
PW1          3.25 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        32768
SPO          32768
TIMES            327
DUMMY            4
FREQU        31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM        1.0486 sec
PD           2.0000 sec
SCANS            327
ADBIT            16
RGAIN            58
BF           1.00 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-3-006-f6-18 13C C6D6-1.jdf
SF
LKSET        13.20 KHz
LKFIN        69.6 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED        0 Hz
FILDC
FILDF
CTEMP        20.6 c
SLVNT    C6D6
EXREF        128.06 ppm
```

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-044-f10-18-1.jdf

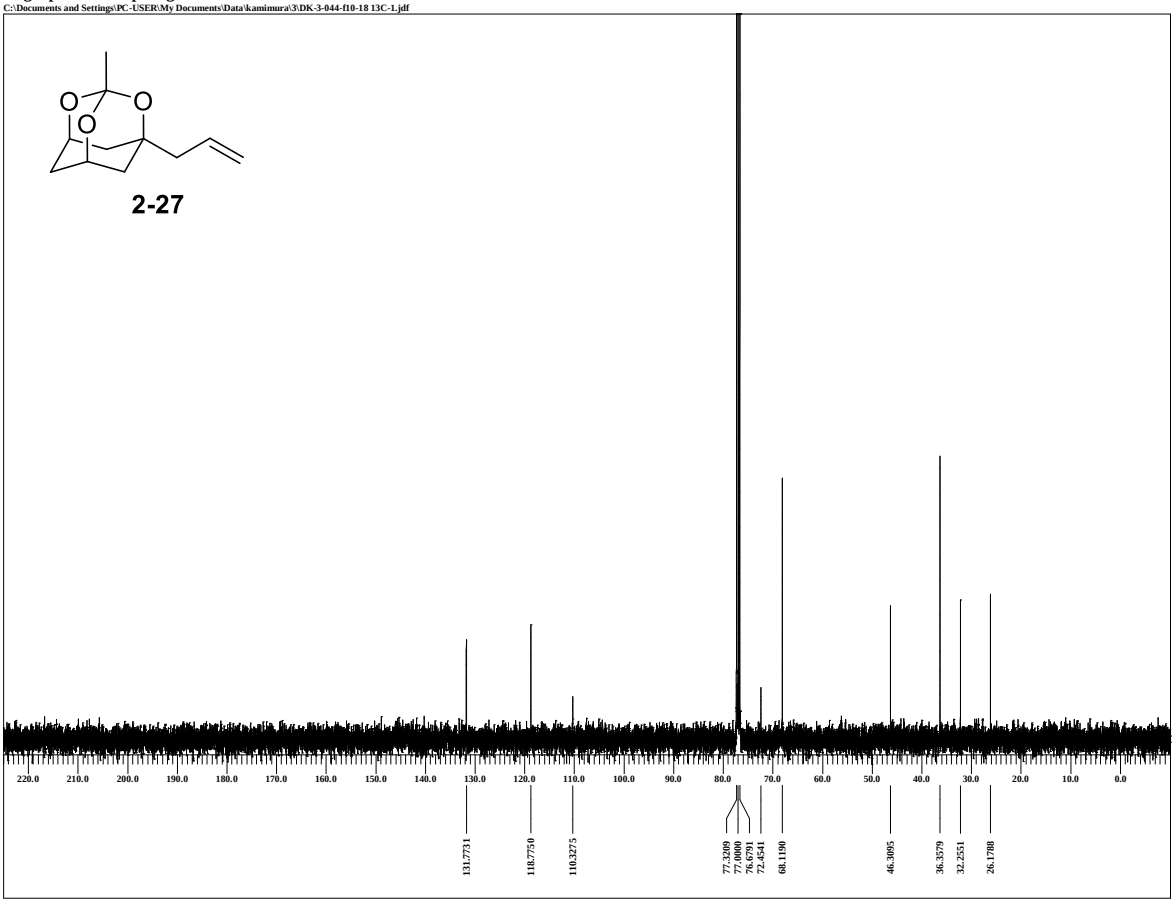


2-27

DFILE	DK-3-044-f10-18-1.jdf
COMNT	single_pulse
DATIM	25-12-2012 12:36:40
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	44
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-044-f10-18-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-044-f10-18 13C-1.jdf



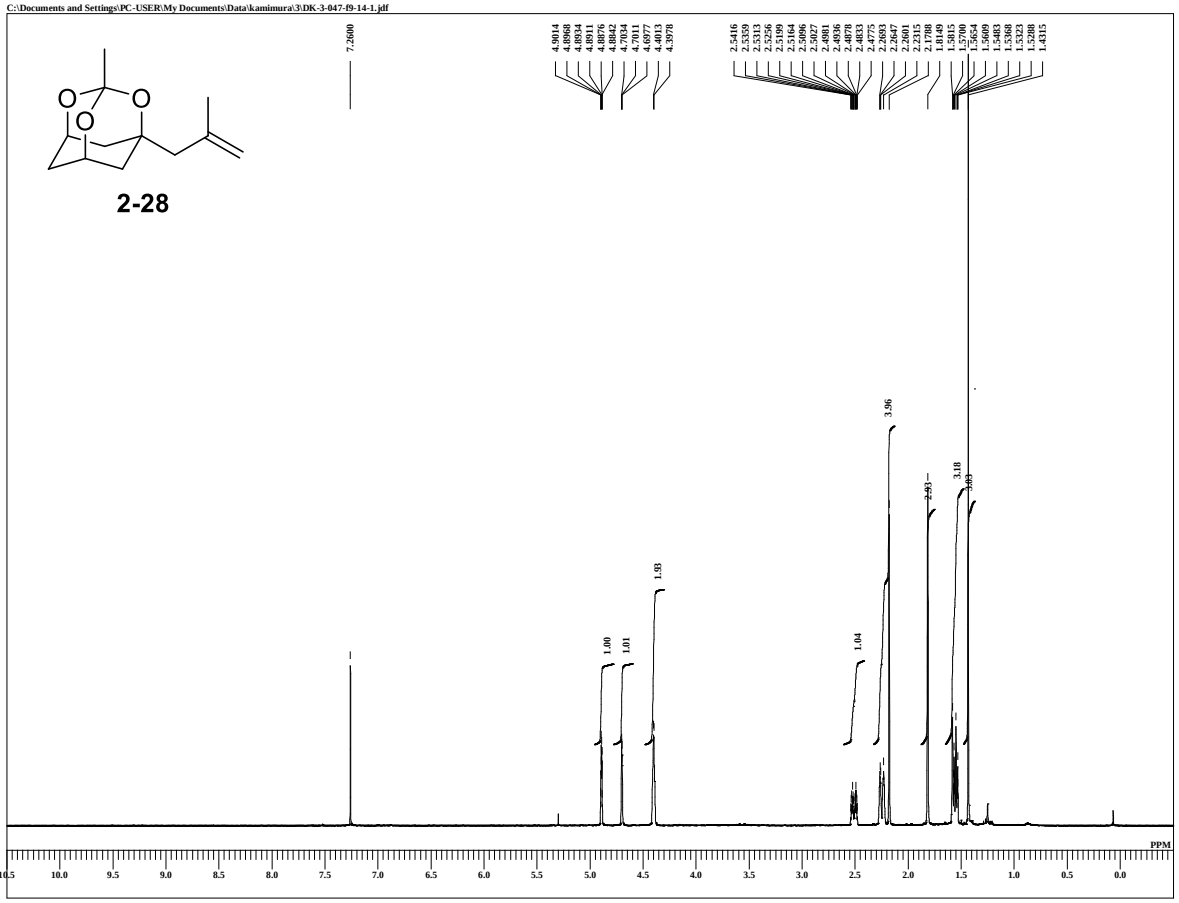
2-27

DFILE	DK-3-044-f10-18 13C-1.jc
COMNT	single pulse decoupled gat
DATIM	25-12-2012 12:46:57
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	131072
SPO	131072
TIMES	183
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	183
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-044-f10-18 13C-1.jc
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

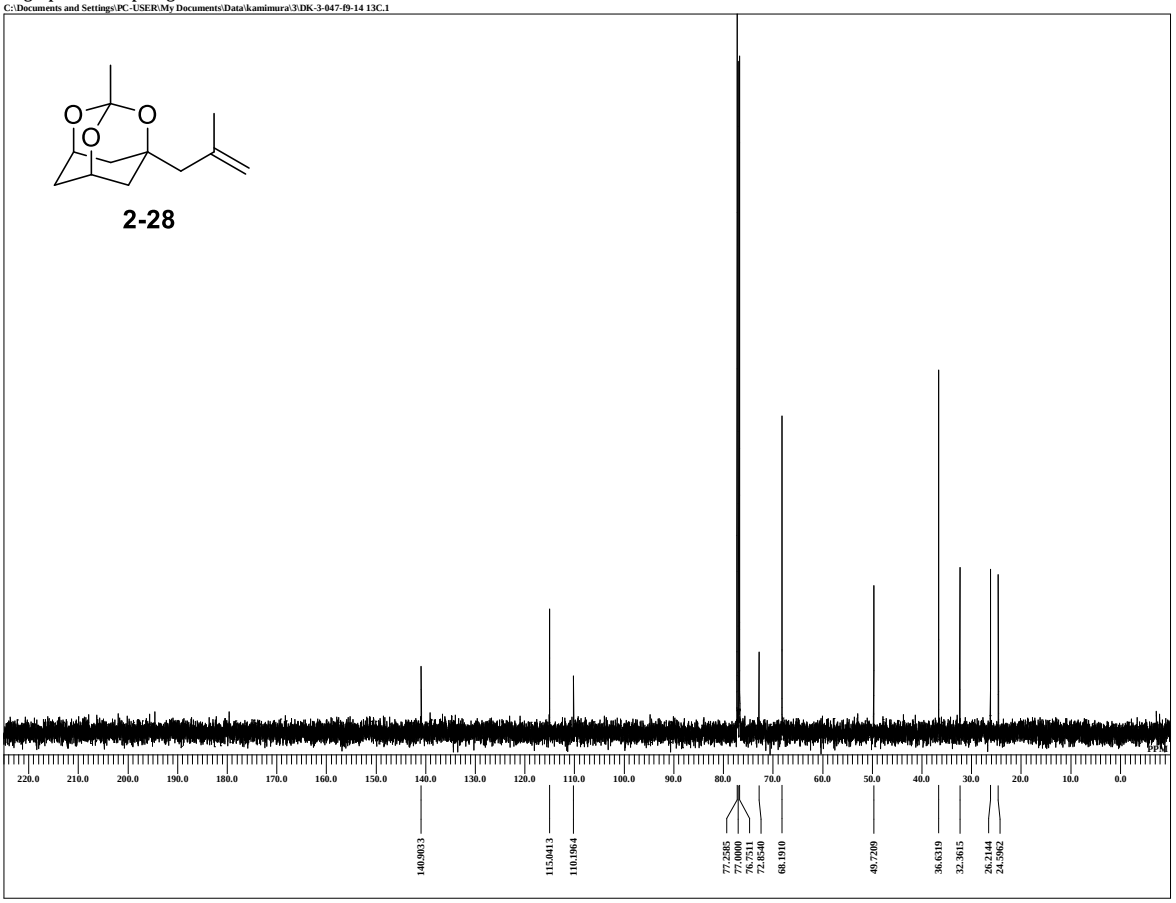
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-047-f9-14-1.jdf



DFILE DK-3-047-f9-14-1.jdf
COMNT single_pulse
DATIM 04-01-2013 12:28:60
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.38 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 44
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-047-f9-14-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.3 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-047-f9-14 13C.1

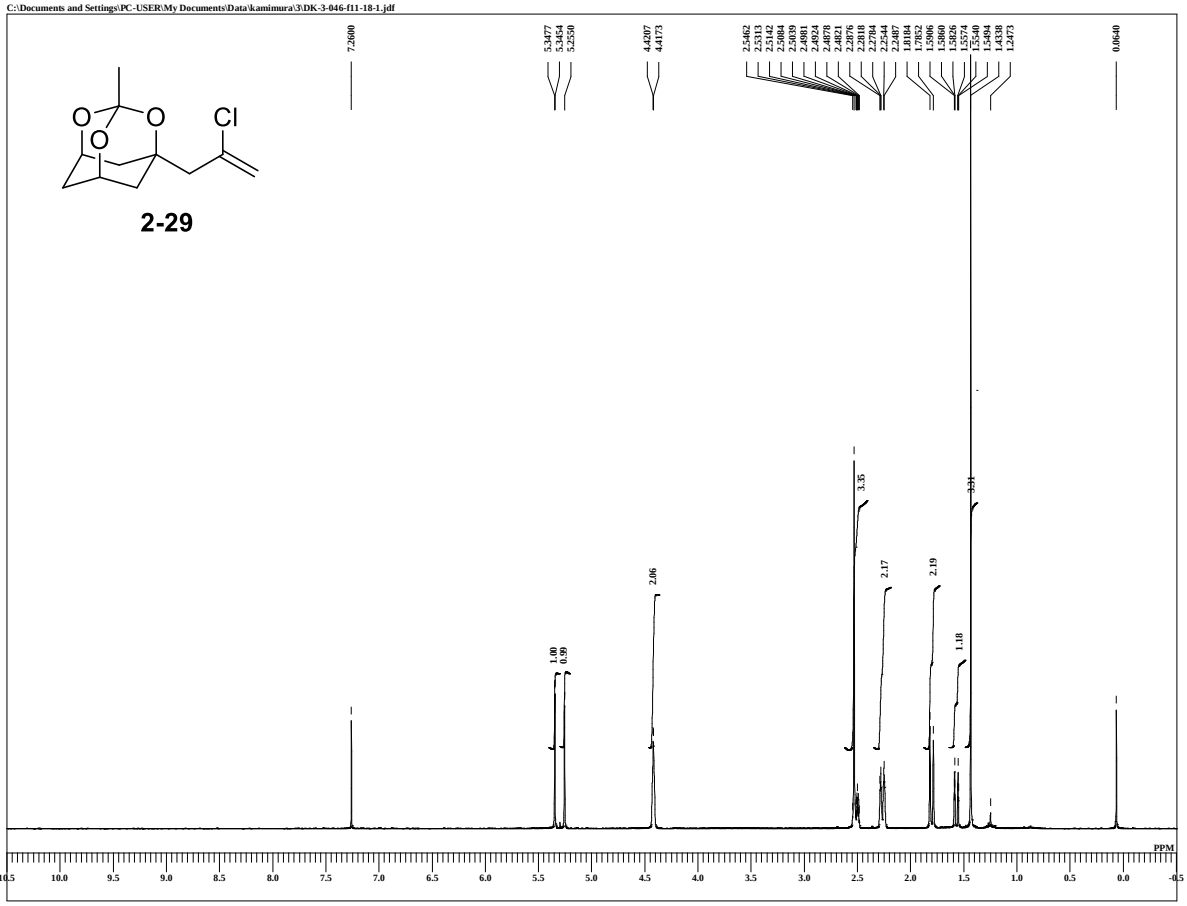


DFILE DK-3-047-f9-14 13C.1
COMNT single pulse decoupled gat
DATIM 27-03-2013 18:26:02
MENUF
OBNUC 13C
OFR 124.51 MHz
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
PW1 3.70 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 94
DUMMY 4
FREQU 39062.50 Hz
FLT 157000 Hz
DELAY 20.80 usec
ACQTM 0.8389 sec
PD 2.0000 sec
SCANS 94
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE DK-3-047-f9-14 13C.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-046-f11-18-1.jdf

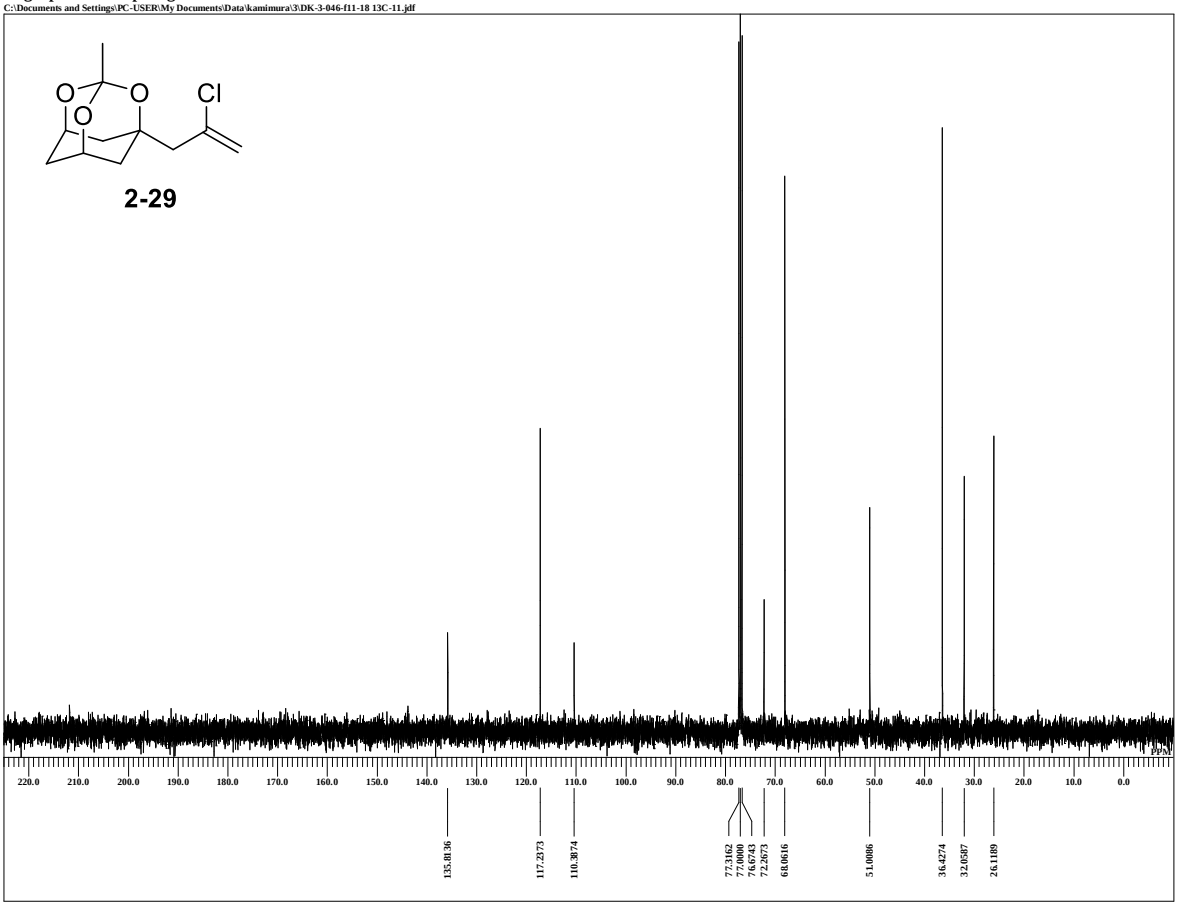


2-29

DFILE DK-3-046-f11-18-1.jdf
COMNT single_pulse
DATIM 04-01-2013 11:58:22
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.38 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 44
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-046-f11-18-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.0 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-046-f11-18 13C-11.jdf



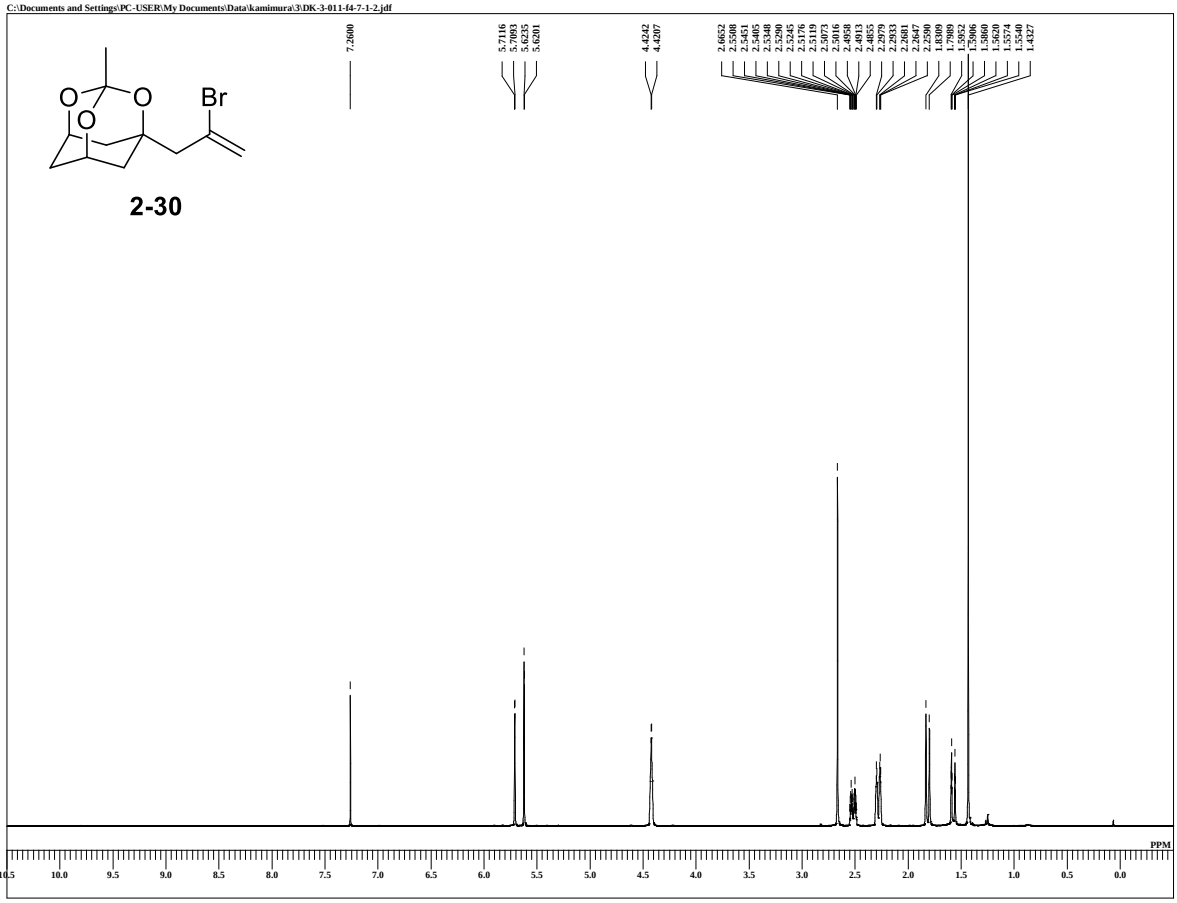
2-29

DFILE DK-3-046-f11-18 13C-11.j
COMNT single pulse decoupled gat
DATIM 27-03-2013 17:10:50
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.25 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 52
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 52
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-046-f11-18 13C-11.j
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-011-f4-7-1-2.jdf

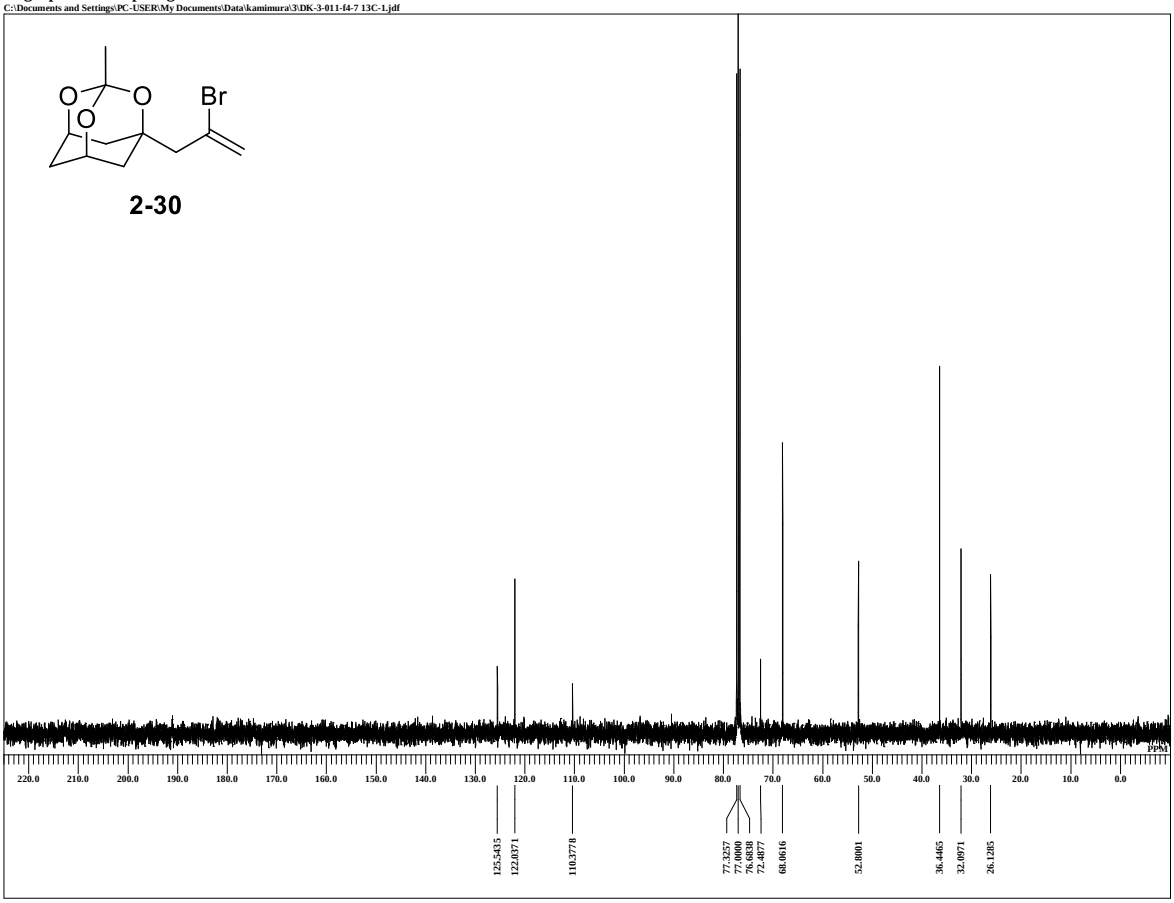


2-30

DFILE	DK-3-011-f4-7-1-2.jdf
COMNT	single_pulse
DATIM	23-01-2013 16:39:49
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	42
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-011-f4-7-1-2.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-011-f4-7 13C-1.jdf



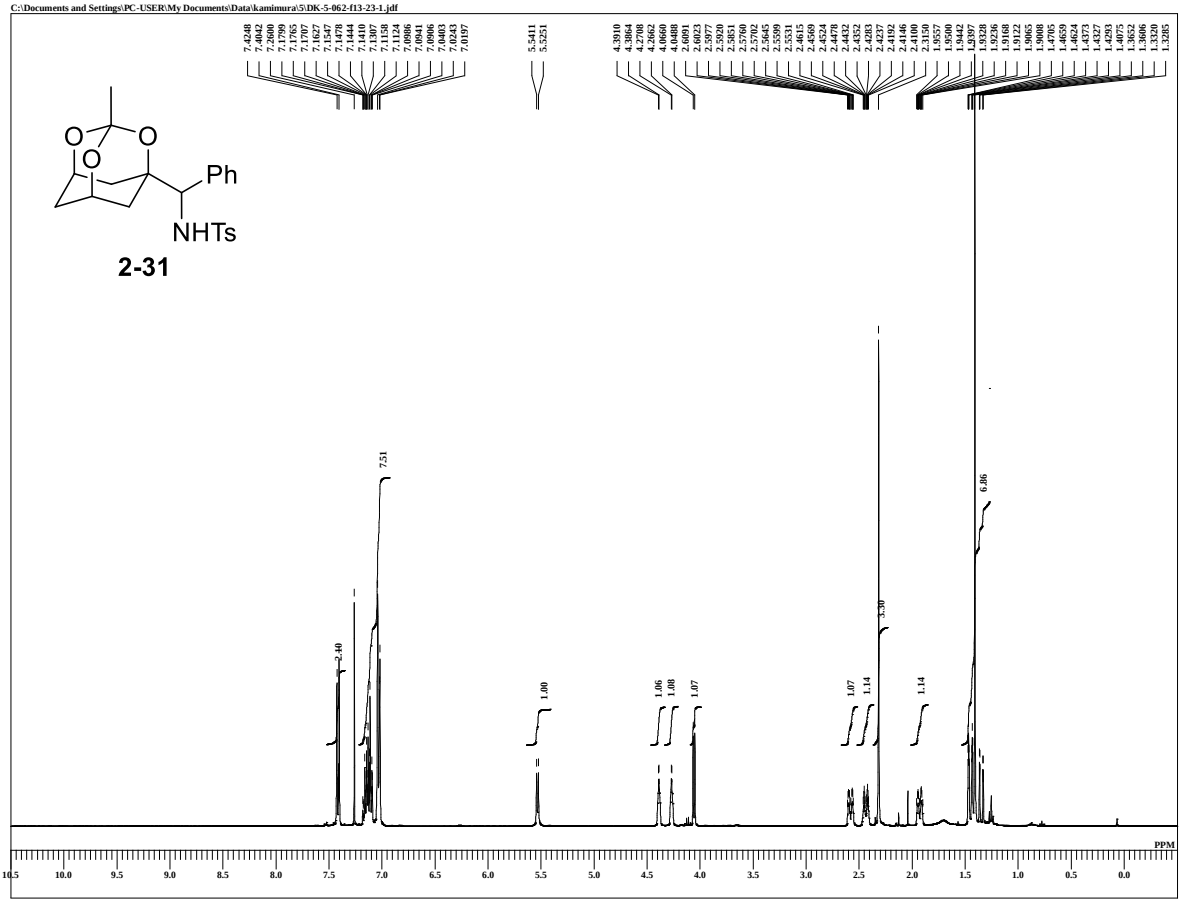
2-30

DFILE	DK-3-011-f4-7 13C-1.jdf
COMNT	single pulse decoupled gat
DATIM	04-12-2012 17:23:13
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	126
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	126
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-011-f4-7 13C-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

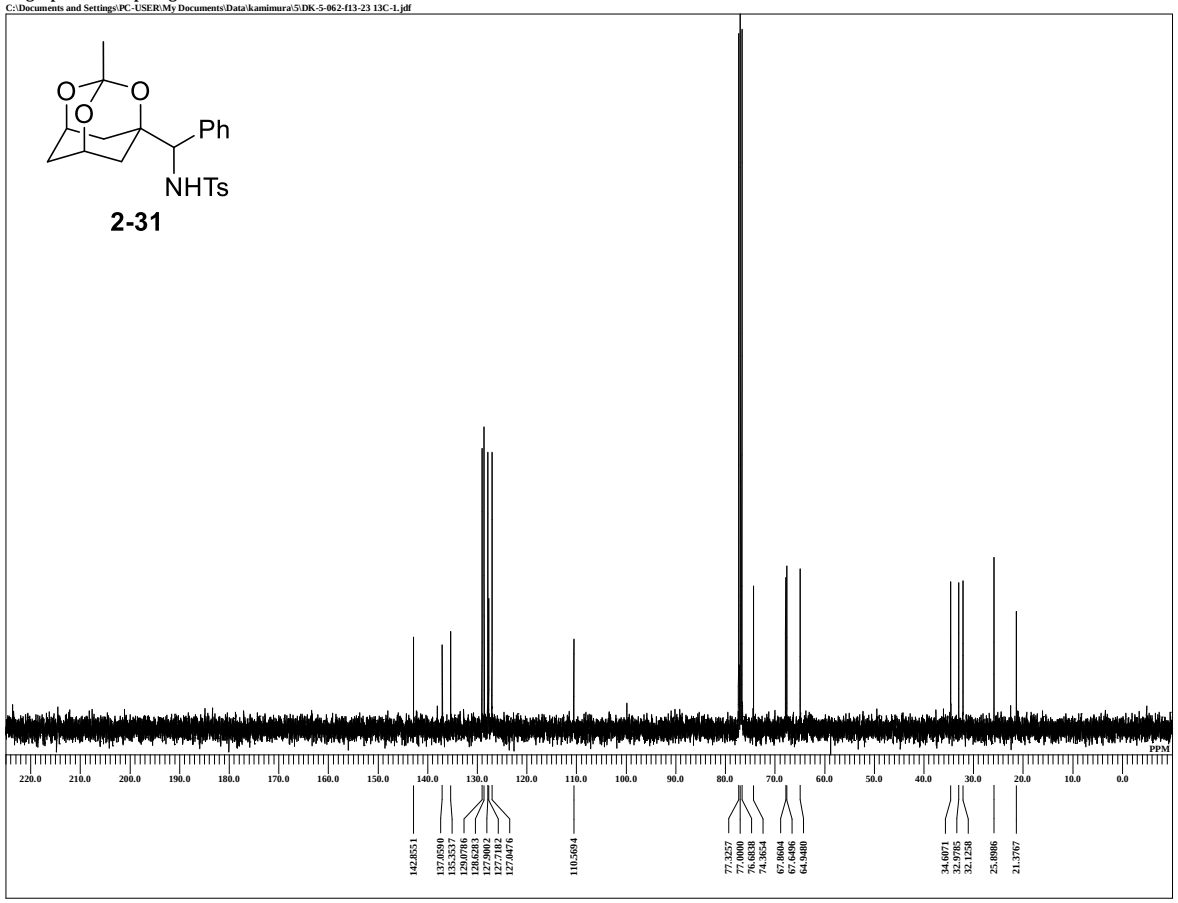
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-062-f13-23-1.jdf



DFILE	DK-5-062-f13-23-1.jdf
COMNT	single_pulse
DATIM	12-09-2013 18:14:26
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-062-f13-23-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-062-f13-23 13C-1.jdf

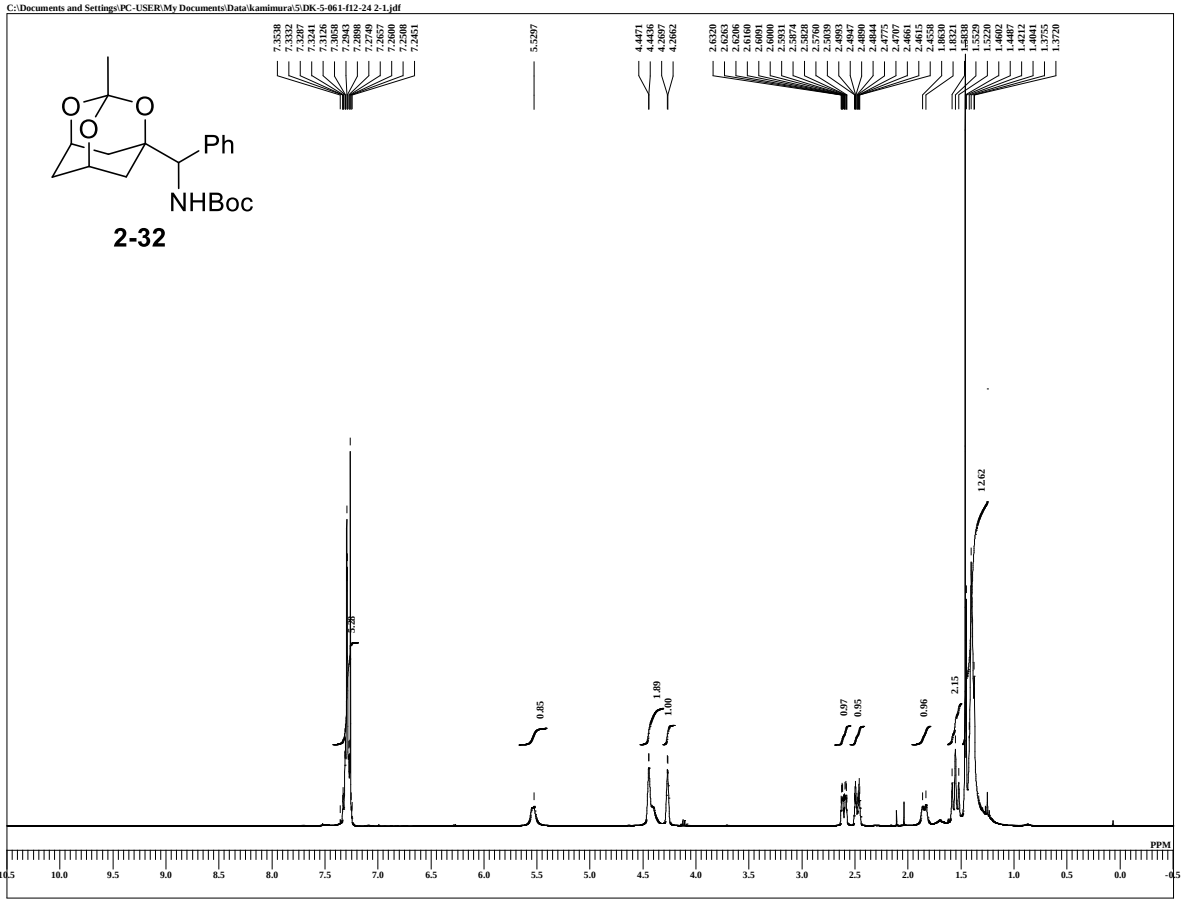


DFILE	DK-5-062-f13-23 13C-1.jc
COMNT	single pulse decoupled gat
DATIM	12-09-2013 18:18:11
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	62
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	62
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-062-f13-23 13C-1.jc
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

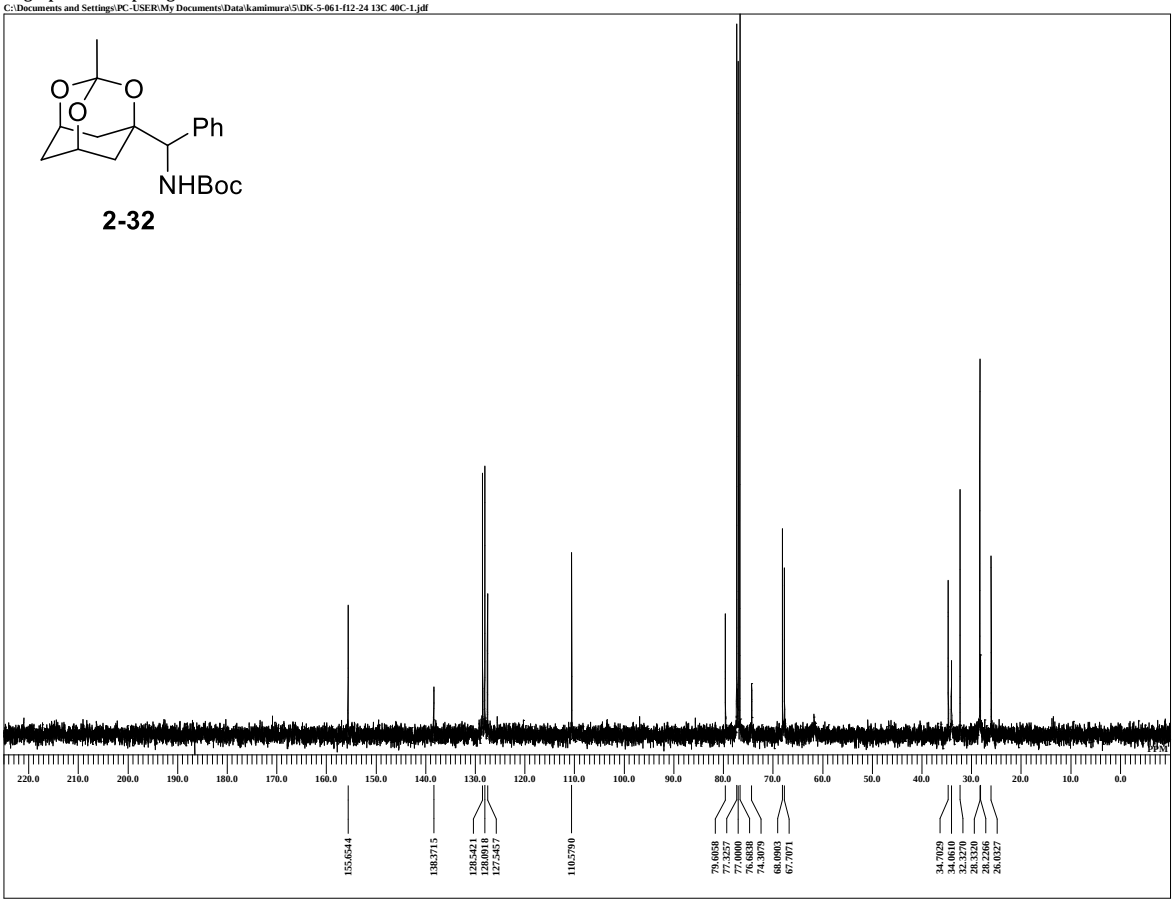
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-061-f12-24 2-1.jdf



DFILE	DK-5-061-f12-24 2-1.jdf
COMNT	single_pulse
DATIM	04-10-2013 08:47:22
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-061-f12-24 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-061-f12-24 13C 40C-1.jdf

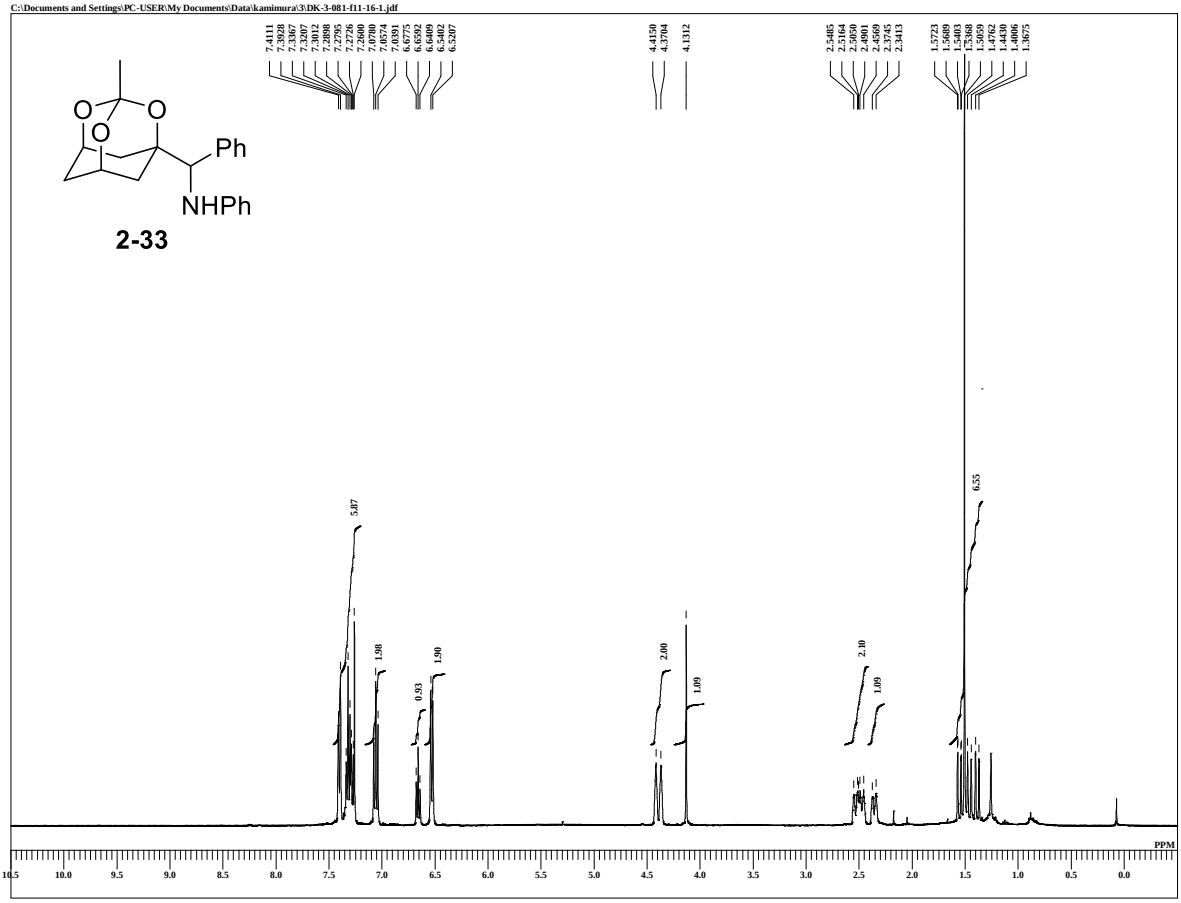


DFILE	DK-5-061-f12-24 13C 40C
COMNT	single pulse decoupled gat
DATIM	13-09-2013 18:37:53
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	128
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	128
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-061-f12-24 13C 40C
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	40.0 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-081-f11-16-1.jdf

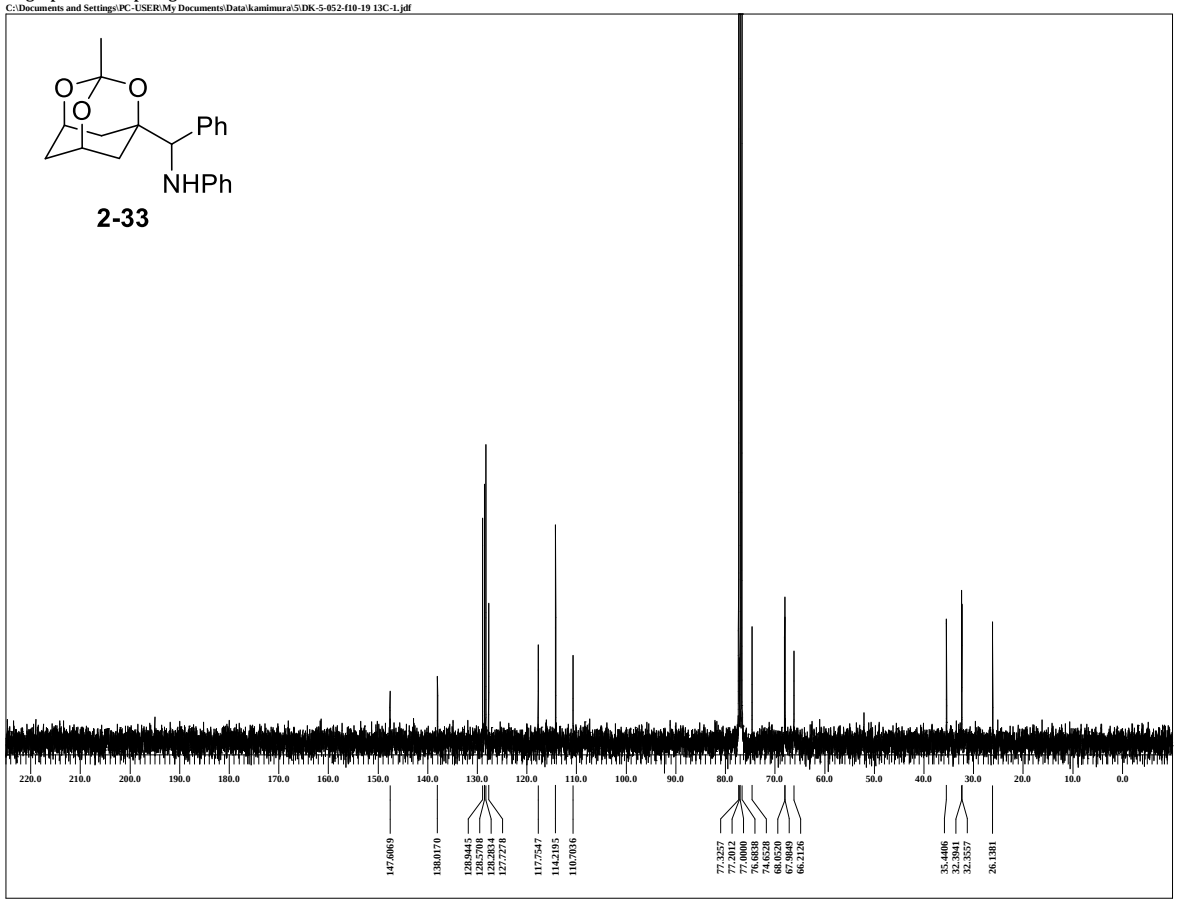


2-33

```
DFILE   DK-3-081-f11-16-1.jdf
COMNT   single_pulse
DATIM   24-01-2013 16:26:39
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.38 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       16384
SPO         16384
TIMES           8
DUMMY           1
FREQU    7422.80 Hz
FLT        30000 Hz
DELAY      16.68 usec
ACQTM      2.2073 sec
PD         2.0000 sec
SCANS           8
ADBIT          16
RGAIN          42
BF          0.10 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW        115 usec
IRATN           79
DFILE   DK-3-081-f11-16-1.jdf
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED       0 Hz
FILDC
FILDF
CTEMP       20.6 c
SLVNT   CDCL3
EXREF       7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-052-f10-19 13C-1.jdf



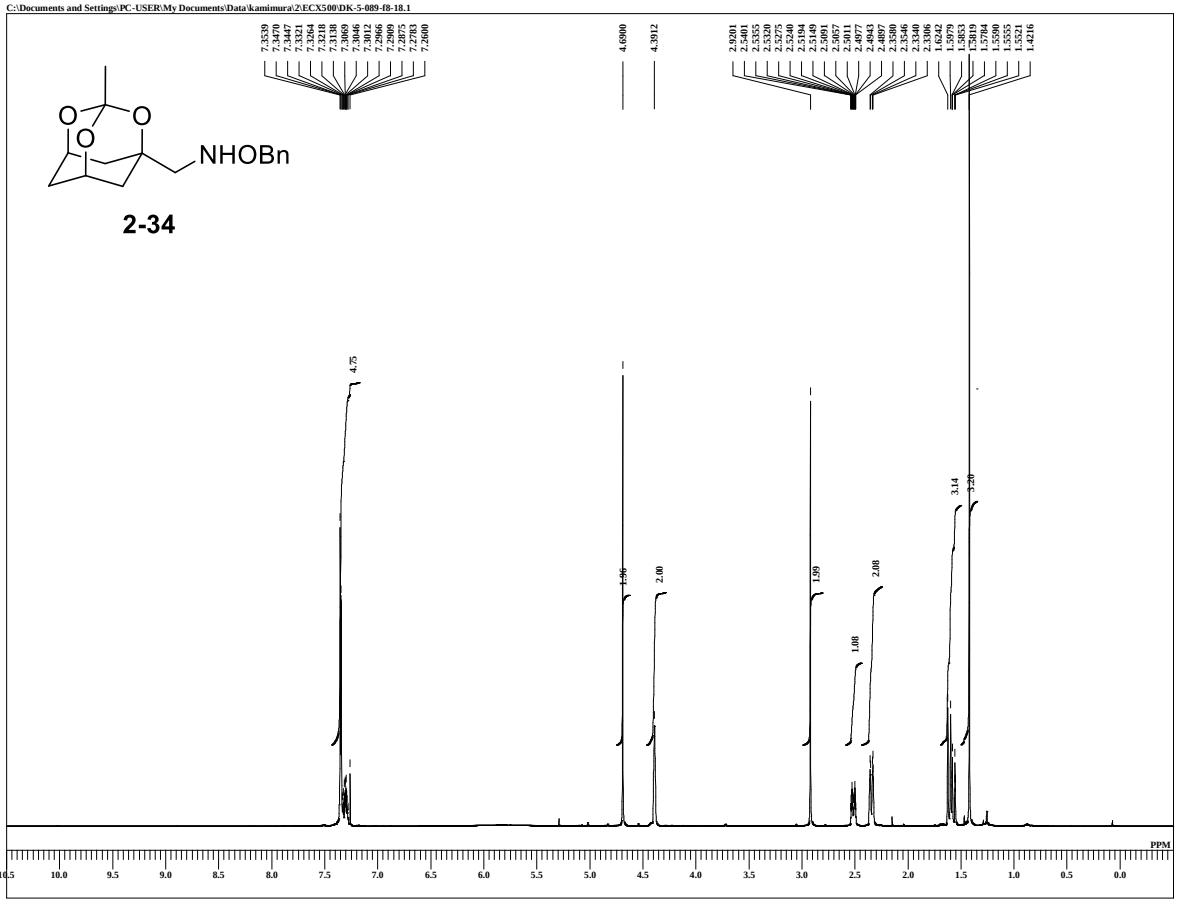
2-33

```
DFILE   DK-5-052-f10-19 13C-1.jd
COMNT   single pulse decoupled gat
DATIM   13-09-2013 19:43:04
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET       5.13 KHz
OBFIN       0.98 Hz
PW1         3.03 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       32768
SPO         32768
TIMES         291
DUMMY           4
FREQU   31250.00 Hz
FLT       125000 Hz
DELAY      20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS         291
ADBIT          16
RGAIN          60
BF          1.00 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW        115 usec
IRATN           79
DFILE   DK-5-052-f10-19 13C-1.jd
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED       0 Hz
FILDC
FILDF
CTEMP       23.9 c
SLVNT   CDCL3
EXREF       77.00 ppm
```

スペクトルデータ

single_pulse

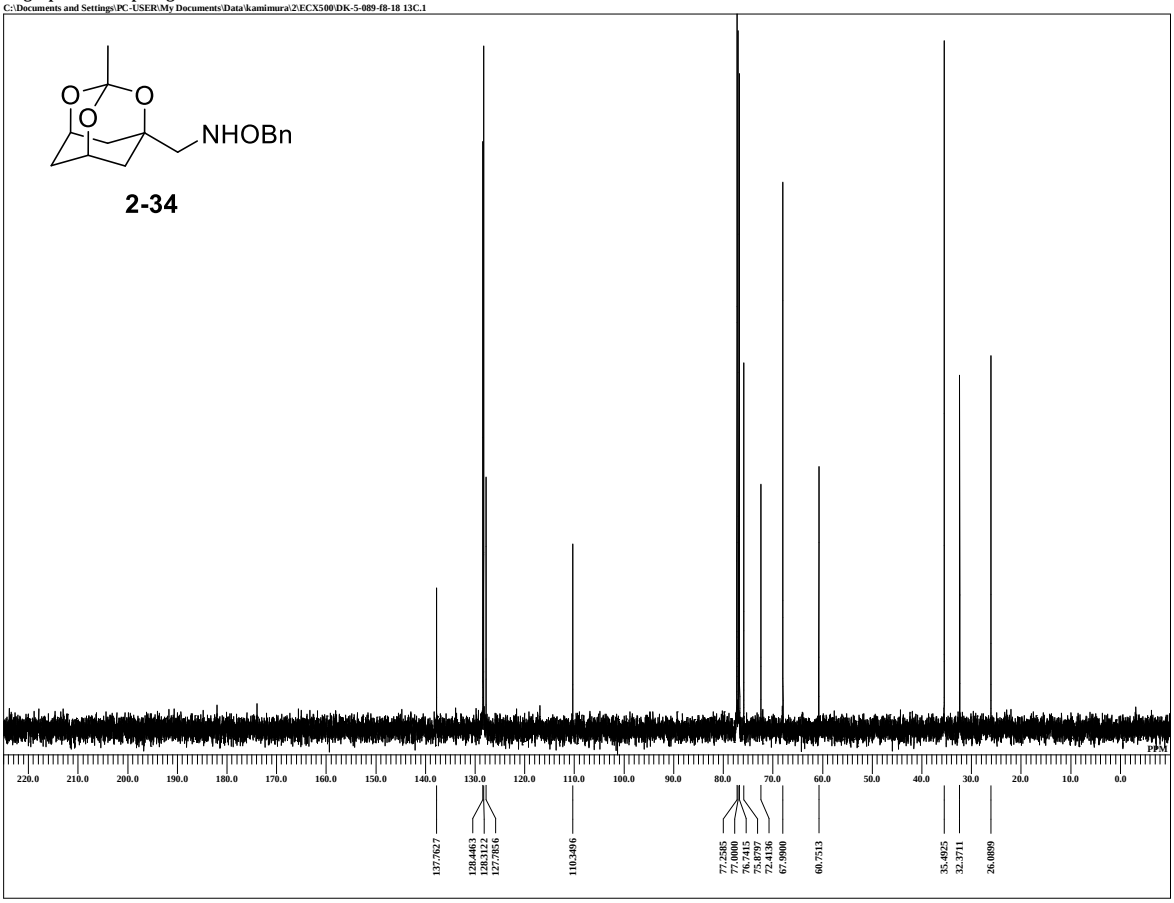
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-089-f8-18.1



DFILE DK-5-089-f8-18.1
COMNT single_pulse
DATIM 26-09-2013 19:43:24
MENUF
OBNUC 1H
OFR 495.13 MHz
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
PW1 6.00 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 9286.78 Hz
FLT 38000 Hz
DELAY 13.16 usec
ACQTM 1.7642 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 118 usec
IRATN 79
DFILE DK-5-089-f8-18.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.2 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-089-f8-18 13C.1

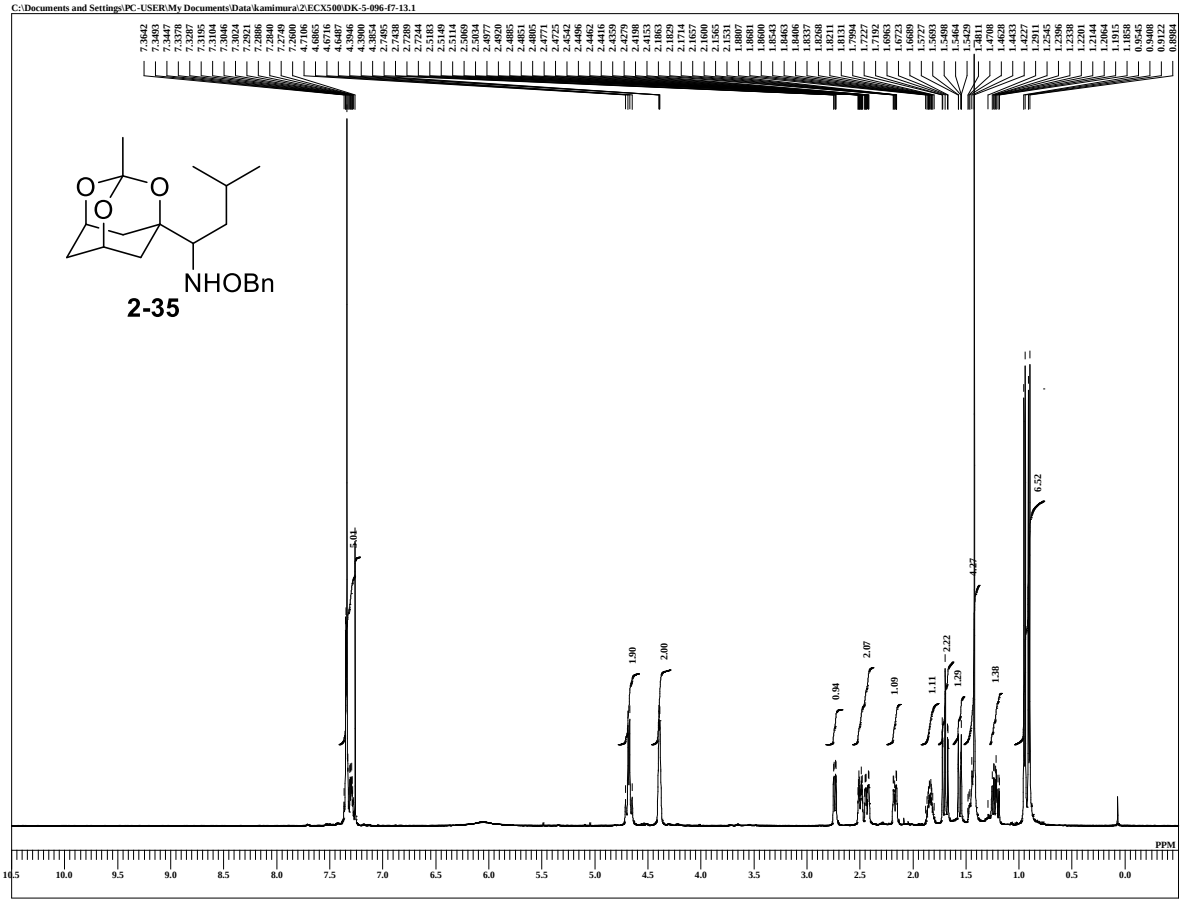


DFILE DK-5-089-f8-18 13C.1
COMNT single pulse decoupled gat
DATIM 26-09-2013 19:48:26
MENUF
OBNUC 13C
OFR 124.51 MHz
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
PW1 3.70 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 83
DUMMY 4
FREQU 39062.50 Hz
FLT 157000 Hz
DELAY 20.80 usec
ACQTM 0.8389 sec
PD 2.0000 sec
SCANS 83
ADBIT 16
RGAIN 46
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE DK-5-089-f8-18 13C.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.8 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

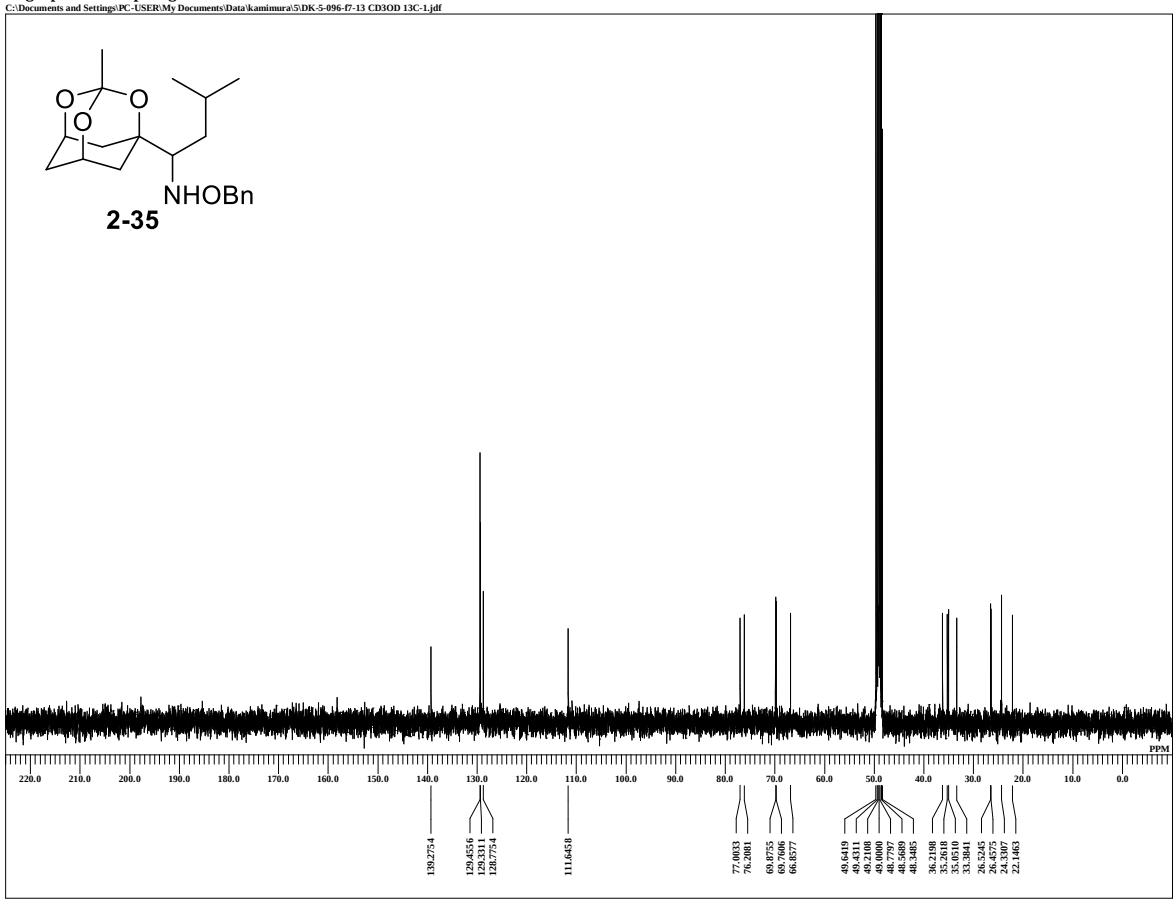
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-096-f7-13.1



DFILE	DK-5-096-f7-13.1
COMNT	single_pulse
DATIM	28-09-2013 17:25:30
MENUF	
OBNUC	1H
OFR	495.13 MHz
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
PW1	6.00 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	9286.78 Hz
FLT	38000 Hz
DELAY	13.16 usec
ACQTM	1.7642 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	42
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-096-f7-13.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-096-f7-13 CD3OD 13C-1.jdf

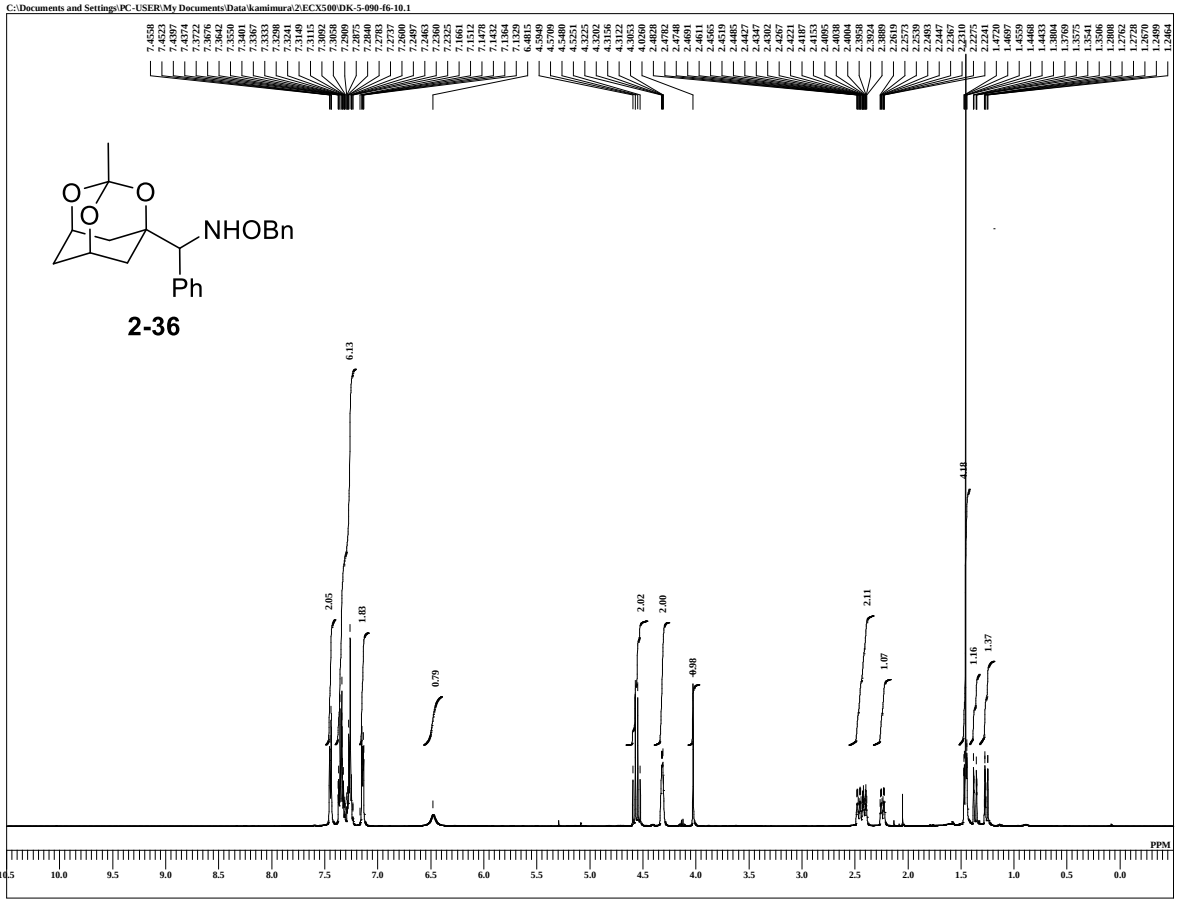


DFILE	DK-5-096-f7-13 CD3OD 1
COMNT	single pulse decoupled gat
DATIM	08-10-2013 16:34:18
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	225
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	225
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-096-f7-13 CD3OD 1
SF	
LKSET	13.00 KHz
LKFIN	35.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.7 c
SLVNT	CD3OD
EXREF	49.00 ppm

スペクトルデータ

single_pulse

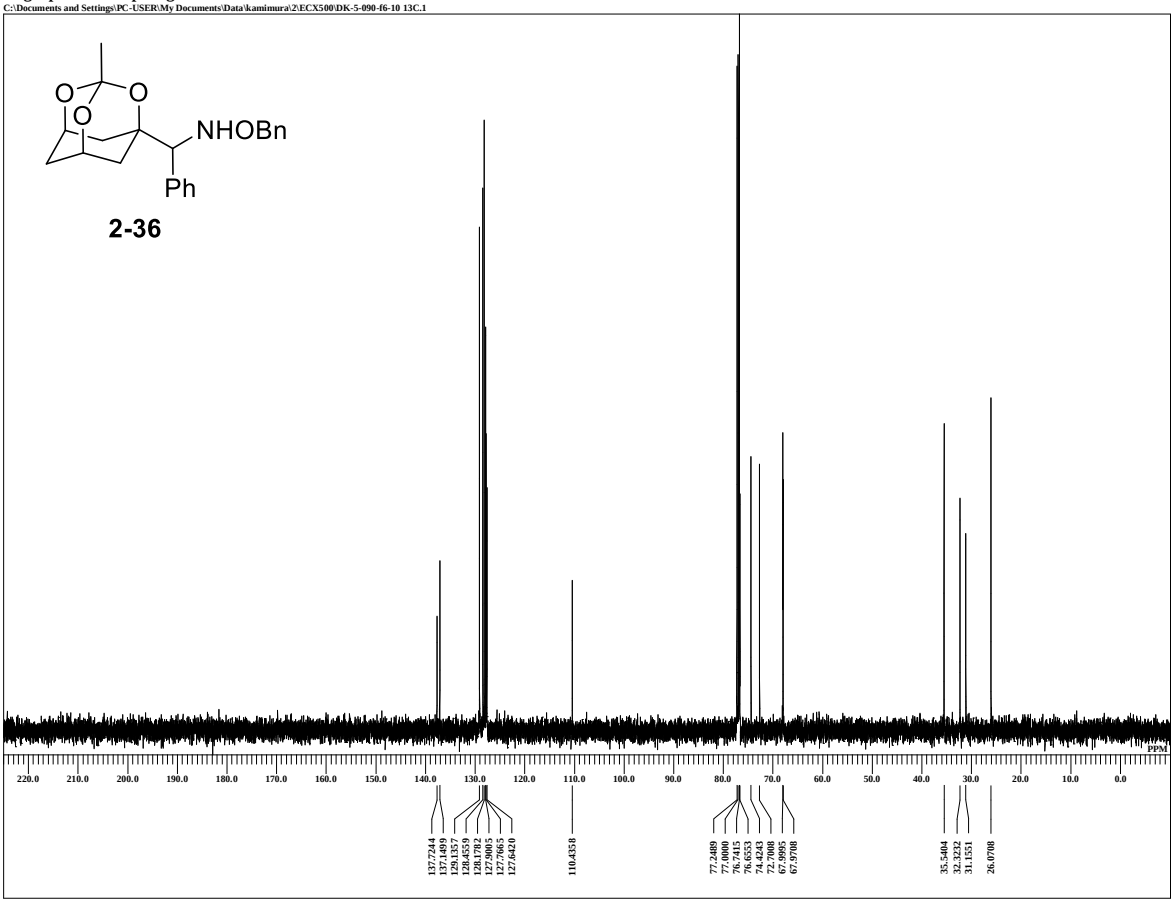
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-090-f6-10.1



DFILE	DK-5-090-f6-10.1
COMNT	single_pulse
DATIM	26-09-2013 20:04:55
MENUF	
OBNUC	1H
OFR	495.13 MHz
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
PW1	6.00 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	9286.78 Hz
FLT	38000 Hz
DELAY	13.16 usec
ACQTM	1.7642 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	36
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-090-f6-10.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-090-f6-10 13C.1

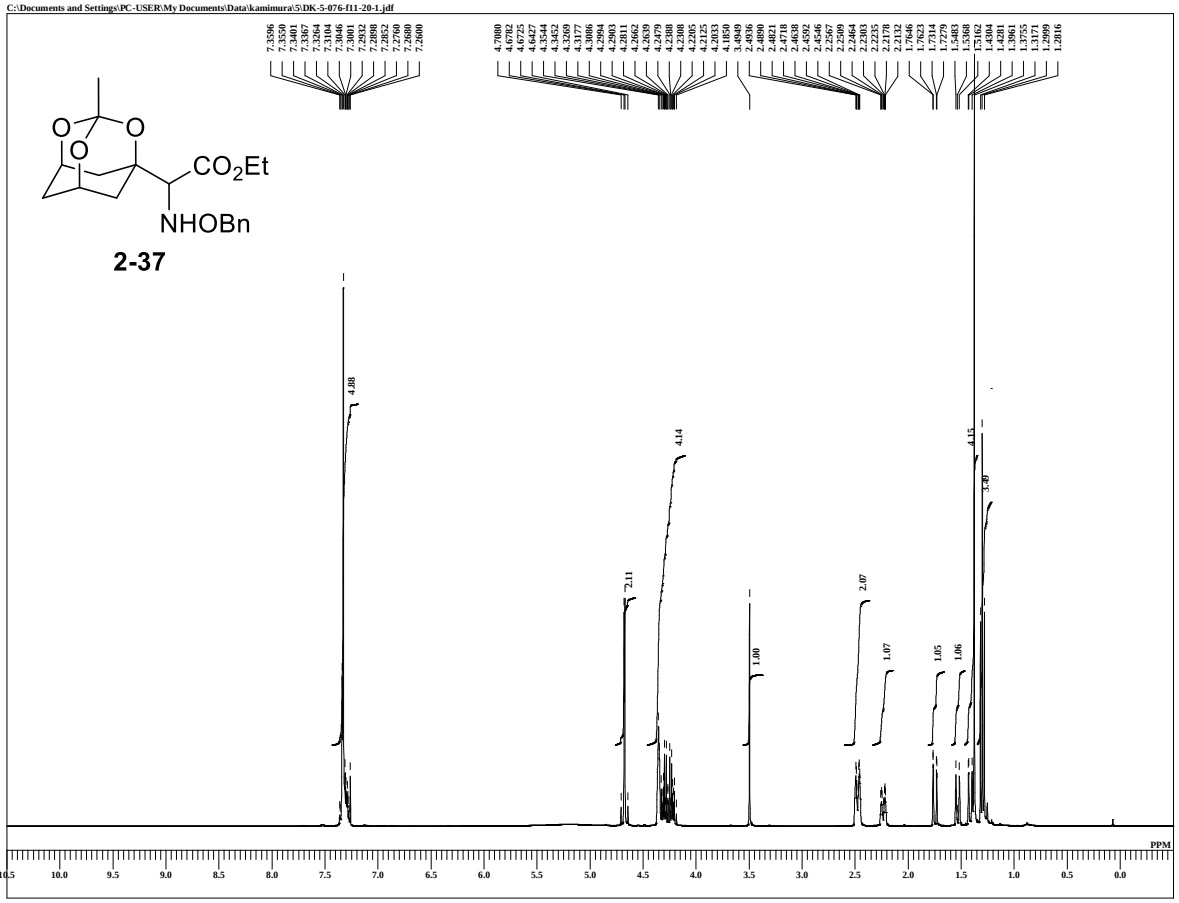


DFILE	DK-5-090-f6-10 13C.1
COMNT	single pulse decoupled gat
DATIM	26-09-2013 20:11:16
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	111
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	111
ADBIT	16
RGAIN	46
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-090-f6-10 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.8 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

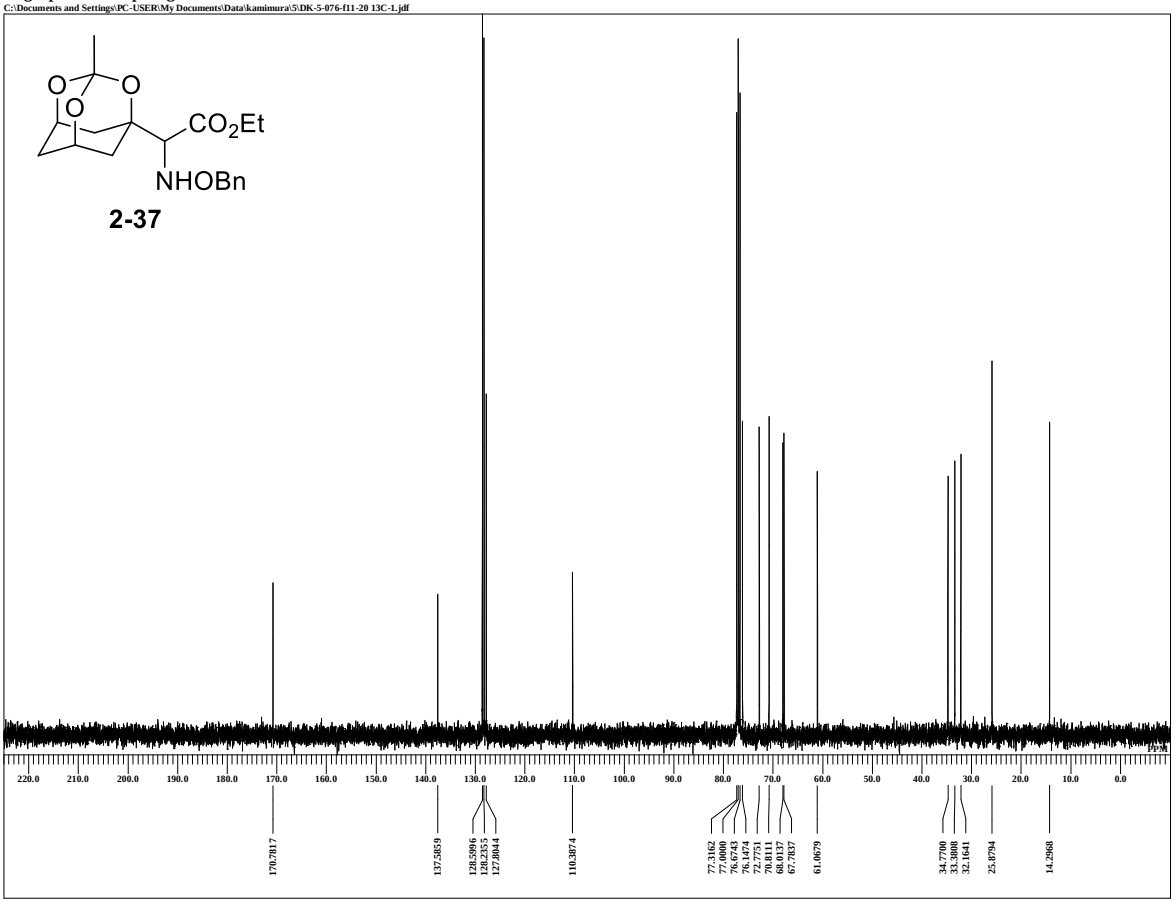
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-076-f11-20-1.jdf



DFILE DK-5-076-f11-20-1.jdf
COMNT single_pulse
DATIM 20-09-2013 15:30:39
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-5-076-f11-20-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.7 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-076-f11-20 13C-1.jdf

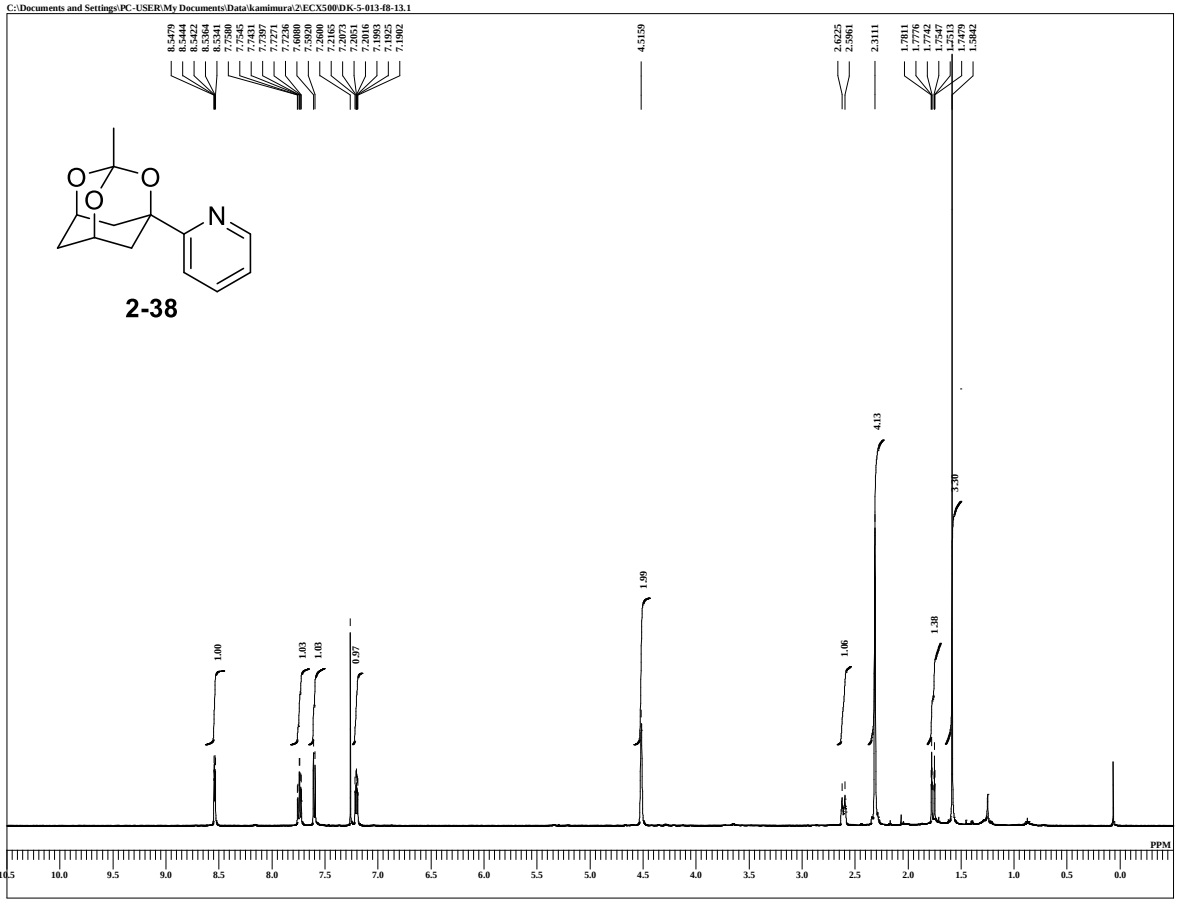


DFILE DK-5-076-f11-20 13C-1.jd
COMNT single pulse decoupled gat
DATIM 20-09-2013 15:35:21
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 81
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 81
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-076-f11-20 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.8 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

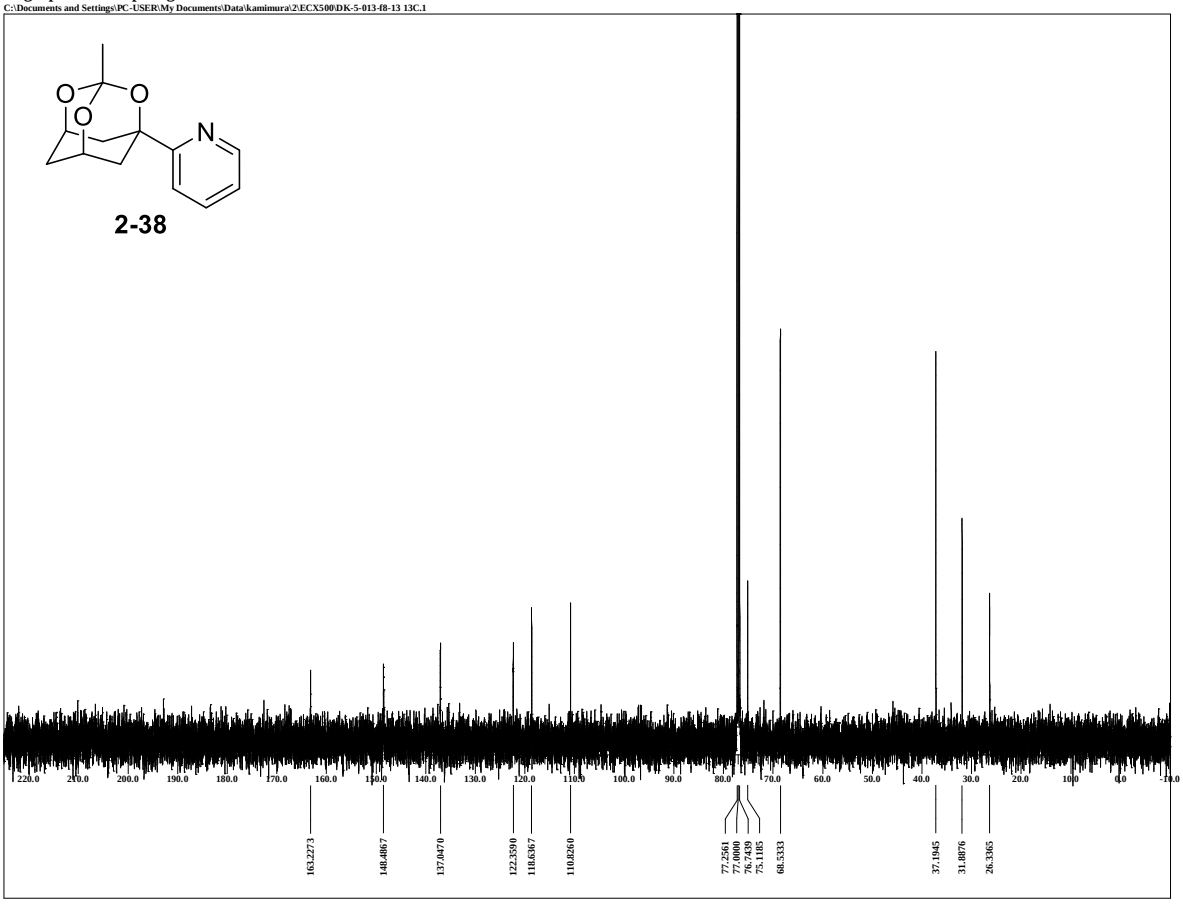
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-013-f8-13.1



DFILE	DK-5-013-f8-13.1
COMNT	single_pulse
DATIM	09-09-2013 14:21:59
MENUF	
OBNUC	1H
OFR	495.13 MHz
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
PW1	6.00 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	9286.78 Hz
FLT	38000 Hz
DELAY	13.16 usec
ACQTM	1.7642 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	54
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-013-f8-13.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-013-f8-13 13C.1

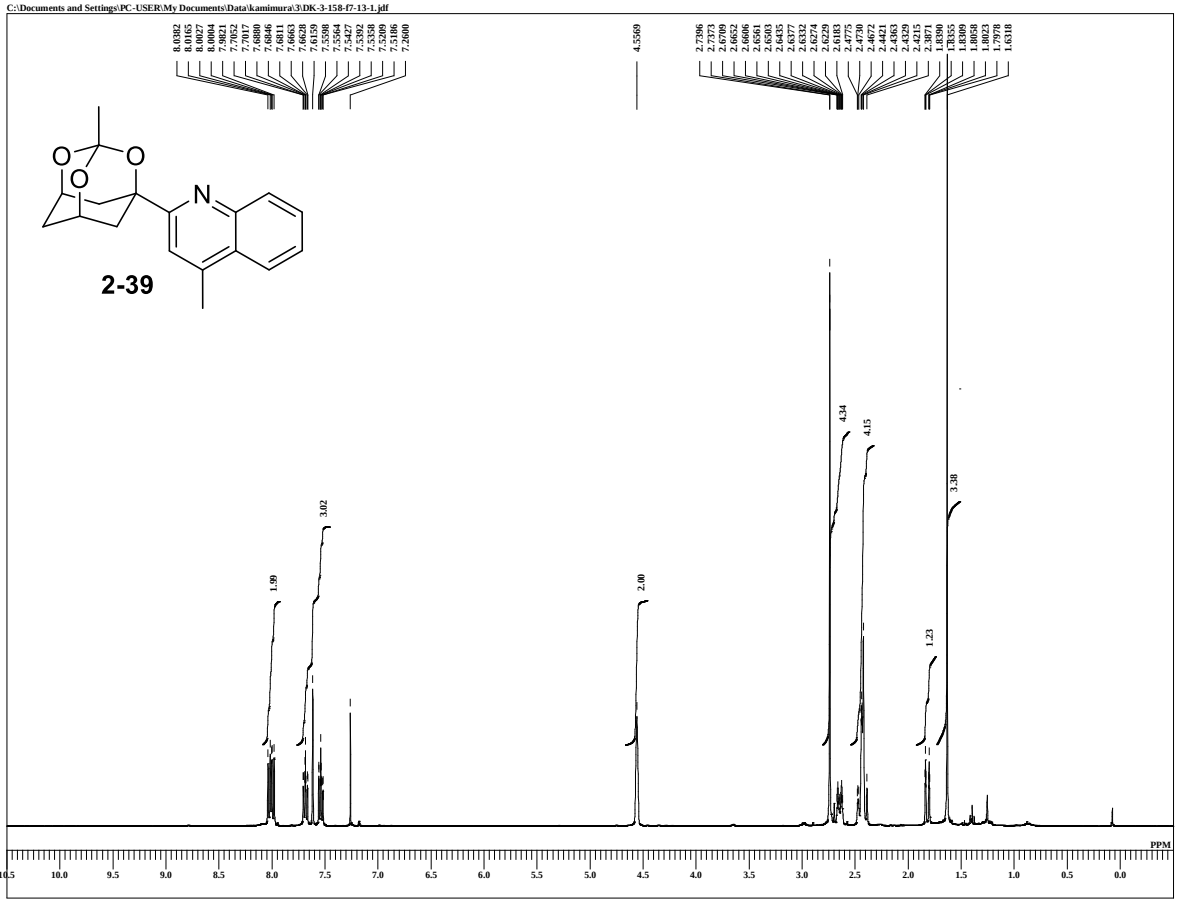


DFILE	DK-5-013-f8-13 13C.1
COMNT	single pulse decoupled gat
DATIM	09-09-2013 14:52:42
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	131072
SPO	131072
TIMES	318
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	318
ADBIT	16
RGAIN	48
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-013-f8-13 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

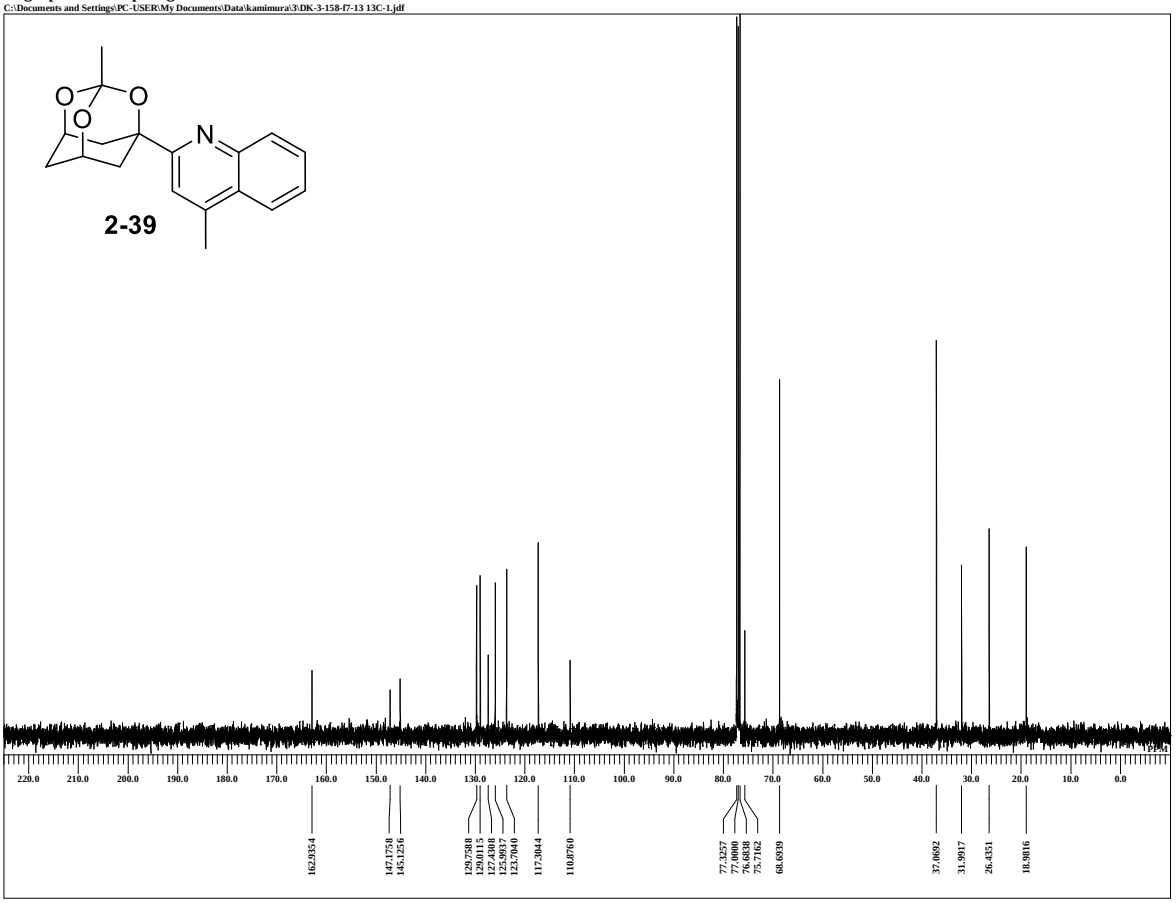
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-158-f7-13-1.jdf



DFILE DK-3-158-f7-13-1.jdf
COMNT single_pulse
DATIM 13-03-2013 08:28:59
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.38 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-158-f7-13-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.6 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-158-f7-13 13C-1.jdf

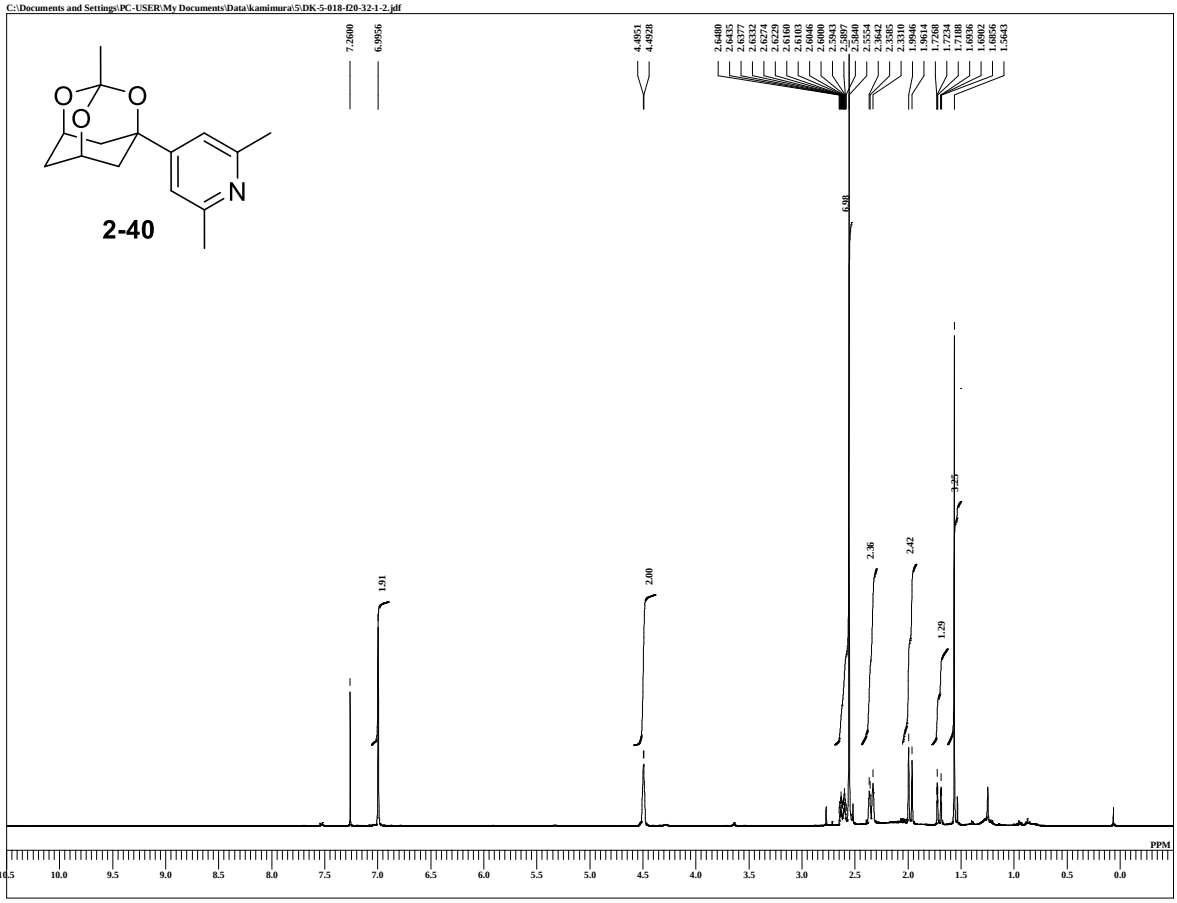


DFILE DK-3-158-f7-13 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 13-03-2013 08:35:38
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.25 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 117
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 117
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-158-f7-13 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

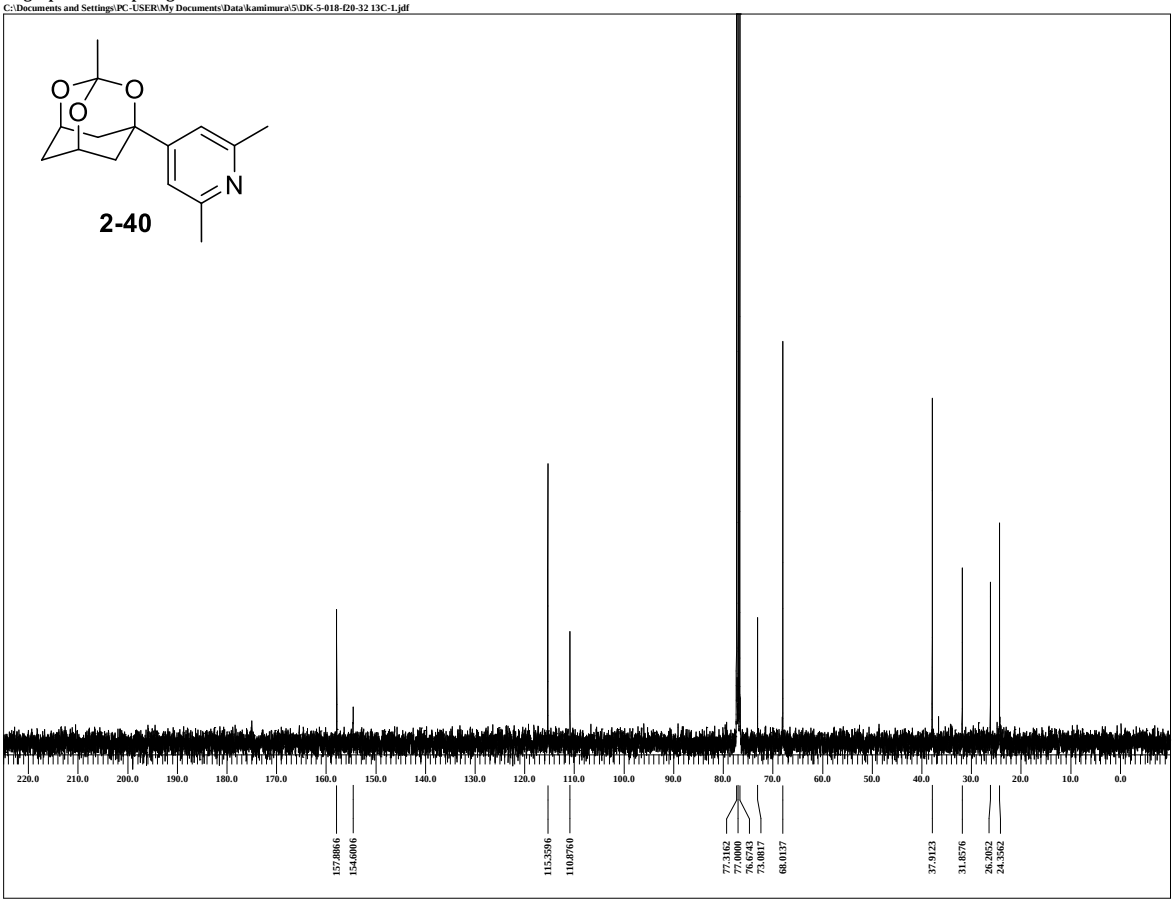
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-018-f20-32-1-2.jdf



DFILE DK-5-018-f20-32-1-2.jdf
COMNT single_pulse
DATIM 10-09-2013 08:11:59
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-5-018-f20-32-1-2.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-018-f20-32 13C-1.jdf

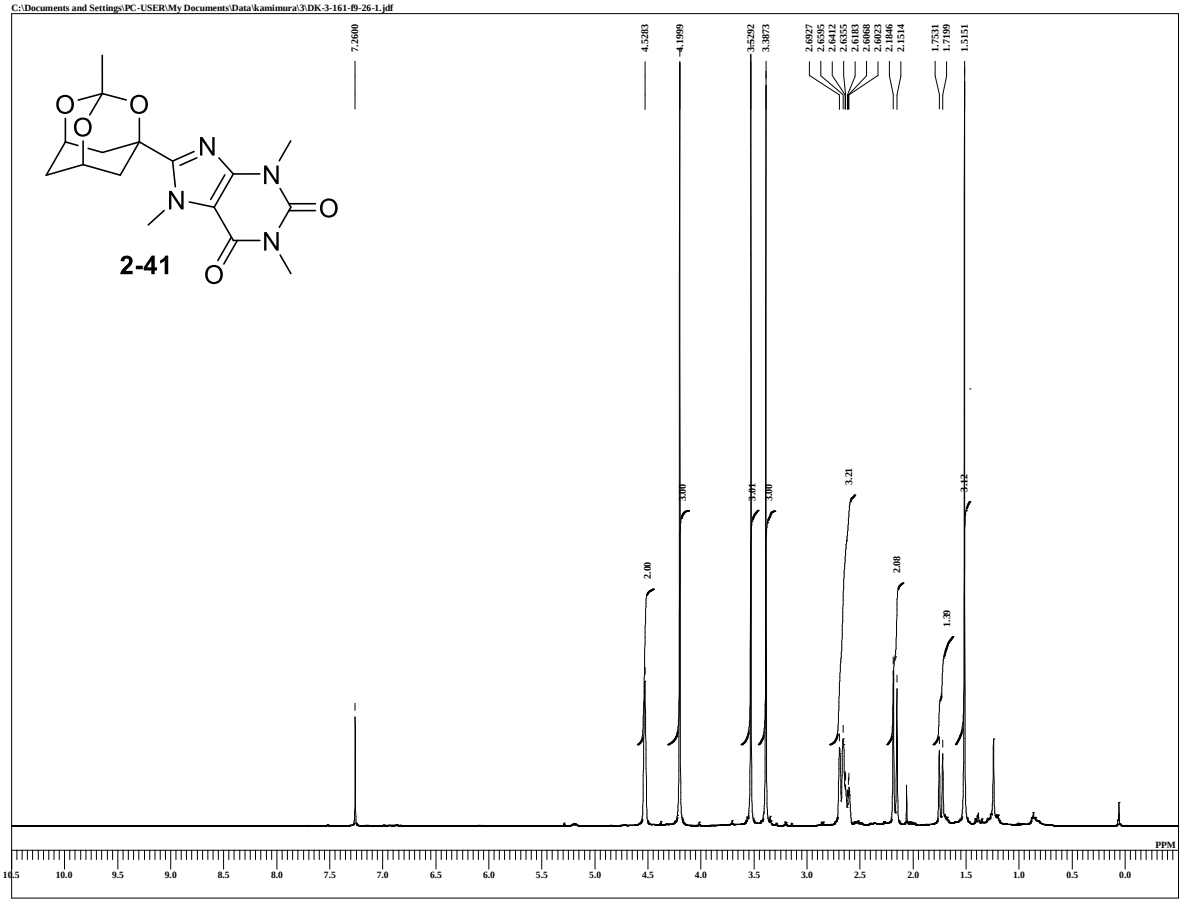


DFILE DK-5-018-f20-32 13C-1.jd
COMNT single pulse decoupled gat
DATIM 10-09-2013 09:12:37
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 329
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 329
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-018-f20-32 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

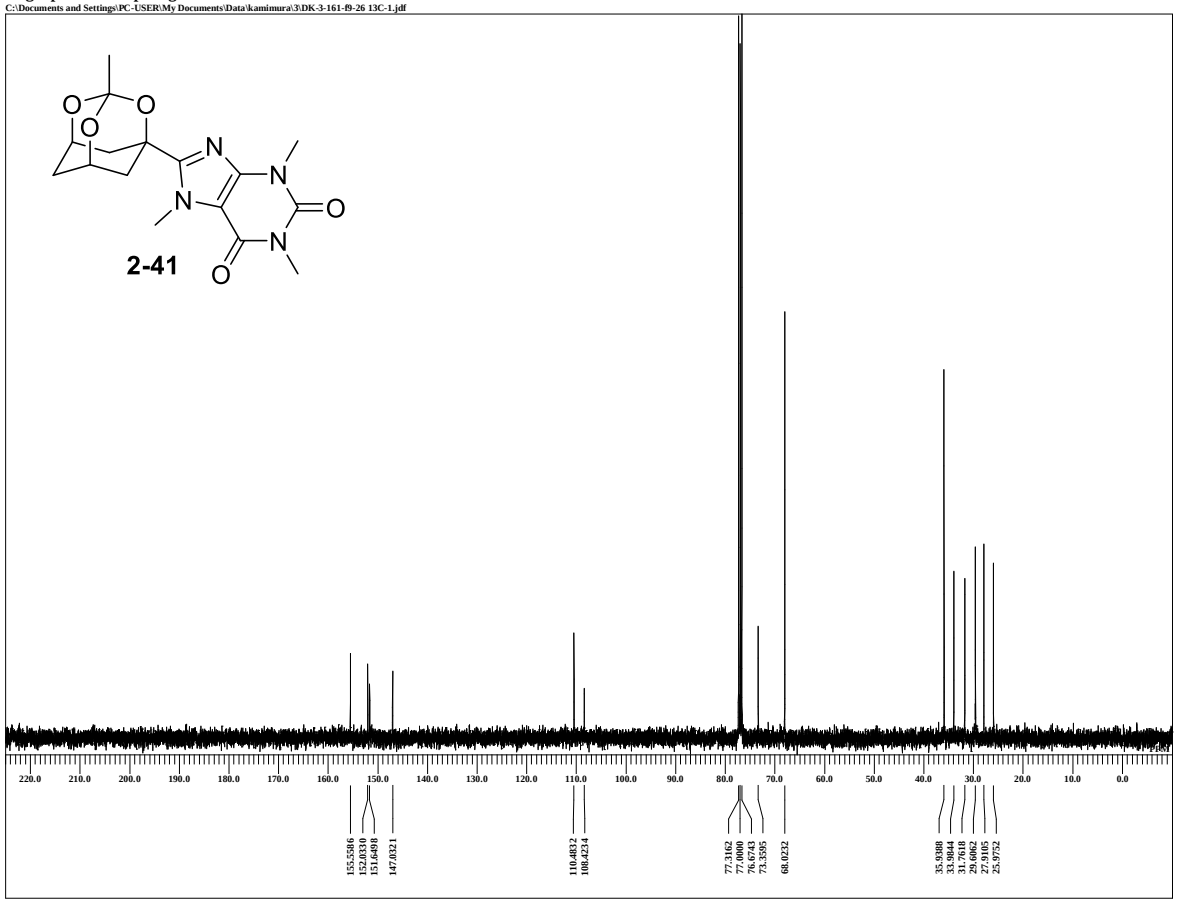
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-161-f9-26-1.jdf




```
DFILE   DK-3-161-f9-26-1.jdf
COMNT   single_pulse
DATIM   15-03-2013 11:27:07
MENUF
OBNUC   1H
OFR         395.88 MHz
OBFRQ       395.88 MHz
OBSET         6.28 KHz
OBFIN         0.87 Hz
PW1           6.38 usec
DEADT         0.00 usec
PREDL         0.00000 msec
IWT           1.0000 sec
POINT        16384
SPO          16384
TIMES           16
DUMMY            1
FREQU      7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM         2.2073 sec
PD            2.0000 sec
SCANS           16
ADBIT           16
RGAIN           36
BF            0.10 Hz
T1            0.00
T2            0.00
T3          100.00
T4          100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR         395.88 MHz
IRSET         6.28 KHz
IRFIN         0.87 Hz
IRRPW          147 usec
IRATN           79
DFILE   DK-3-161-f9-26-1.jdf
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED           0 Hz
FILDC
FILDF
CTEMP        20.4 c
SLVNT   CDCL3
EXREF        7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-161-f9-26 13C-1.jdf

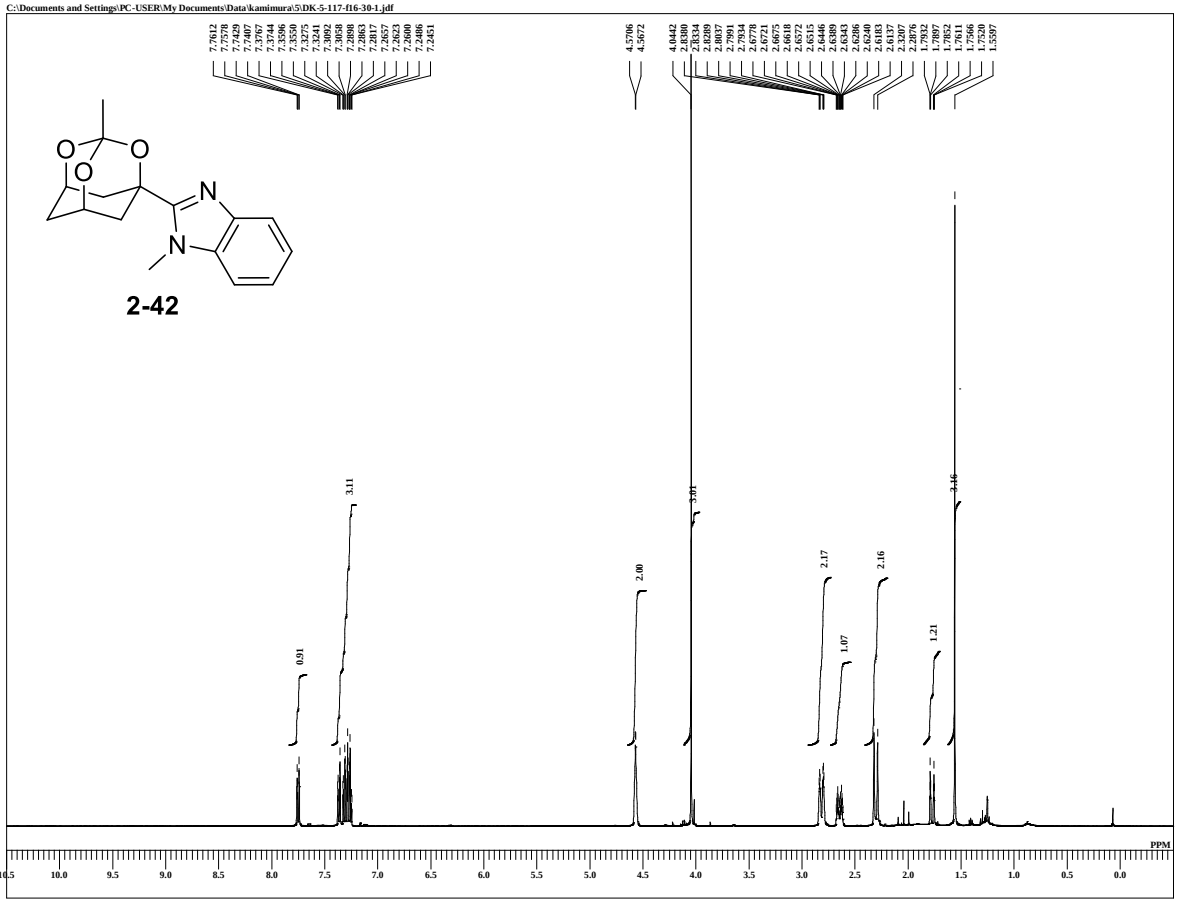



```
DFILE   DK-3-161-f9-26 13C-1.jdf
COMNT   single pulse decoupled gat
DATIM   15-03-2013 11:35:52
MENUF
OBNUC   13C
OFR         99.55 MHz
OBFRQ       99.55 MHz
OBSET         5.13 KHz
OBFIN         0.98 Hz
PW1           3.25 usec
DEADT         0.00 usec
PREDL         0.00000 msec
IWT           1.0000 sec
POINT        32768
SPO          32768
TIMES          160
DUMMY            4
FREQU      31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM         1.0486 sec
PD            2.0000 sec
SCANS          160
ADBIT           16
RGAIN           60
BF            1.00 Hz
T1            0.00
T2            0.00
T3          100.00
T4          100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR         395.88 MHz
IRSET         6.28 KHz
IRFIN         0.87 Hz
IRRPW          115 usec
IRATN           79
DFILE   DK-3-161-f9-26 13C-1.jdf
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED           0 Hz
FILDC
FILDF
CTEMP        20.5 c
SLVNT   CDCL3
EXREF        77.00 ppm
```

スペクトルデータ

single_pulse

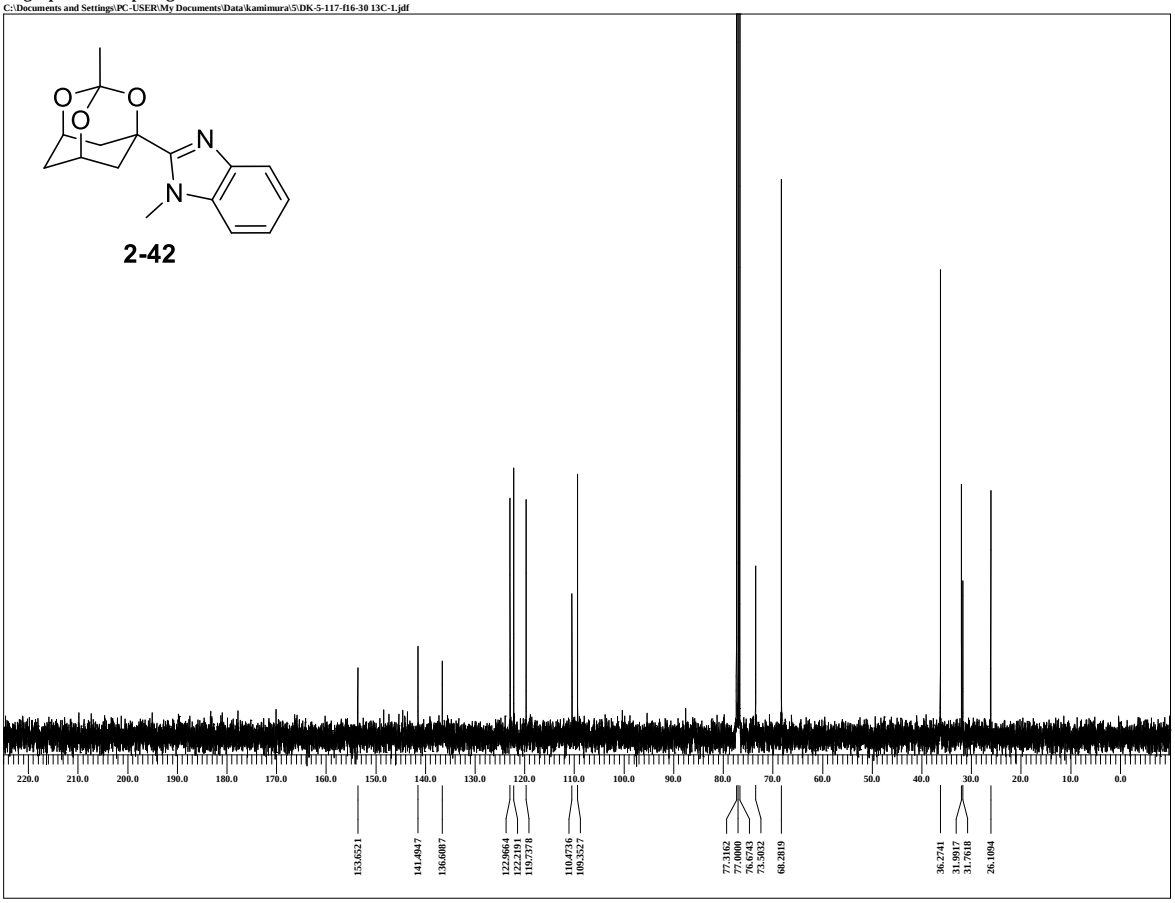
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-117-f16-30-1.jdf




```
DFILE   DK-5-117-f16-30-1.jdf
COMNT   single_pulse
DATIM   08-10-2013 10:40:15
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.44 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT      16384
SPO        16384
TIMES          8
DUMMY          1
FREQU    7422.80 Hz
FLT       30000 Hz
DELAY      16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS          8
ADBIT         16
RGAIN         36
BF          0.10 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       115 usec
IRATN          79
DFILE   DK-5-117-f16-30-1.jdf
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV          0
LGAIN          0
LKPHS          0
LKSIG          0
CSPED          0 Hz
FILDC
FILDF
CTEMP       22.7 c
SLVNT   CDCL3
EXREF       7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-117-f16-30 13C-1.jdf

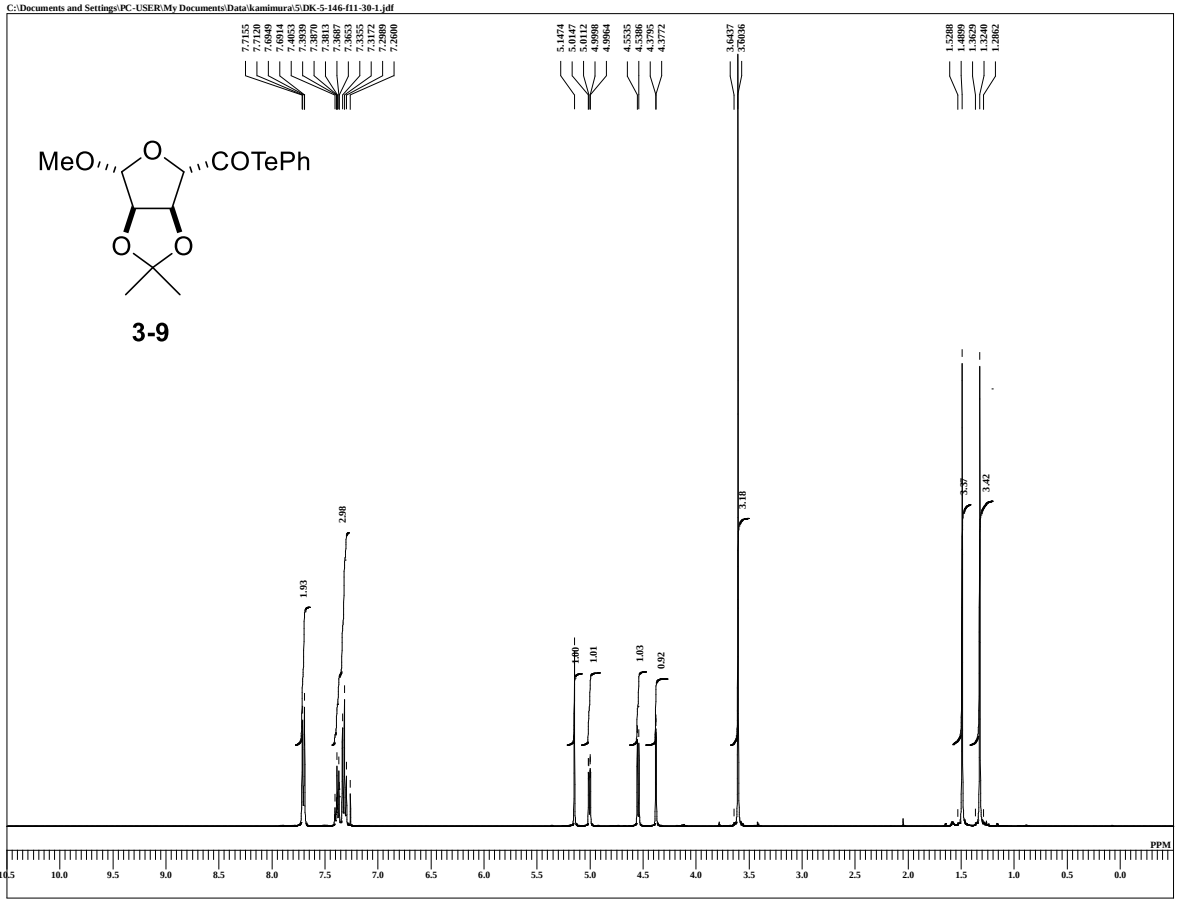



```
DFILE   DK-5-117-f16-30 13C-1.jd
COMNT   single pulse decoupled gat
DATIM   08-10-2013 10:46:36
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
PW1        3.03 usec
DEADT      0.00 usec
PREDL      0.00000 msec
IWT        1.0000 sec
POINT     32768
SPO       32768
TIMES       113
DUMMY         4
FREQU   31250.00 Hz
FLT      125000 Hz
DELAY     20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS       113
ADBIT        16
RGAIN        60
BF         1.00 Hz
T1         0.00
T2         0.00
T3       100.00
T4       100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET      6.28 KHz
IRFIN      0.87 Hz
IRRPW      115 usec
IRATN         79
DFILE   DK-5-117-f16-30 13C-1.jd
SF
LKSET     13.20 KHz
LKFIN      75.7 Hz
LKLEV         0
LGAIN         0
LKPHS         0
LKSIG         0
CSPED         0 Hz
FILDC
FILDF
CTEMP      22.9 c
SLVNT   CDCL3
EXREF      77.00 ppm
```

スペクトルデータ

single_pulse

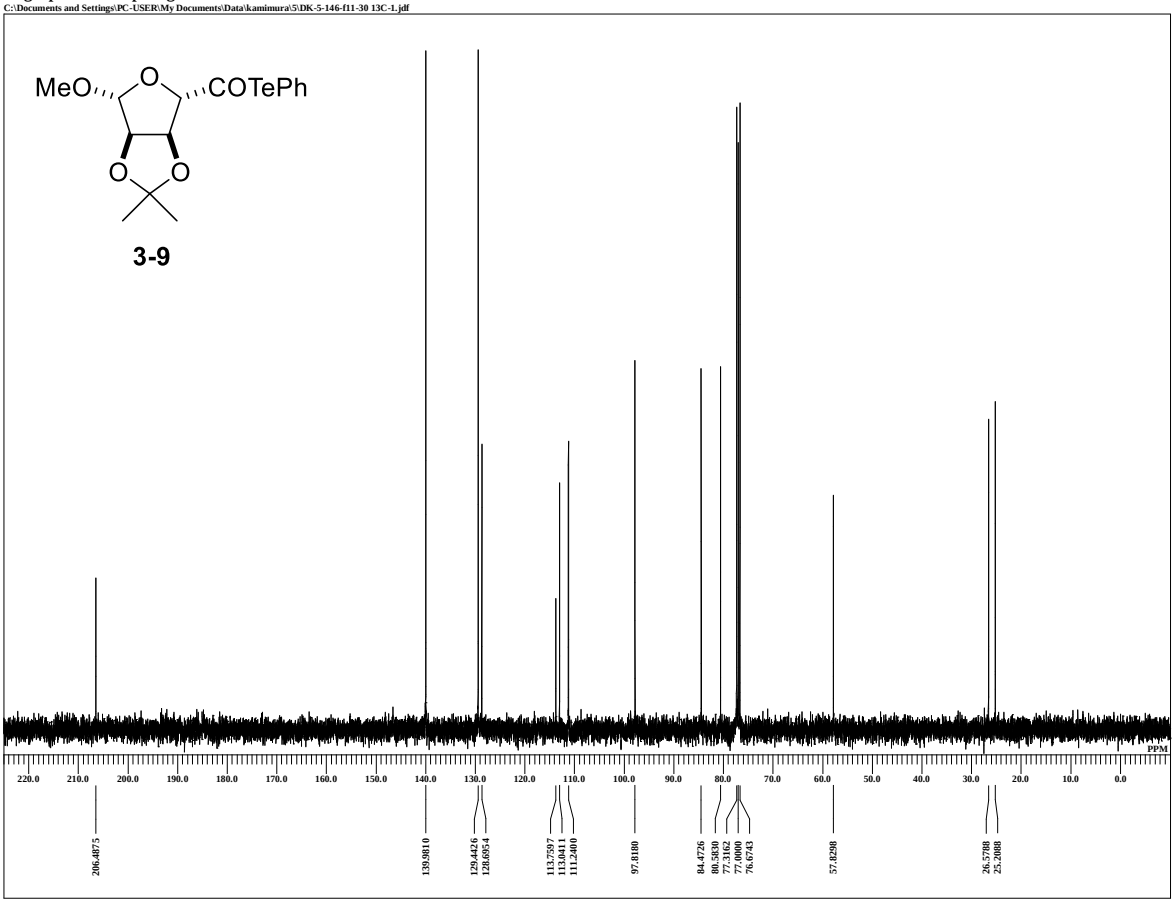
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-146-f11-30-1.jdf



DFILE DK-5-146-f11-30-1.jdf
COMNT single_pulse
DATIM 28-10-2013 14:38:45
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-5-146-f11-30-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.1 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-146-f11-30 13C-1.jdf

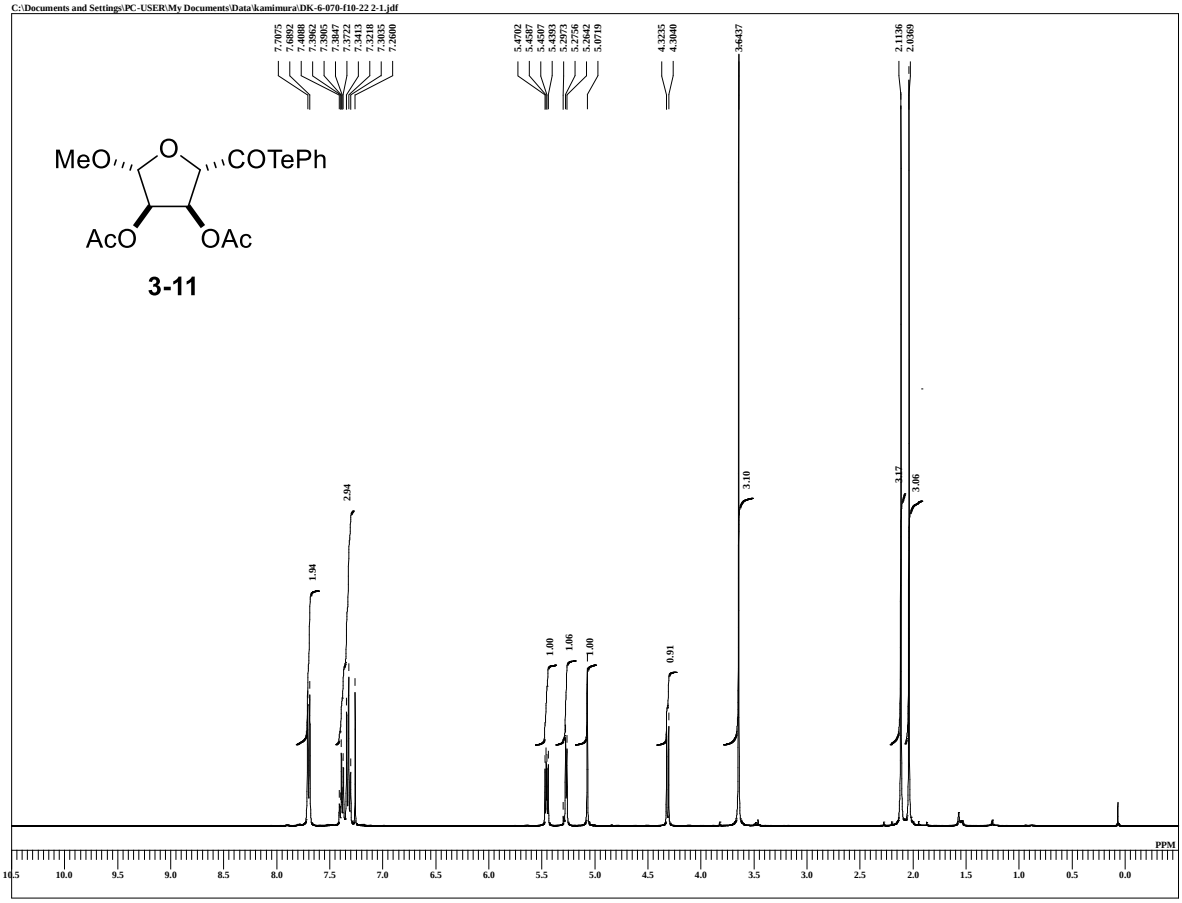


DFILE DK-5-146-f11-30 13C-1.jd
COMNT single pulse decoupled gat
DATIM 28-10-2013 14:41:58
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 52
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 52
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-146-f11-30 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

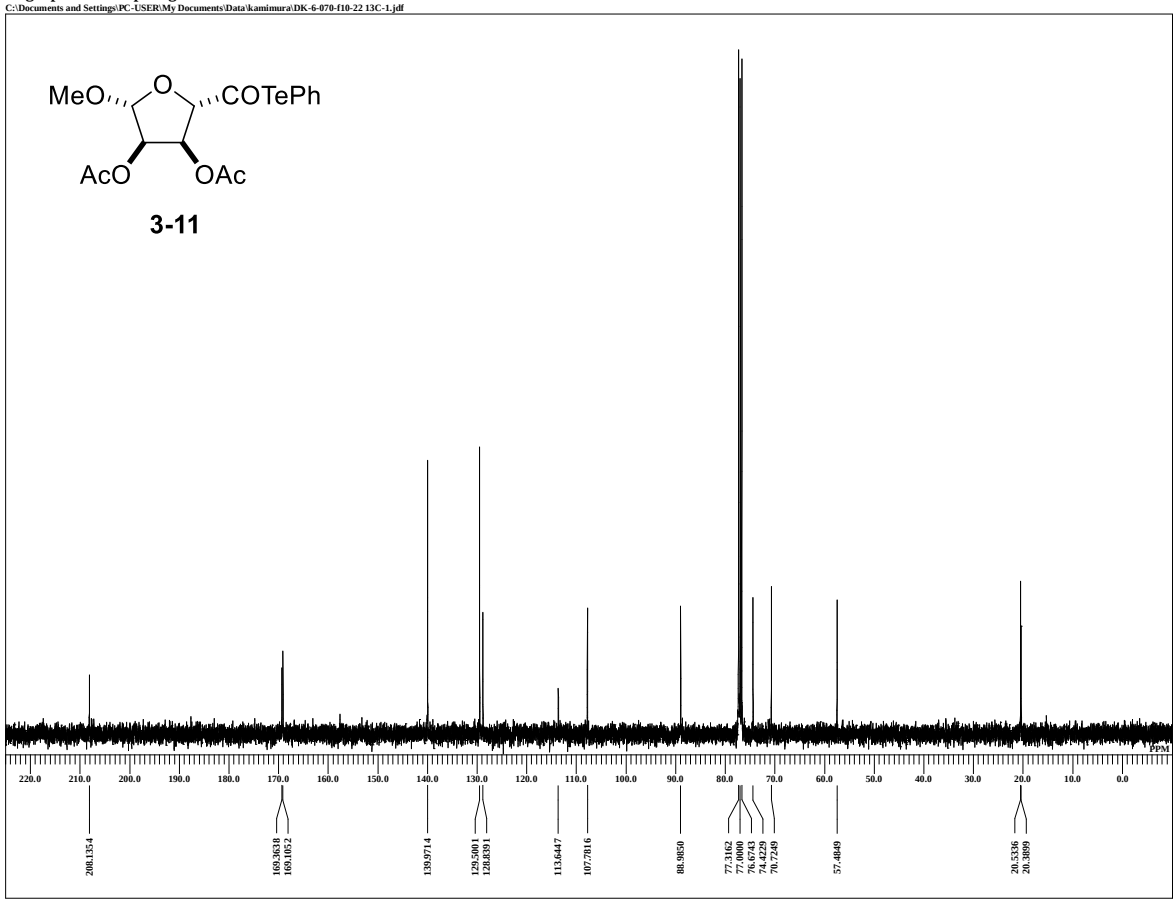
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-070-f10-22 2-1.jdf



DFILE DK-6-070-f10-22 2-1.jdf
COMNT single_pulse
DATIM 01-02-2014 16:20:59
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 42
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-070-f10-22 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-070-f10-22 13C-1.jdf

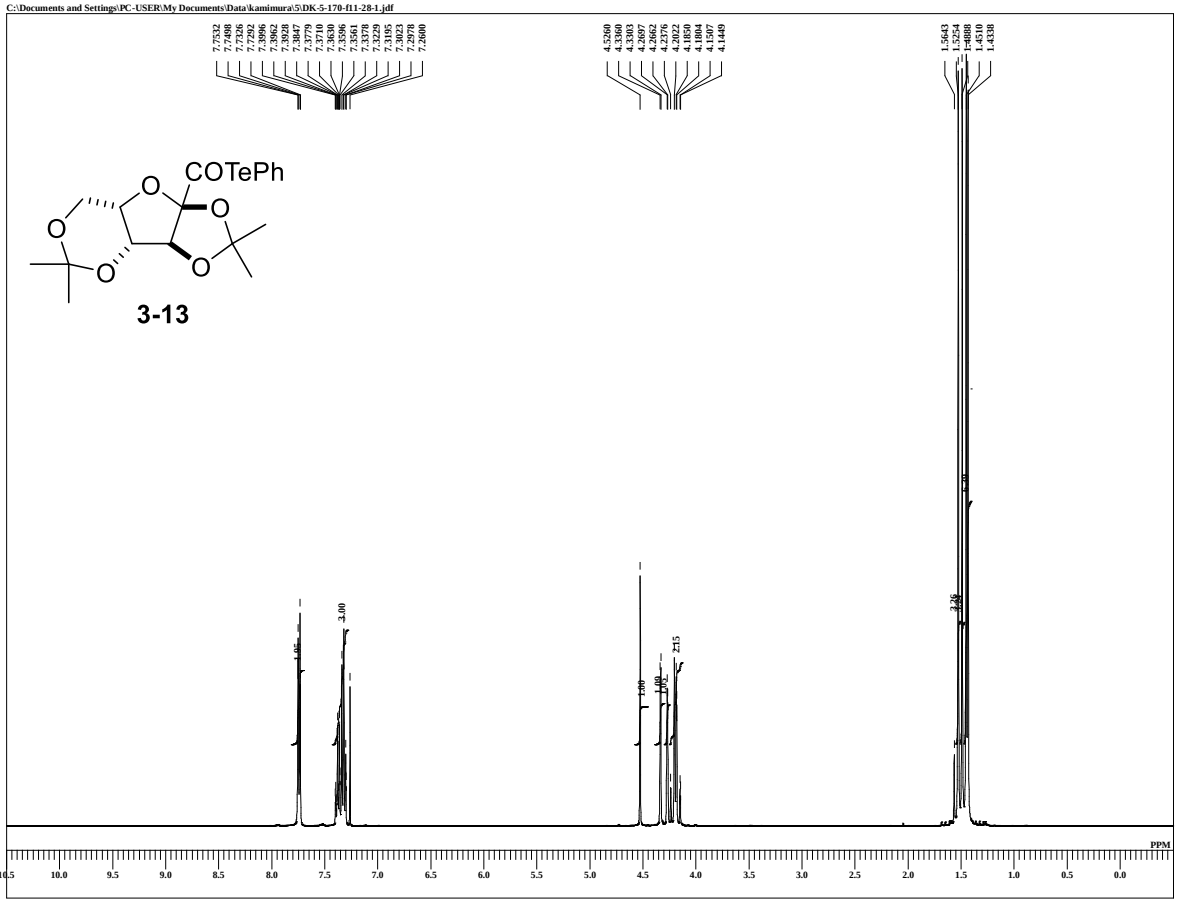


DFILE DK-6-070-f10-22 13C-1.jd
COMNT single pulse decoupled gat
DATIM 06-01-2014 19:04:26
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 151
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 151
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-070-f10-22 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

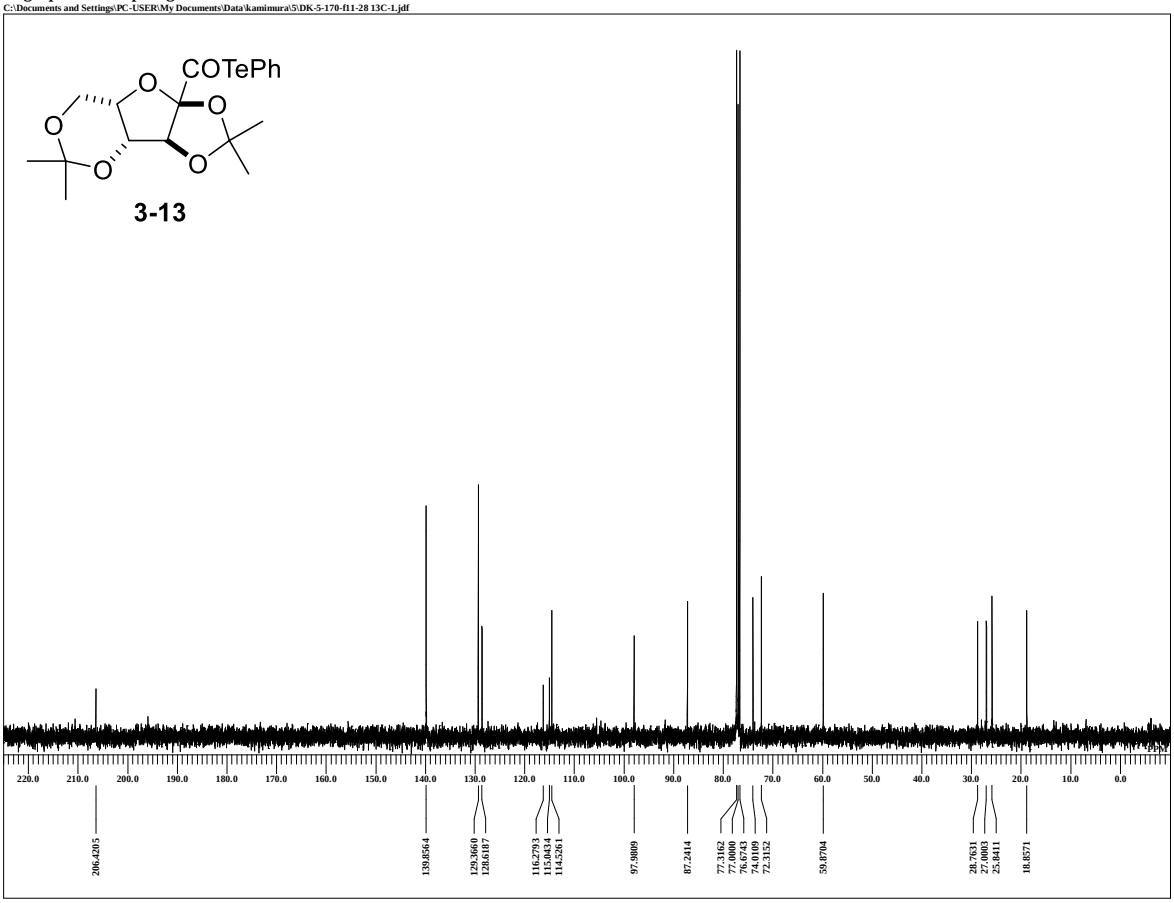
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-170-f11-28-1.jdf



DFILE	DK-5-170-f11-28-1.jdf
COMNT	single_pulse
DATIM	07-11-2013 09:53:18
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	38
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-170-f11-28-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-170-f11-28 13C-1.jdf

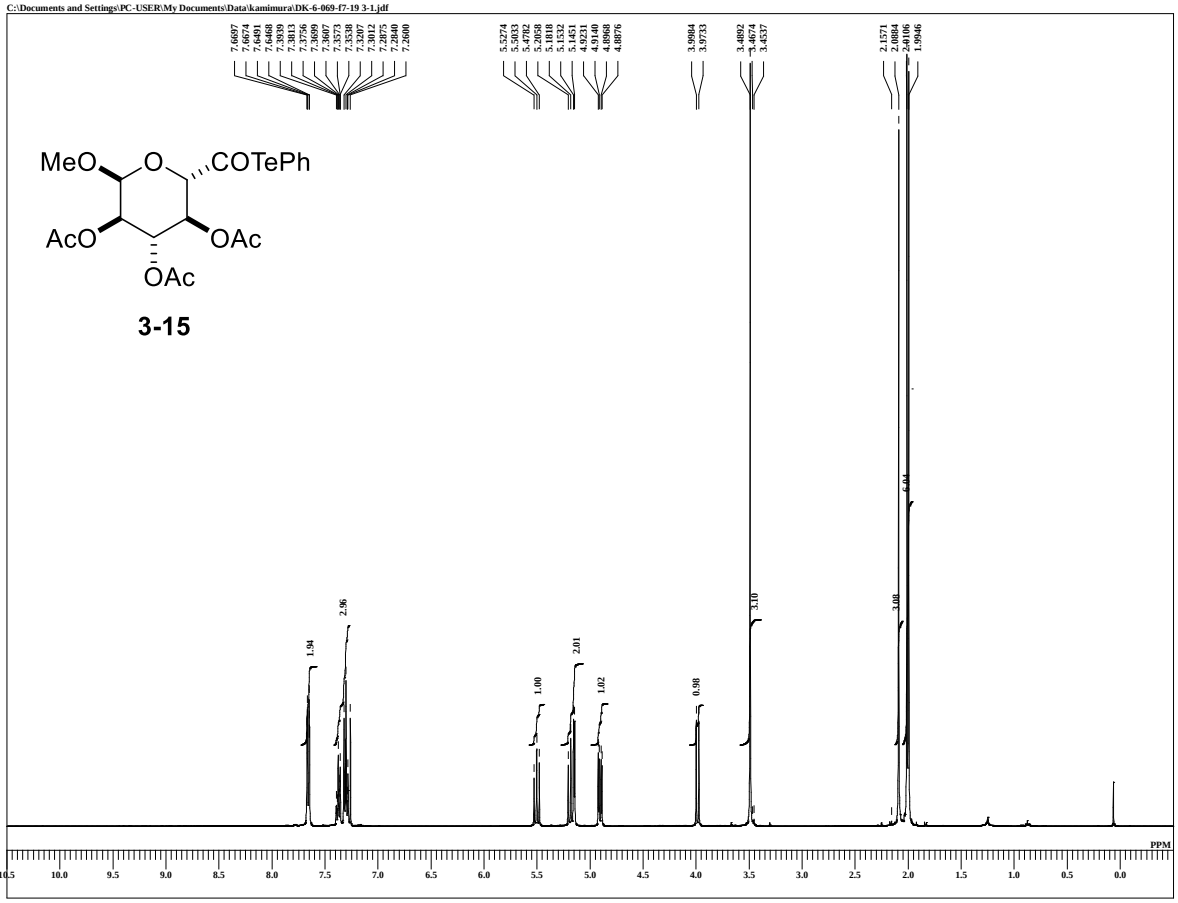


DFILE	DK-5-170-f11-28 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	07-11-2013 10:42:49
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	104
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	104
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-170-f11-28 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

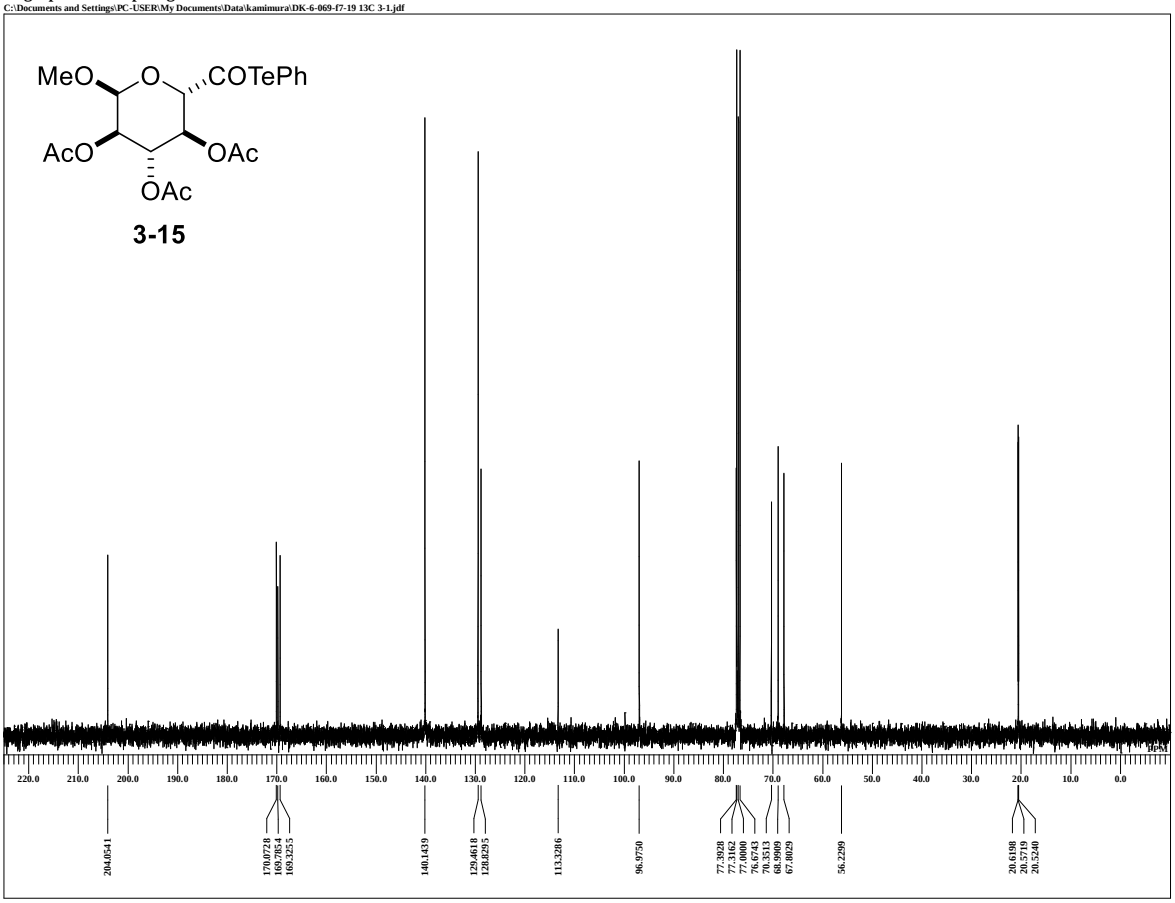
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-069-f7-19 3-1.jdf



DFILE	DK-6-069-f7-19 3-1.jdf
COMNT	single_pulse
DATIM	01-02-2014 16:58:33
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-069-f7-19 3-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-069-f7-19 13C 3-1.jdf

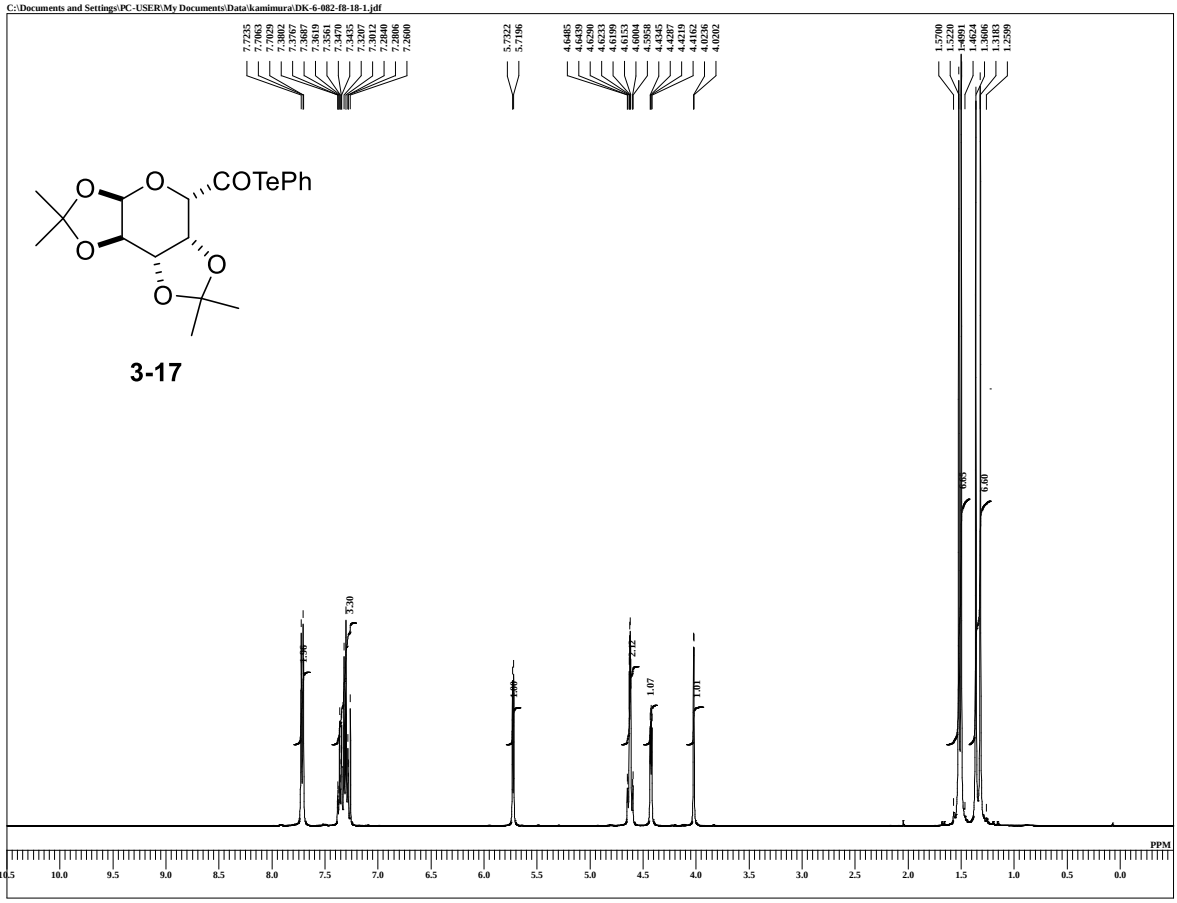


DFILE	DK-6-069-f7-19 13C 3-1.j
COMNT	single pulse decoupled gat
DATIM	01-02-2014 17:04:23
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	101
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	101
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-069-f7-19 13C 3-1.j
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

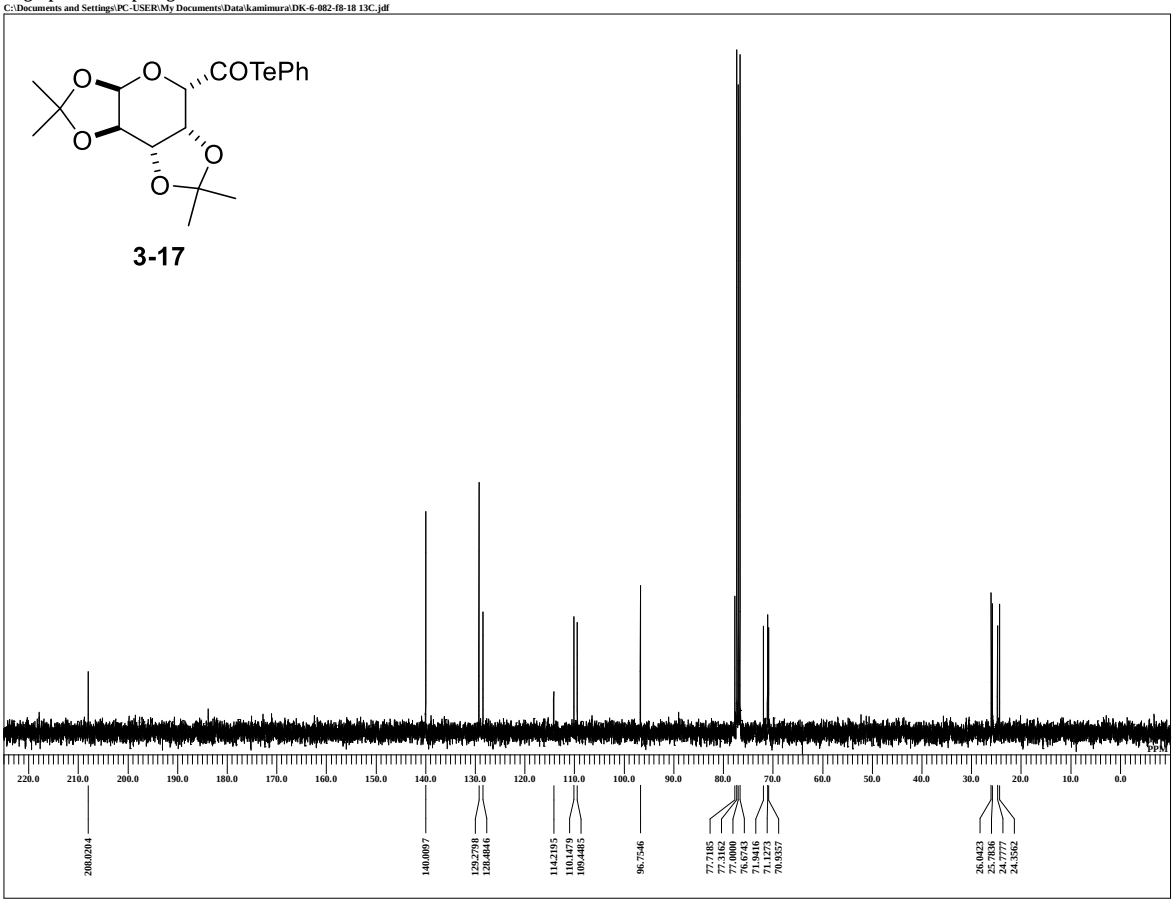
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-082-f8-18-1.jdf



DFILE DK-6-082-f8-18-1.jdf
COMNT single_pulse
DATIM 14-01-2014 18:18:19
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-082-f8-18-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-082-f8-18 13C.jdf

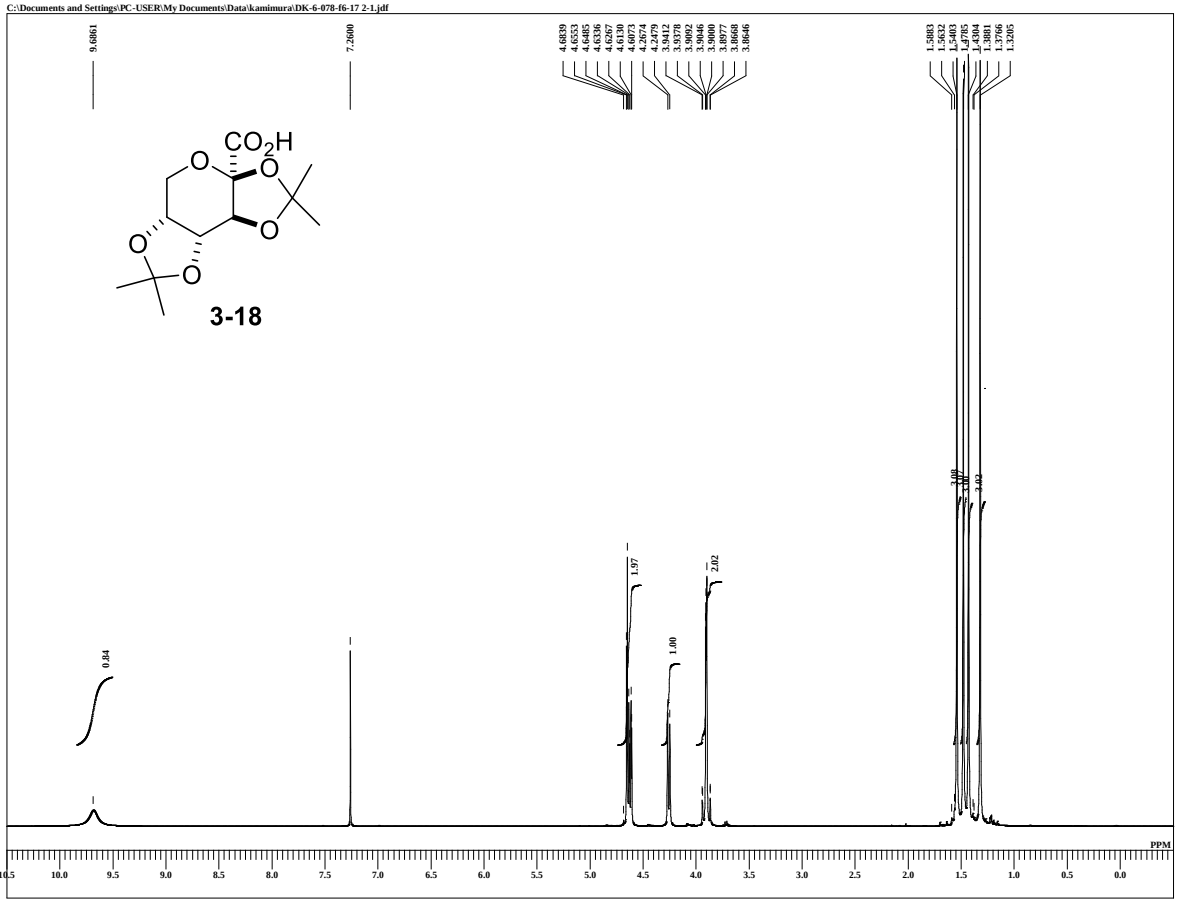


DFILE DK-6-082-f8-18 13C.jdf
COMNT single pulse decoupled gat
DATIM 14-01-2014 18:24:06
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 103
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 103
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-082-f8-18 13C.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

DK-6-078-f6-17 2

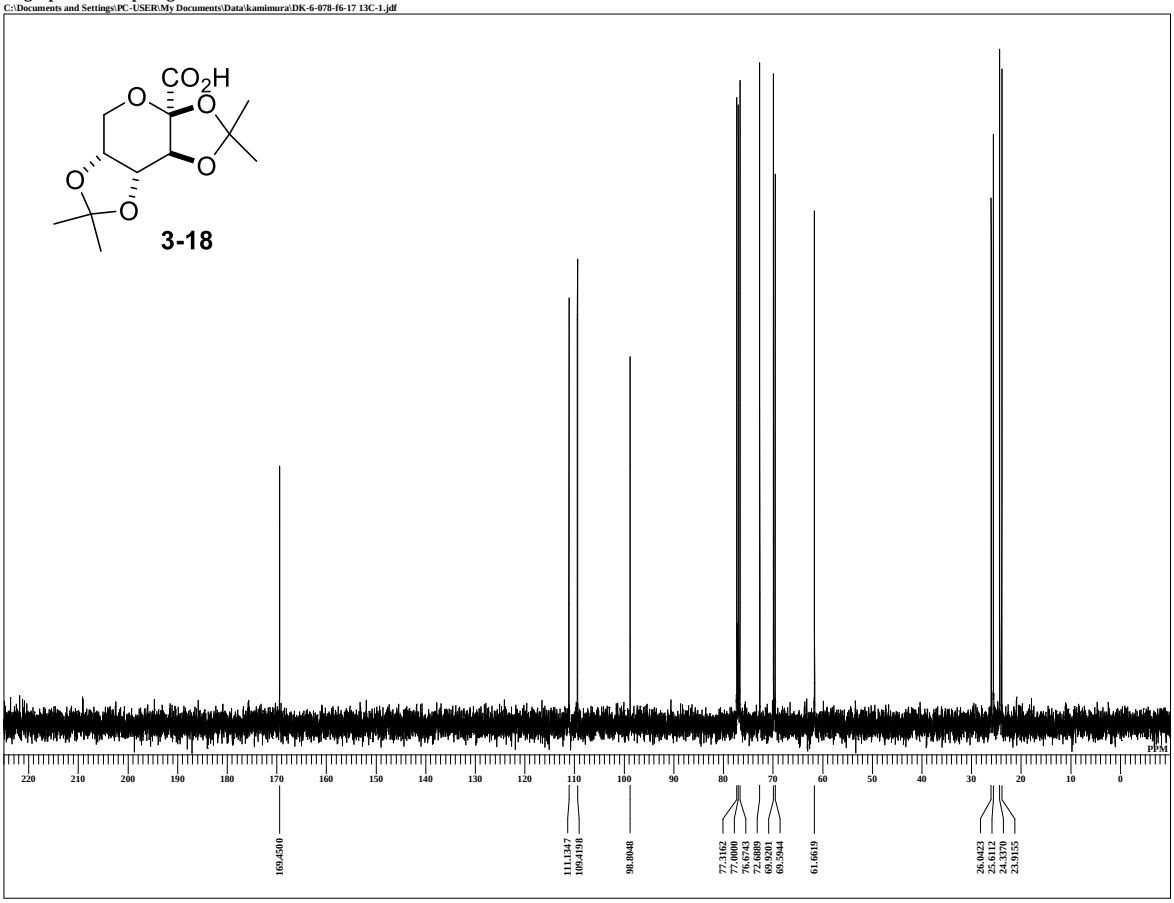
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-078-f6-17 2-1.jdf



DFILE DK-6-078-f6-17 2-1.jdf
COMNT DK-6-078-f6-17 2
DATIM 10-02-2014 19:01:21
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 24
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-078-f6-17 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.1 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-078-f6-17 13C-1.jdf

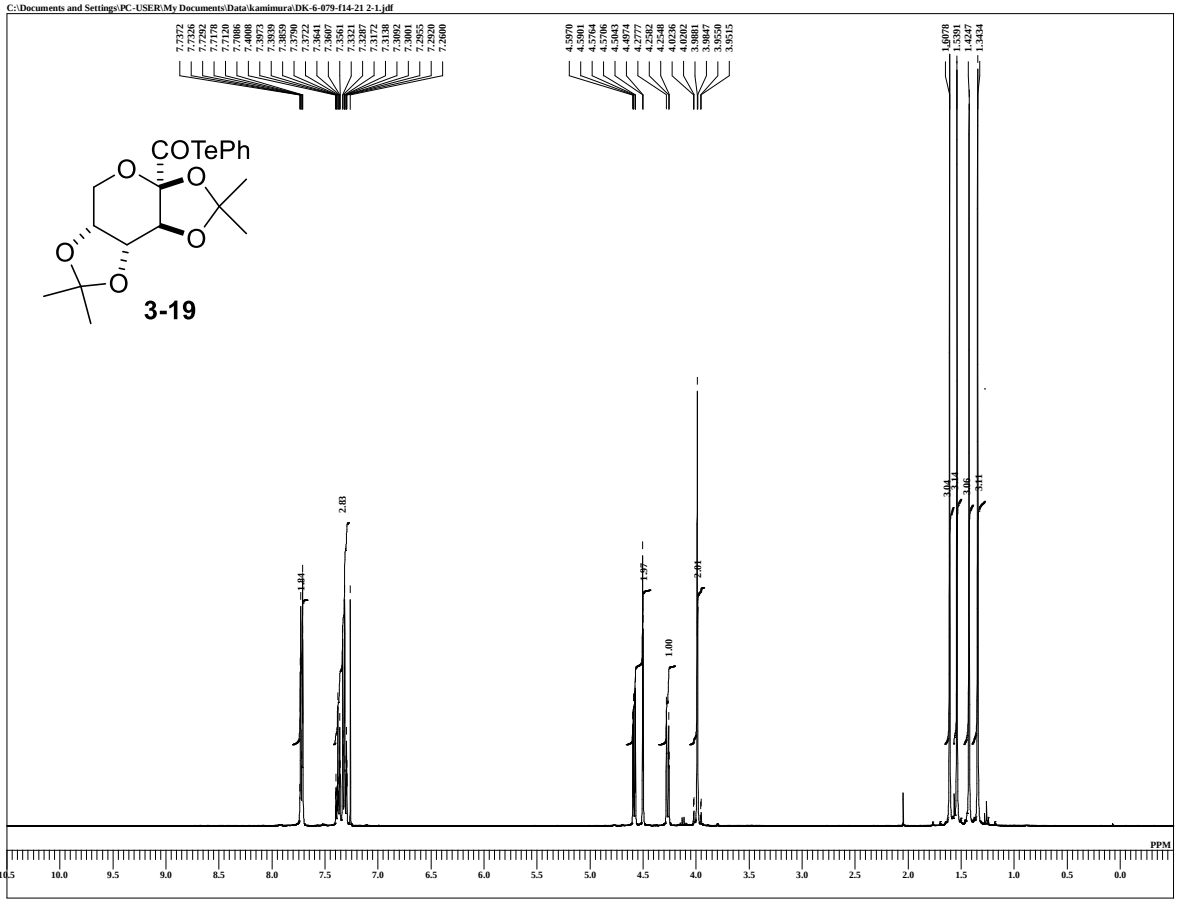


DFILE DK-6-078-f6-17 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 10-02-2014 15:14:06
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 41
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 41
ADBIT 16
RGAIN 58
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-078-f6-17 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

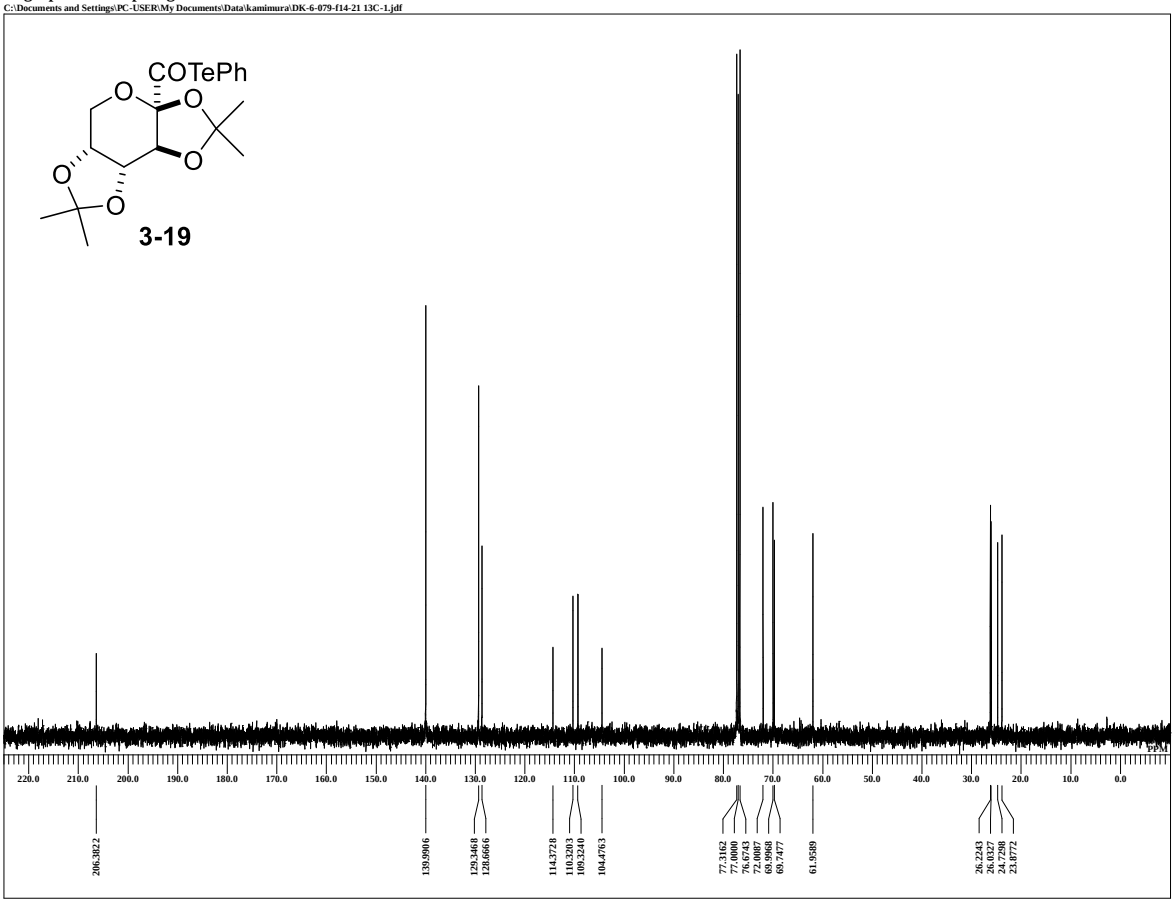
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-079-f14-21 2-1.jdf



DFILE	DK-6-079-f14-21 2-1.jdf
COMNT	single_pulse
DATIM	27-01-2014 09:33:31
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	38
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-079-f14-21 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-079-f14-21 13C-1.jdf

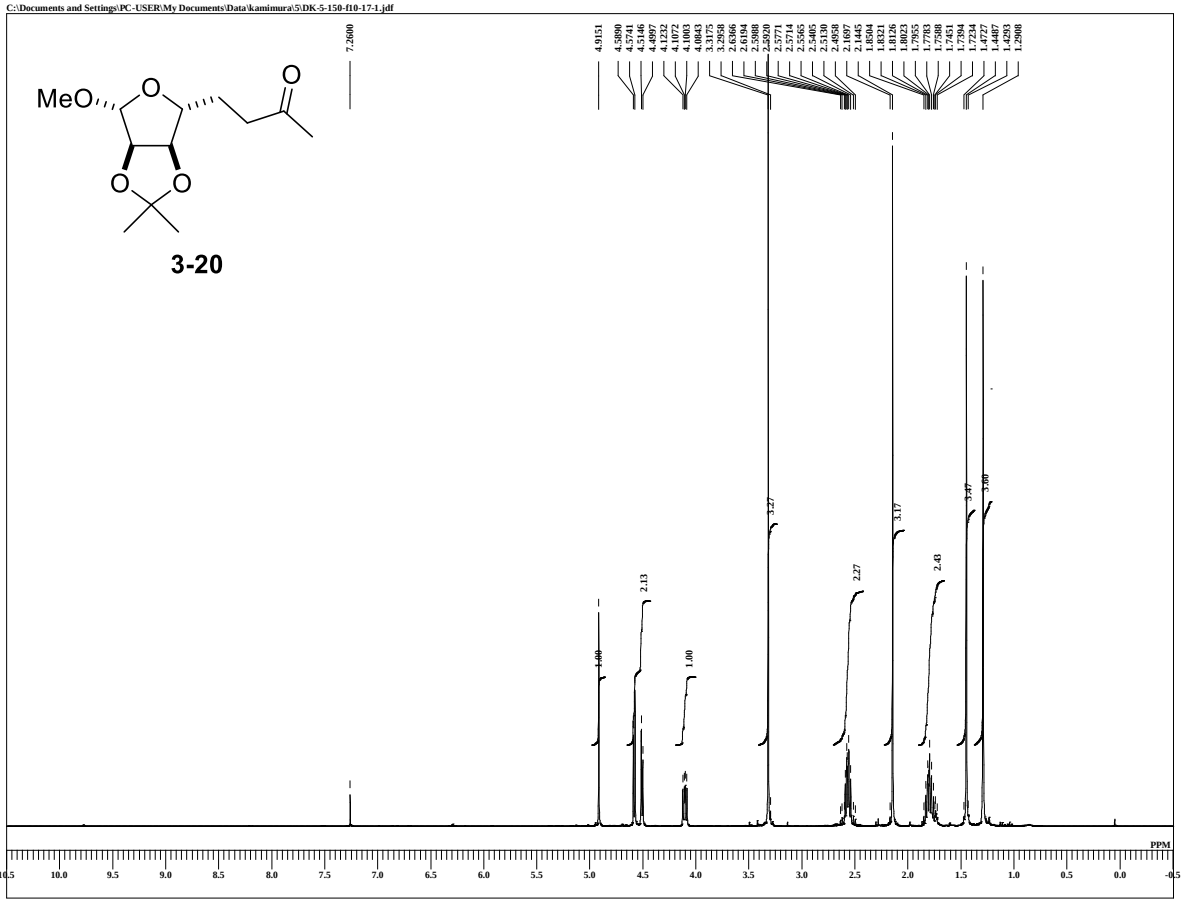


DFILE	DK-6-079-f14-21 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	10-01-2014 17:56:23
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	86
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	86
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-079-f14-21 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

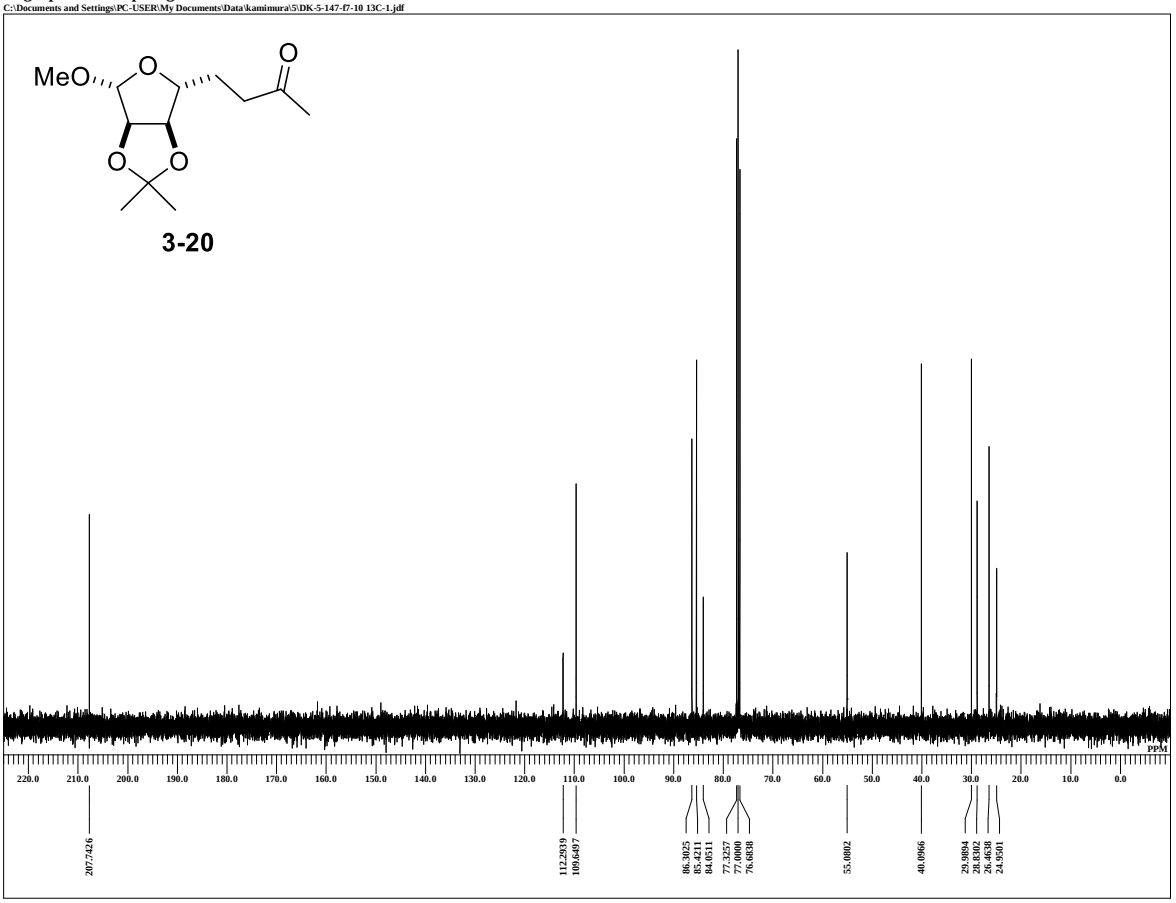
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-150-f10-17-1.jdf



DFILE DK-5-150-f10-17-1.jdf
COMNT single_pulse
DATIM 30-10-2013 08:17:54
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-5-150-f10-17-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.5 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-147-f7-10 13C-1.jdf

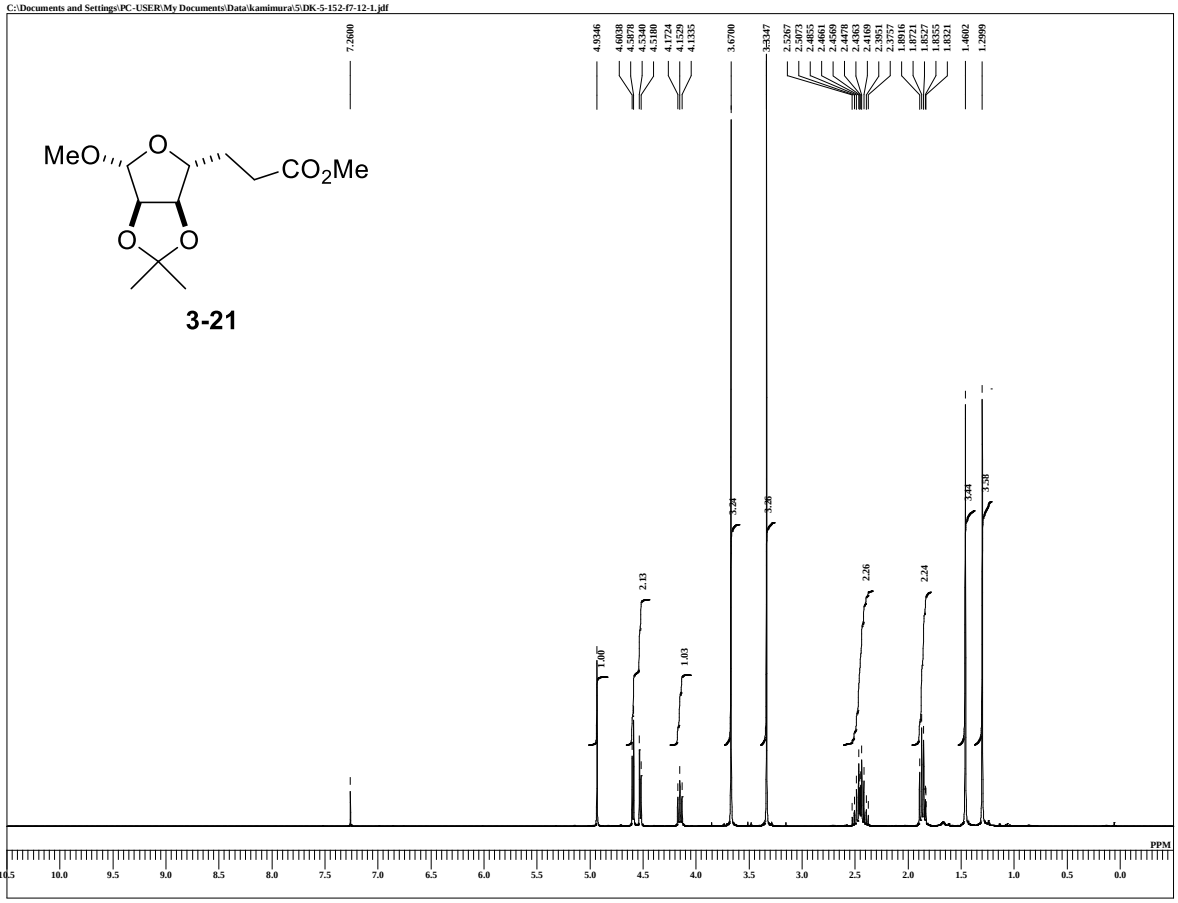


DFILE DK-5-147-f7-10 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 29-10-2013 09:12:19
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 102
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 102
ADBIT 16
RGAIN 60
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-147-f7-10 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.4 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

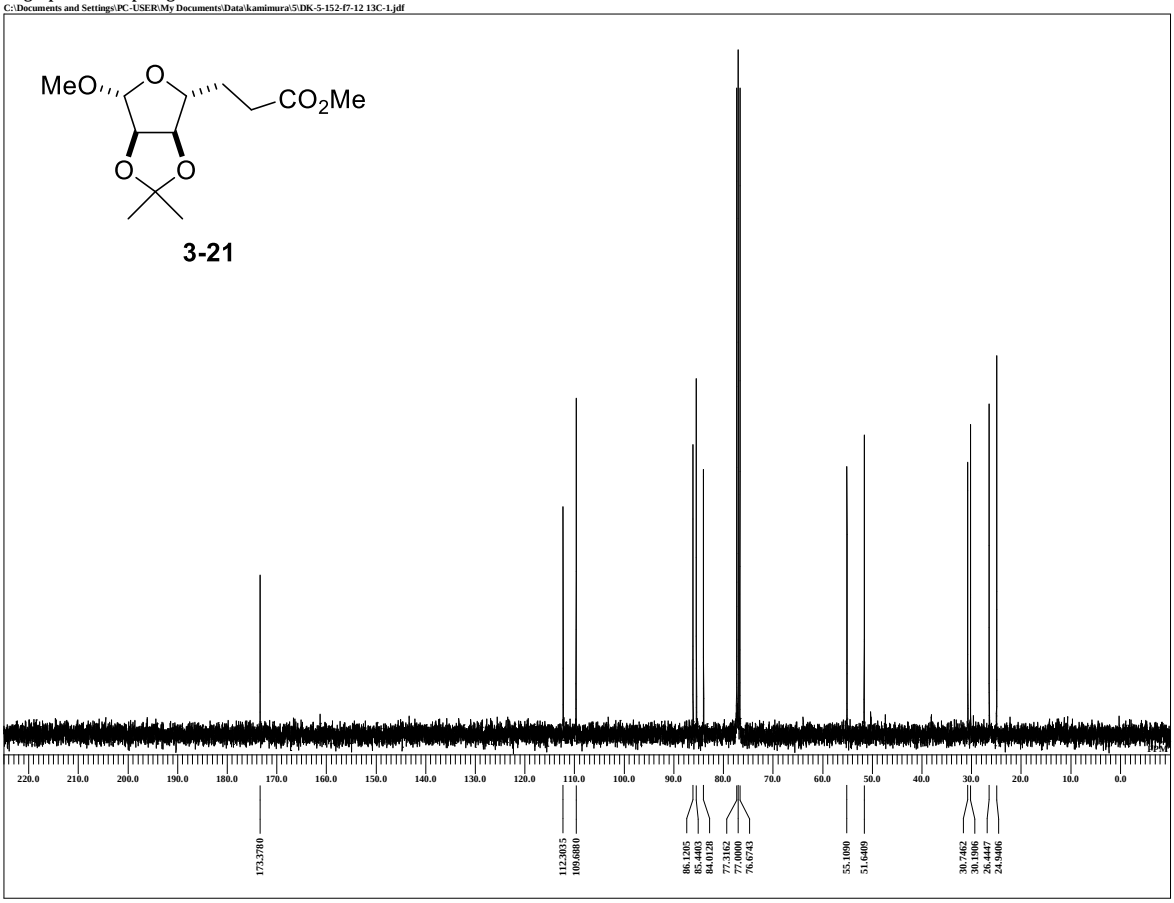
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-152-f7-12-1.jdf



DFILE DK-5-152-f7-12-1.jdf
COMNT single_pulse
DATIM 30-10-2013 18:59:44
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-152-f7-12-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.7 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-152-f7-12 13C-1.jdf

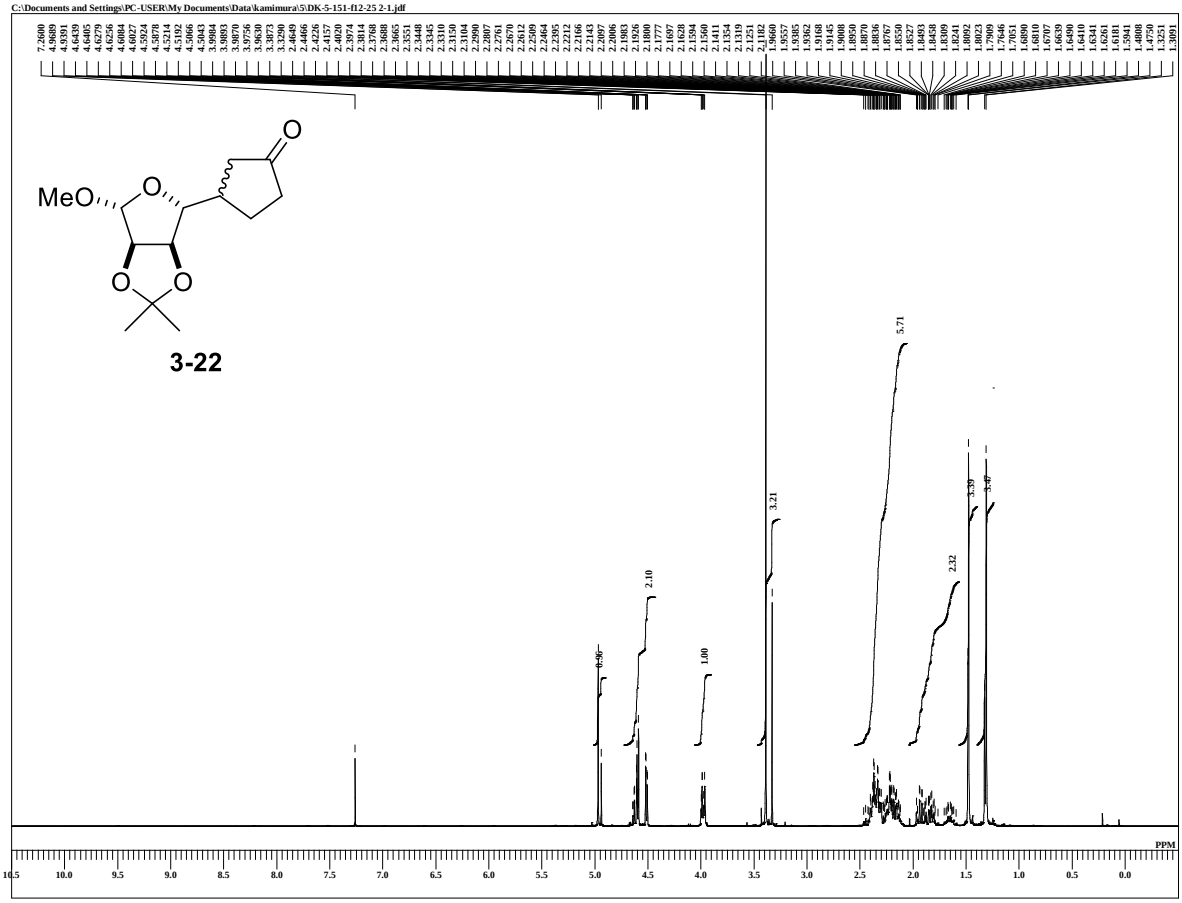


DFILE DK-5-152-f7-12 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 30-10-2013 19:04:04
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 74
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 74
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-152-f7-12 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.9 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

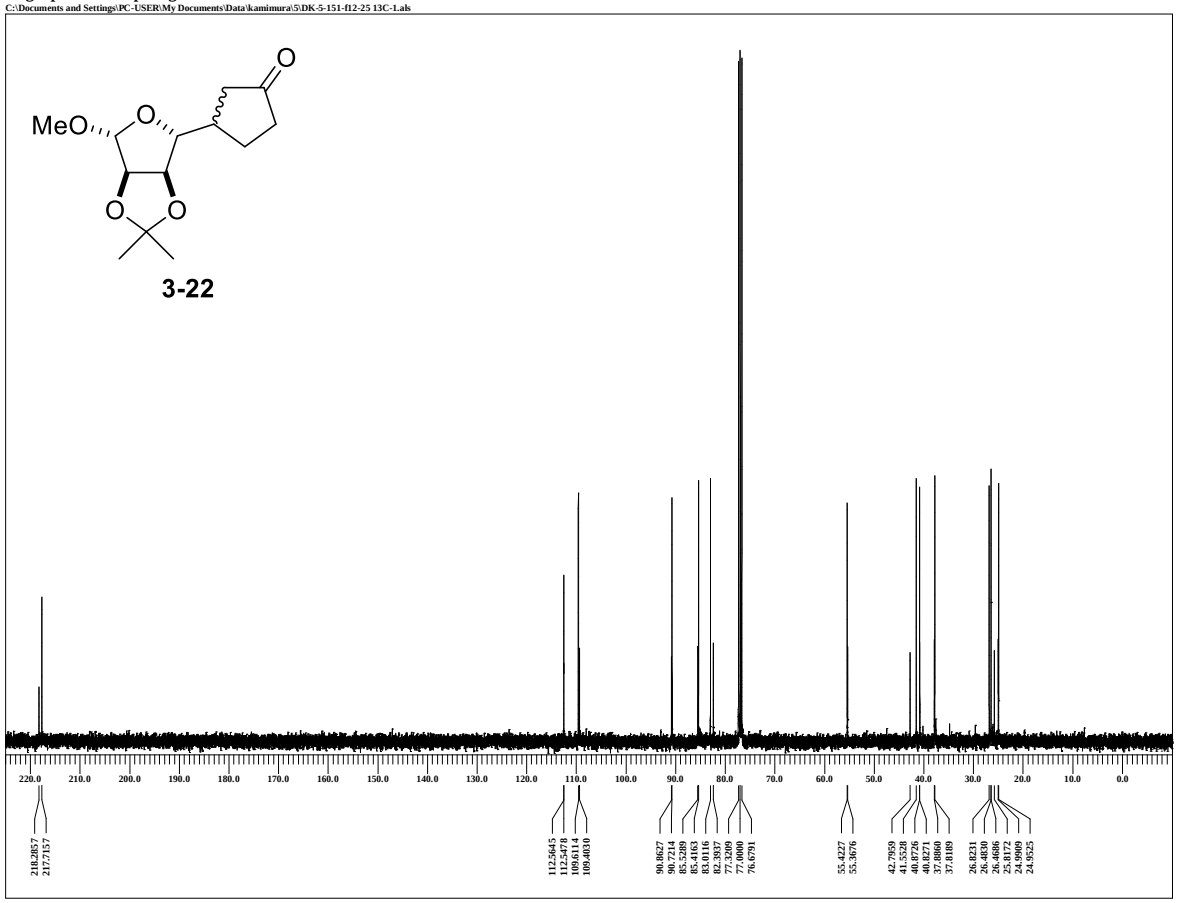
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-151-f12-25 2-1.jdf



DFILE	DK-5-151-f12-25 2-1.jdf
COMNT	single_pulse
DATIM	30-10-2013 16:28:02
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-151-f12-25 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	25.6 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-151-f12-25 13C-1.als

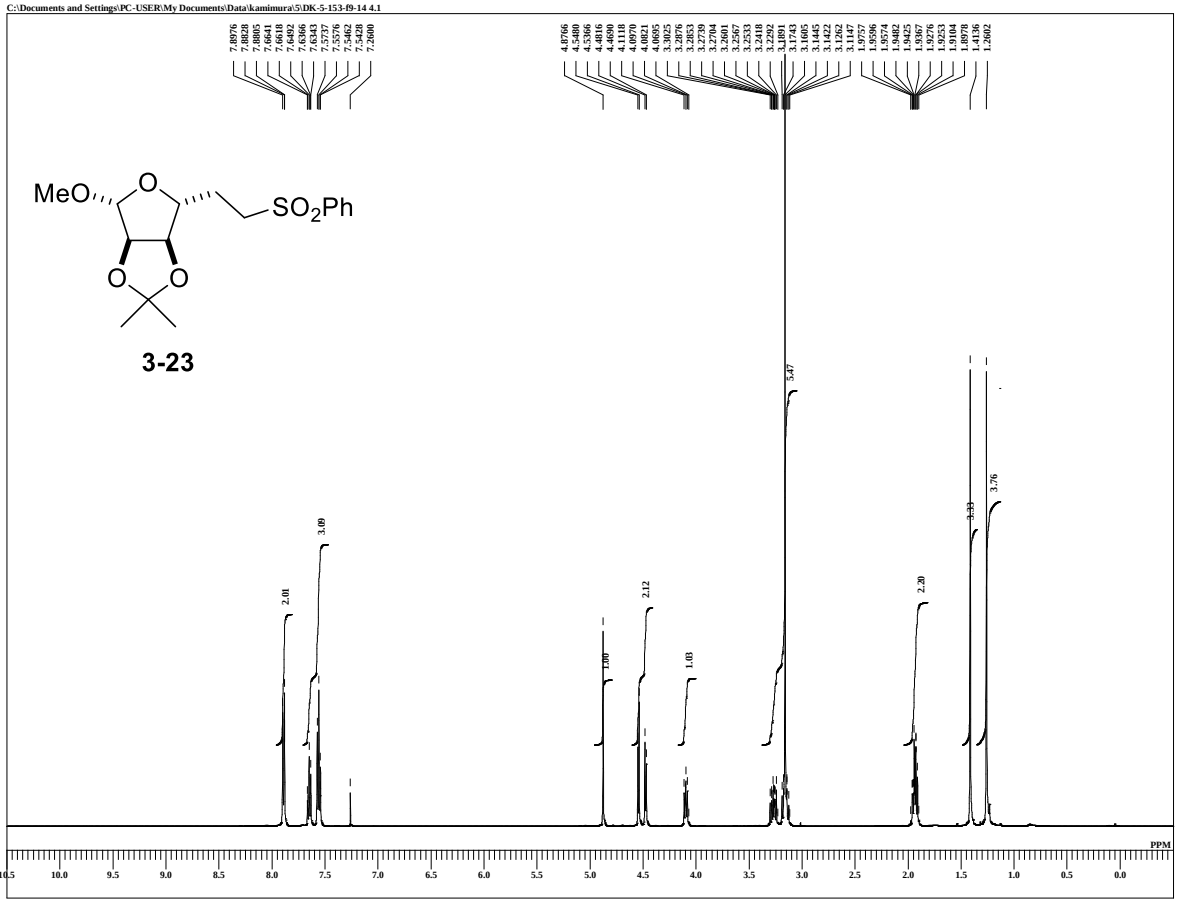


DFILE	DK-5-151-f12-25 13C-1.al
COMNT	single pulse decoupled gat
DATIM	30-10-2013 08:52:27
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	104856
SPO	104856
TIMES	200
DUMMY	4
FREQU	24999.62 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	200
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-151-f12-25 13C-1.al
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	25.7 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

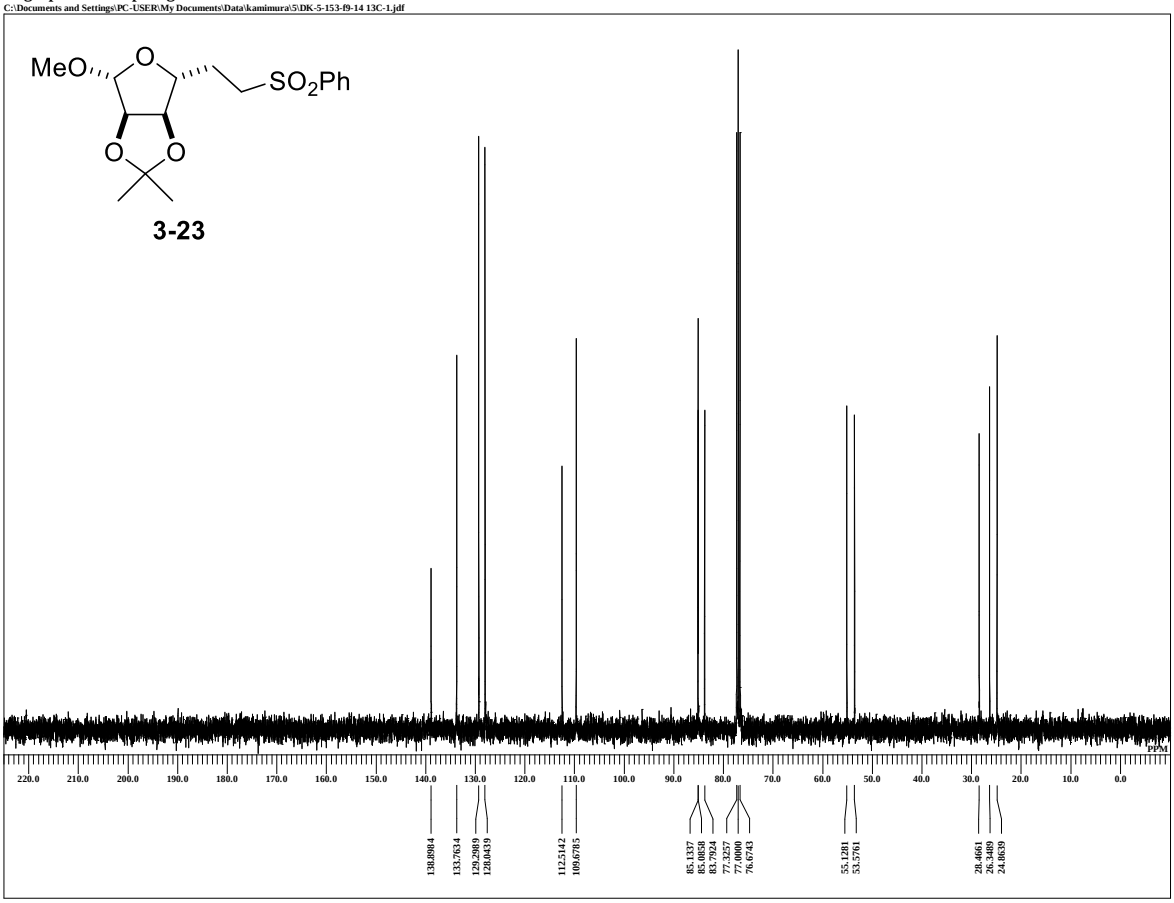
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-153-f9-14 4.1



DFILE	DK-5-153-f9-14 4.1
COMNT	single_pulse
DATIM	23-01-2014 13:46:18
MENUF	
OBNUC	1H
OFR	495.13 MHz
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
PW1	6.00 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	9286.78 Hz
FLT	38000 Hz
DELAY	13.16 usec
ACQTM	1.7642 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-153-f9-14 4.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-153-f9-14 13C-1.jdf

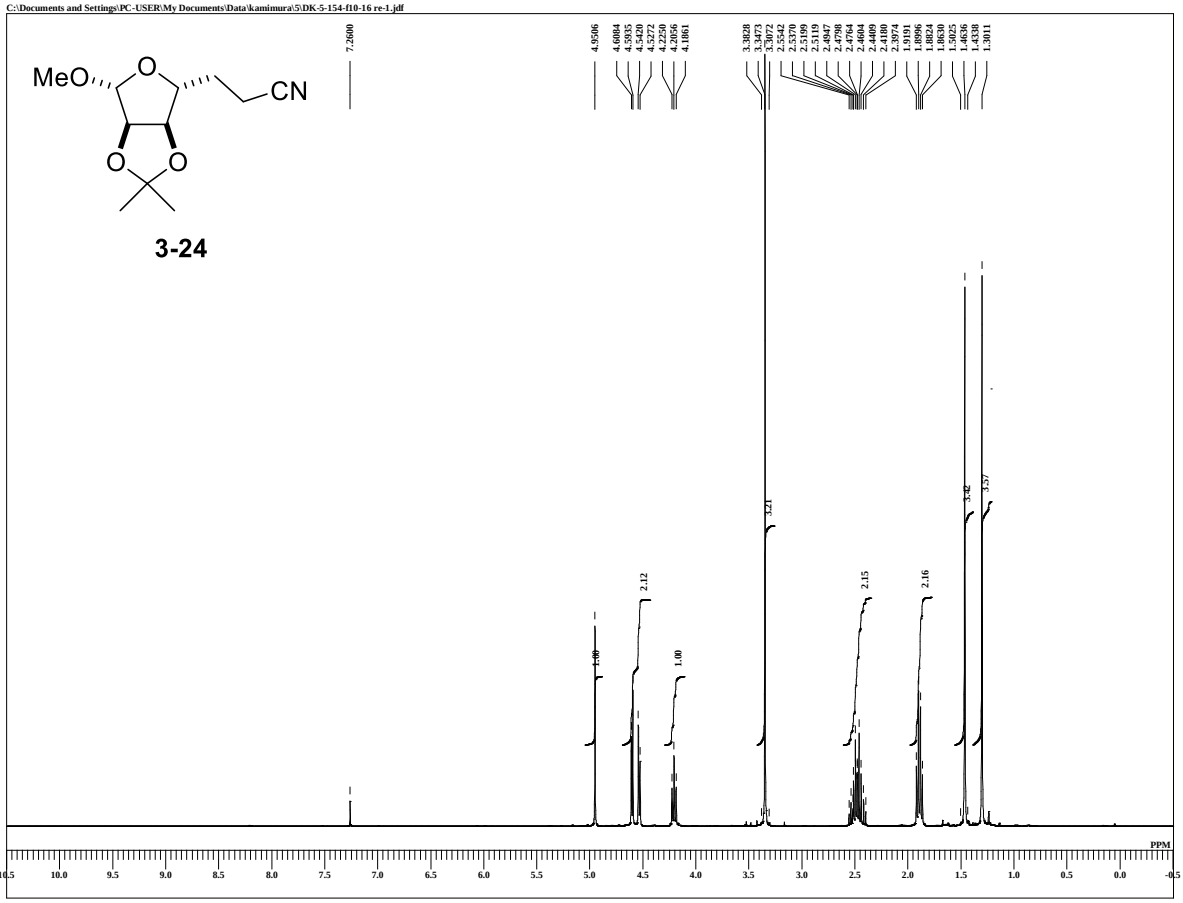


DFILE	DK-5-153-f9-14 13C-1.jdf
COMNT	single pulse decoupled gat
DATIM	31-10-2013 08:21:19
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	76
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	76
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-153-f9-14 13C-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

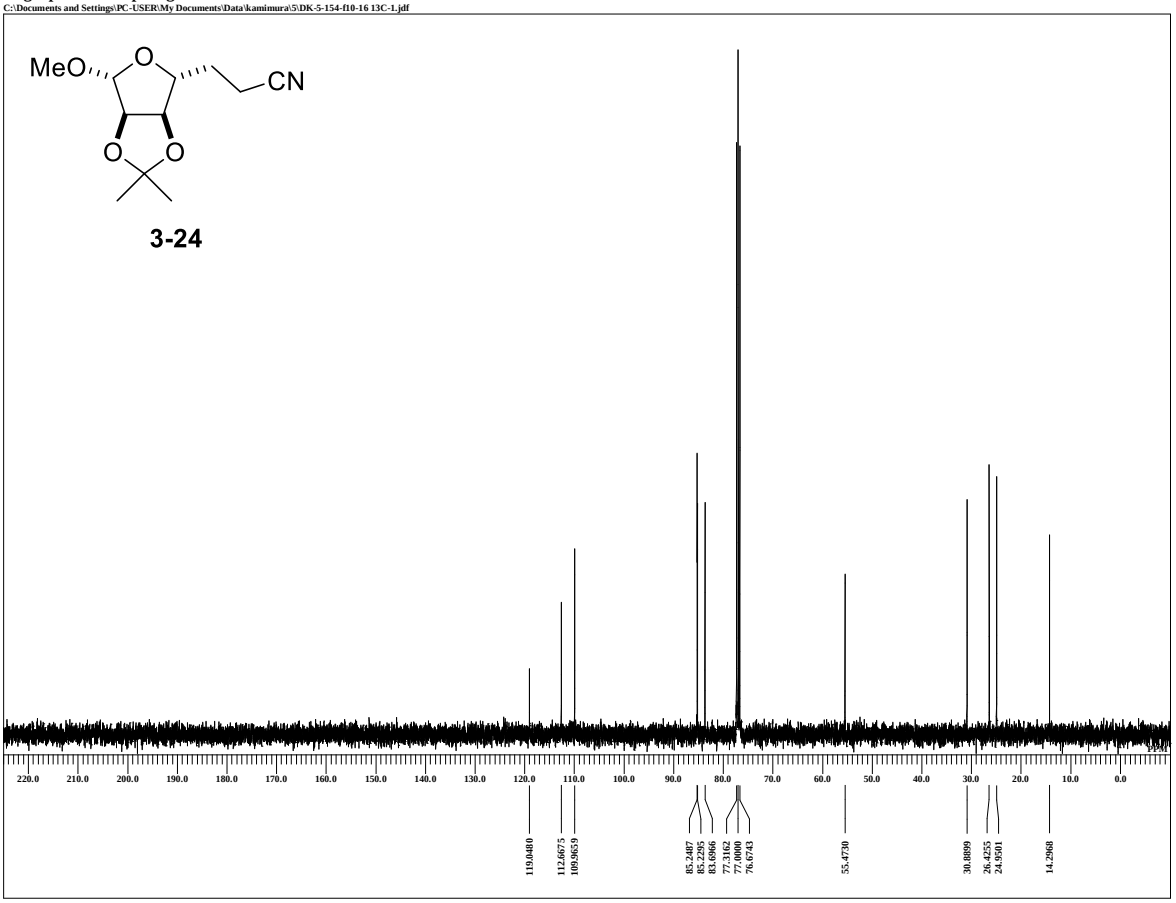
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-154-f10-16 re-1.jdf



DFILE DK-5-154-f10-16 re-1.jdf
COMNT single_pulse
DATIM 21-01-2014 14:26:11
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-154-f10-16 re-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-154-f10-16 13C-1.jdf

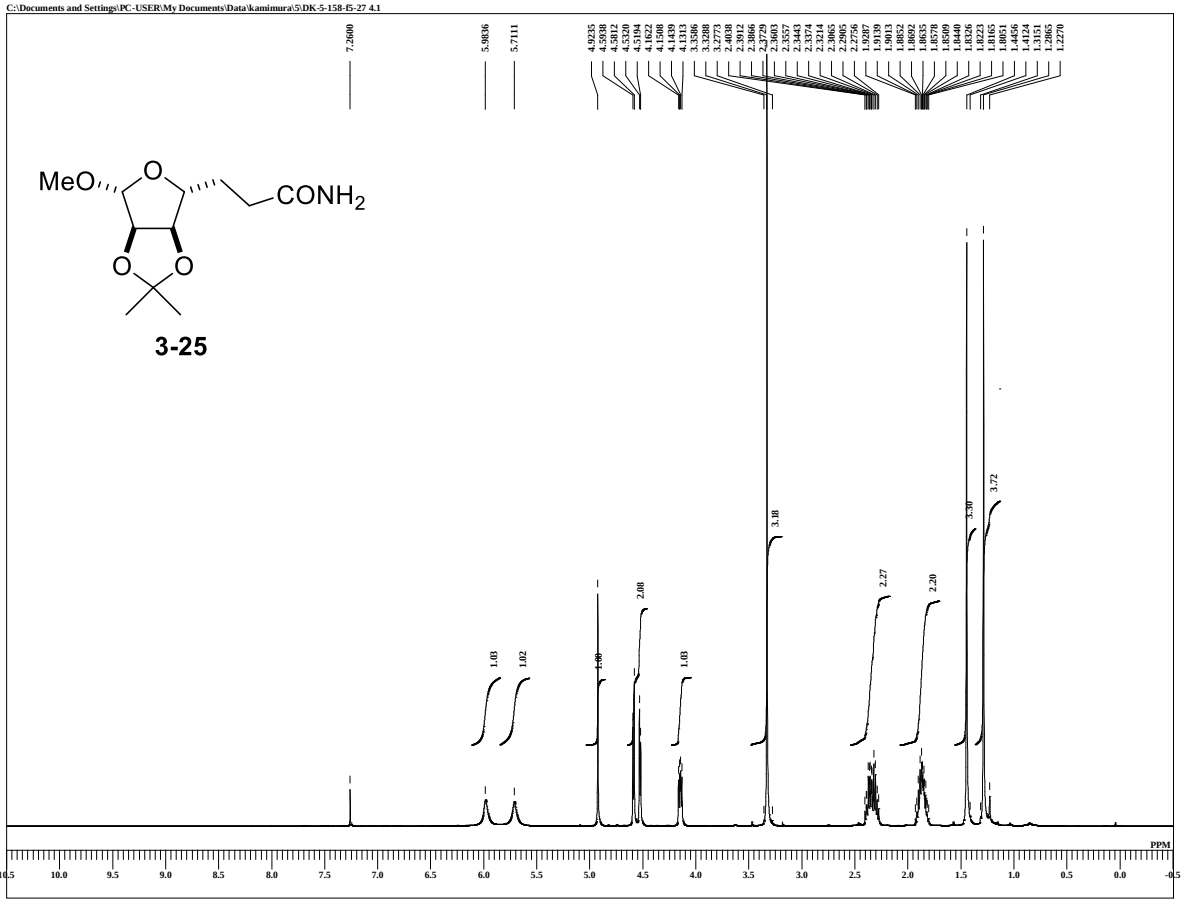


DFILE DK-5-154-f10-16 13C-1.jd
COMNT single pulse decoupled gat
DATIM 31-10-2013 08:49:08
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 93
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 93
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-154-f10-16 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.6 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

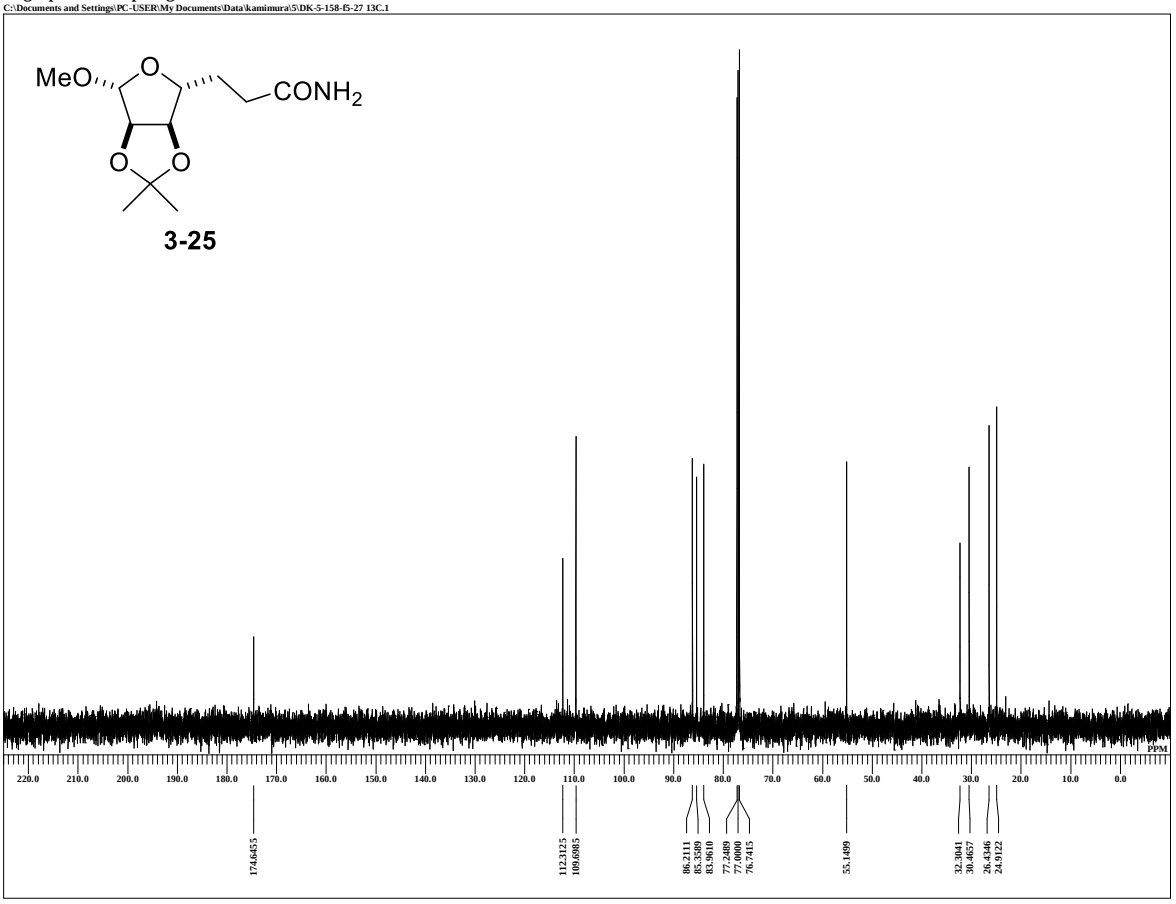
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-158-f5-27 4.1




```
DFILE    DK-5-158-f5-27 4.1
COMNT    single_pulse
DATIM    23-01-2014 13:50:00
MENUF
OBNUC    1H
OFR      495.13 MHz
OBFRQ    495.13 MHz
OBSET    4.38 KHz
OBFIN    9.64 Hz
PW1      6.00 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    9286.78 Hz
FLT      38000 Hz
DELAY    13.16 usec
ACQTM    1.7642 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    34
BF       0.10 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      495.13 MHz
IRSET    4.38 KHz
IRFIN    9.64 Hz
IRRPW    92 usec
IRATN    79
DFILE    DK-5-158-f5-27 4.1
SF
LKSET    748.40 KHz
LKFIN    98.2 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.8 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-158-f5-27 13C.1

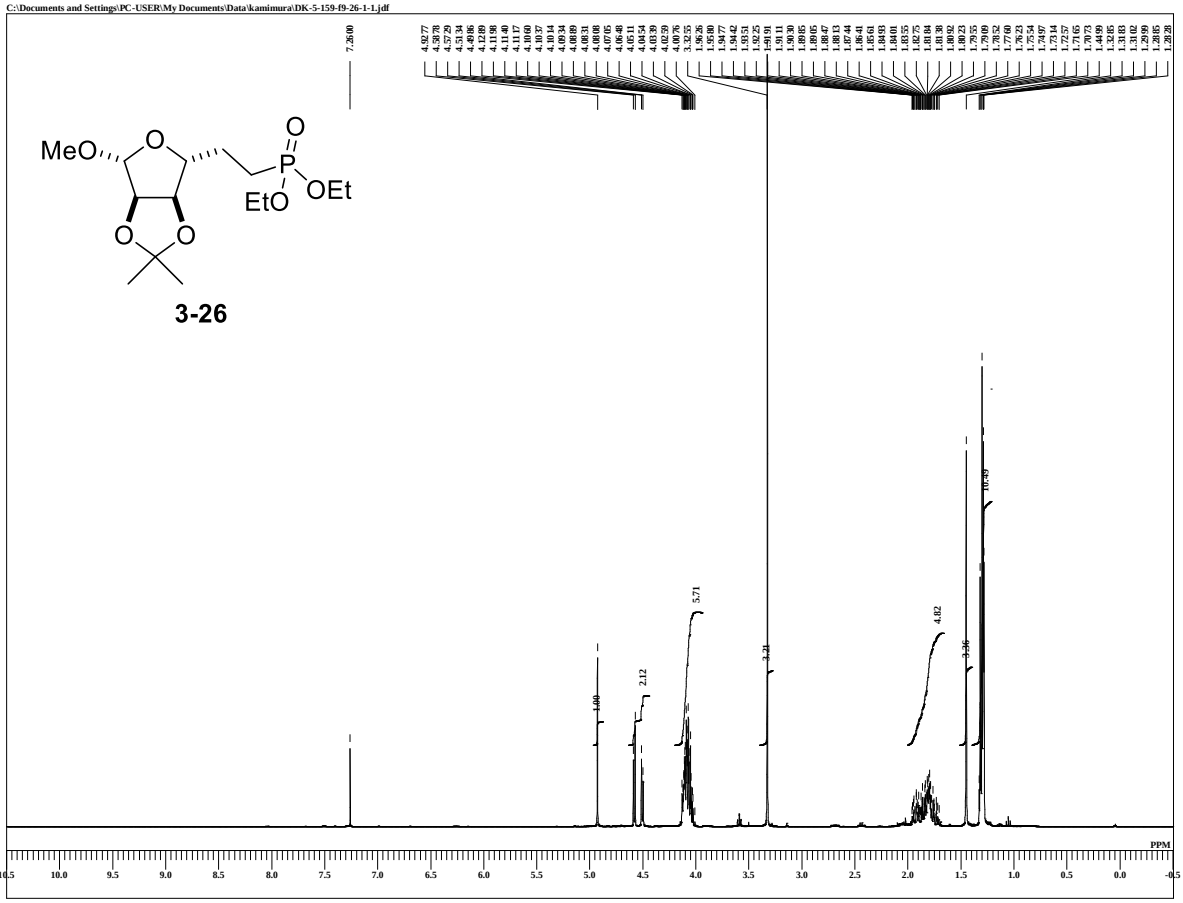



```
DFILE    DK-5-158-f5-27 13C.1
COMNT    single pulse decoupled gat
DATIM    01-11-2013 16:35:49
MENUF
OBNUC    13C
OFR      124.51 MHz
OBFRQ    124.51 MHz
OBSET    3.45 KHz
OBFIN    6.00 Hz
PW1      3.70 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    56
DUMMY    4
FREQU    39062.50 Hz
FLT      157000 Hz
DELAY    20.80 usec
ACQTM    0.8389 sec
PD       2.0000 sec
SCANS    56
ADBIT    16
RGAIN    46
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      495.13 MHz
IRSET    4.38 KHz
IRFIN    9.64 Hz
IRRPW    92 usec
IRATN    79
DFILE    DK-5-158-f5-27 13C.1
SF
LKSET    748.40 KHz
LKFIN    98.2 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    23.8 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

スペクトルデータ

single_pulse

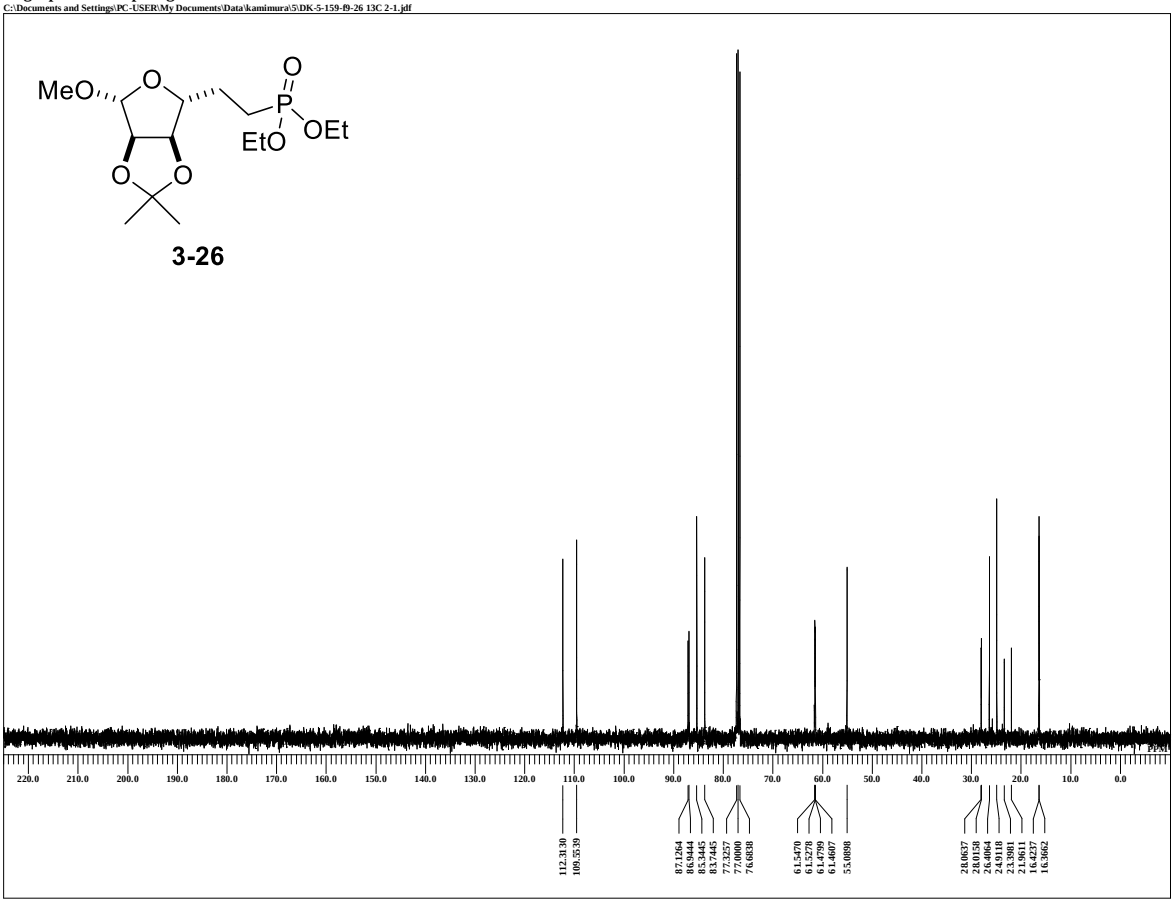
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-5-159-f9-26-1-1.jdf




```
DFILE   DK-5-159-f9-26-1-1.jdf
COMNT   single_pulse
DATIM   24-02-2014 15:29:22
MENUF
OBNUC   1H
OFR        395.88 MHz
OBFRQ      395.88 MHz
OBSET        6.28 KHz
OBFIN        0.87 Hz
PW1          6.44 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT       16384
SPO         16384
TIMES           8
DUMMY           1
FREQU     7422.80 Hz
FLT         30000 Hz
DELAY       16.68 usec
ACQTM        2.2073 sec
PD           2.0000 sec
SCANS           8
ADBIT          16
RGAIN          30
BF           0.10 Hz
T1           0.00
T2           0.00
T3         100.00
T4         100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW       115 usec
IRATN          79
DFILE   DK-5-159-f9-26-1-1.jdf
SF
LKSET       13.20 KHz
LKFIN        75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED           0 Hz
FILDC
FILDF
CTEMP        21.0 c
SLVNT   CDCL3
EXREF        7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-159-f9-26 13C 2-1.jdf

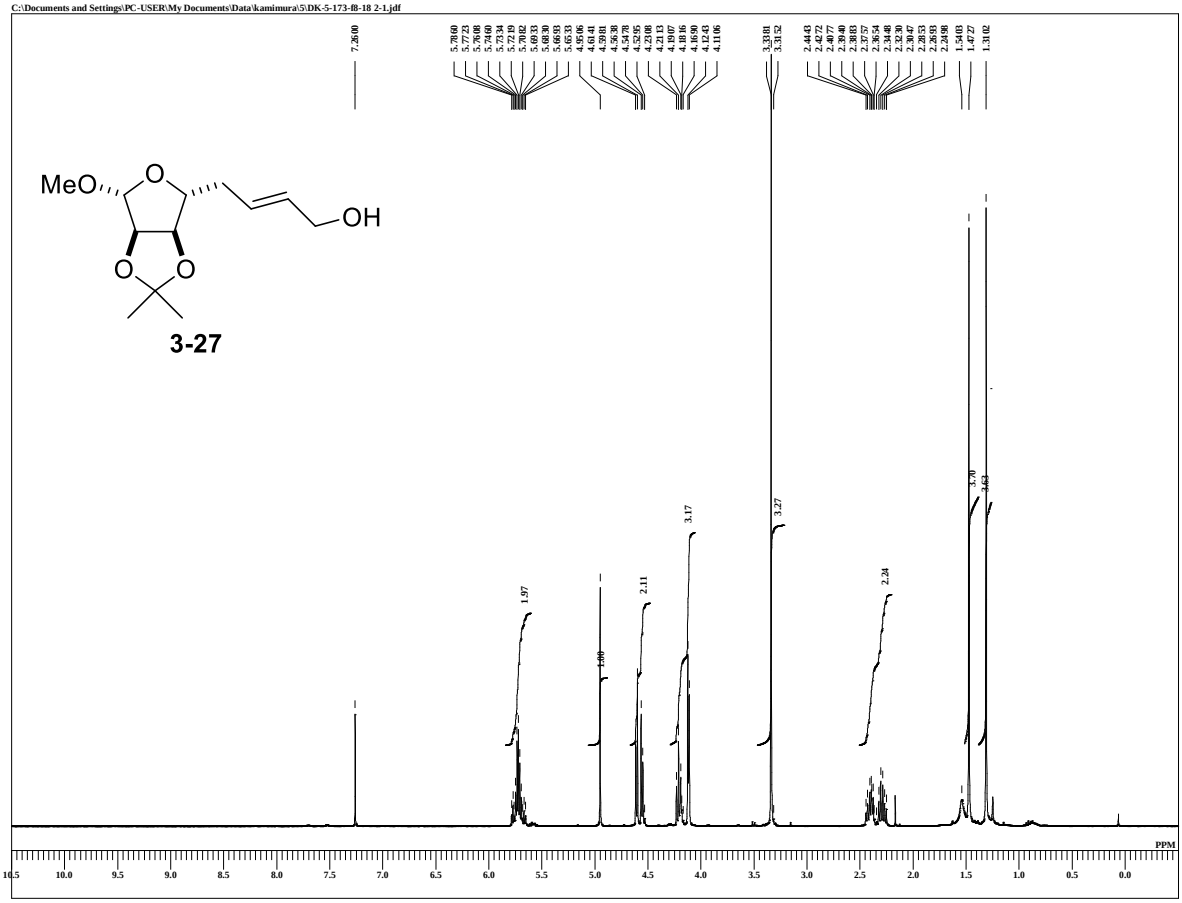



```
DFILE   DK-5-159-f9-26 13C 2-1.j
COMNT   single pulse decoupled gat
DATIM   23-01-2014 17:39:08
MENUF
OBNUC   13C
OFR        99.55 MHz
OBFRQ      99.55 MHz
OBSET       5.13 KHz
OBFIN       0.98 Hz
PW1         3.03 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT      32768
SPO        32768
TIMES        151
DUMMY          4
FREQU    31250.00 Hz
FLT        125000 Hz
DELAY      20.50 usec
ACQTM       1.0486 sec
PD          2.0000 sec
SCANS        151
ADBIT         16
RGAIN         60
BF          1.00 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW       115 usec
IRATN          79
DFILE   DK-5-159-f9-26 13C 2-1.j
SF
LKSET       13.20 KHz
LKFIN        75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED           0 Hz
FILDC
FILDF
CTEMP        21.7 c
SLVNT   CDCL3
EXREF        77.00 ppm
```

スペクトルデータ

single_pulse

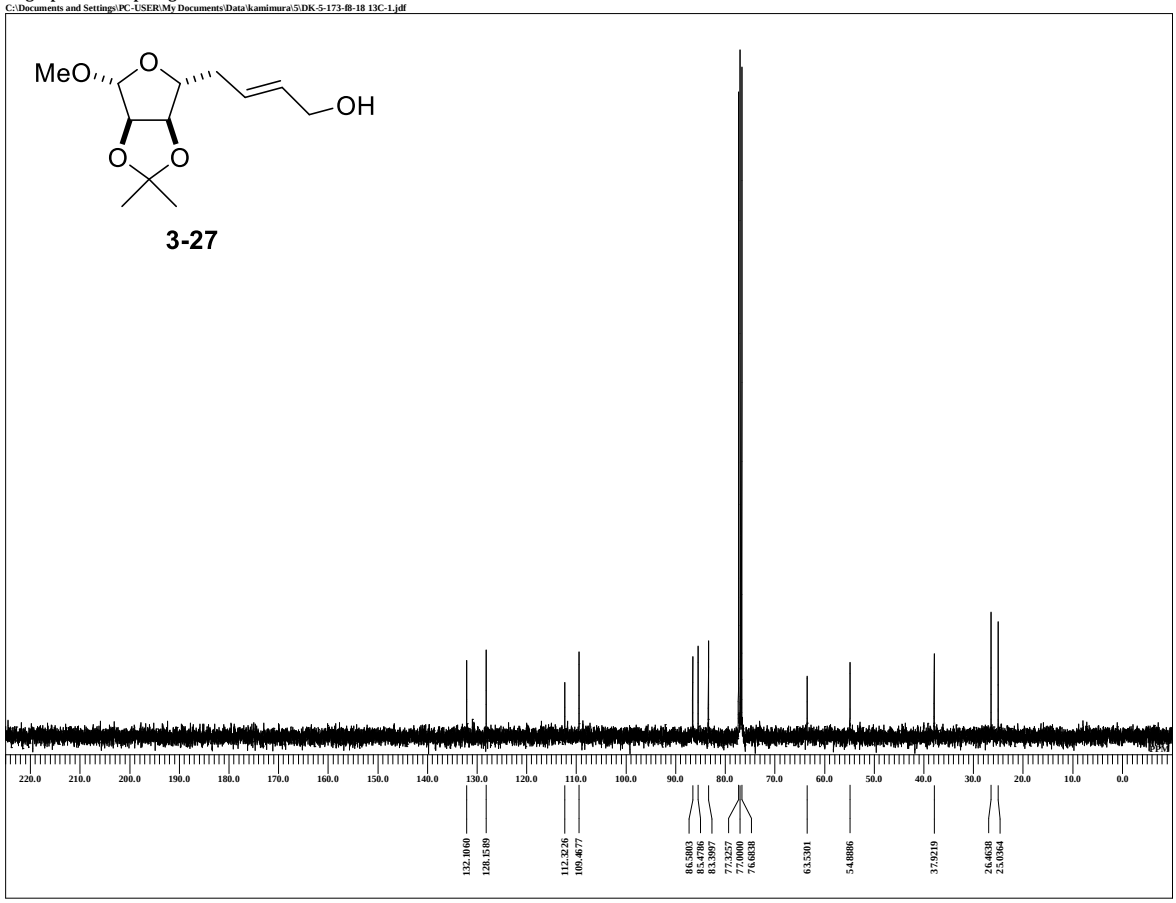
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-173-f8-18 2-1.jdf



DFILE DK-5-173-f8-18 2-1.jdf
COMNT single_pulse
DATIM 24-01-2014 17:14:37
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 38
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-5-173-f8-18 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-173-f8-18 13C-1.jdf

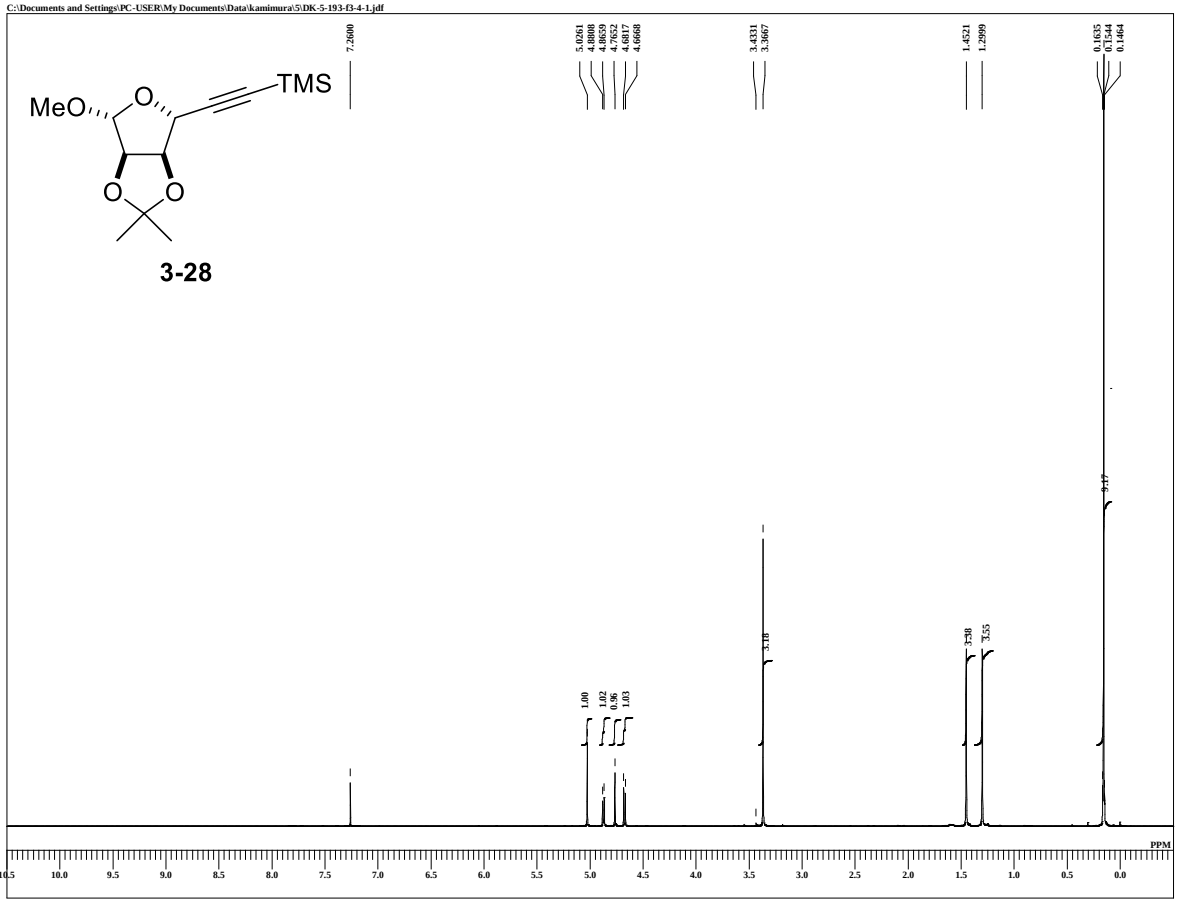


DFILE DK-5-173-f8-18 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 08-11-2013 16:19:56
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 116
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 116
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-173-f8-18 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

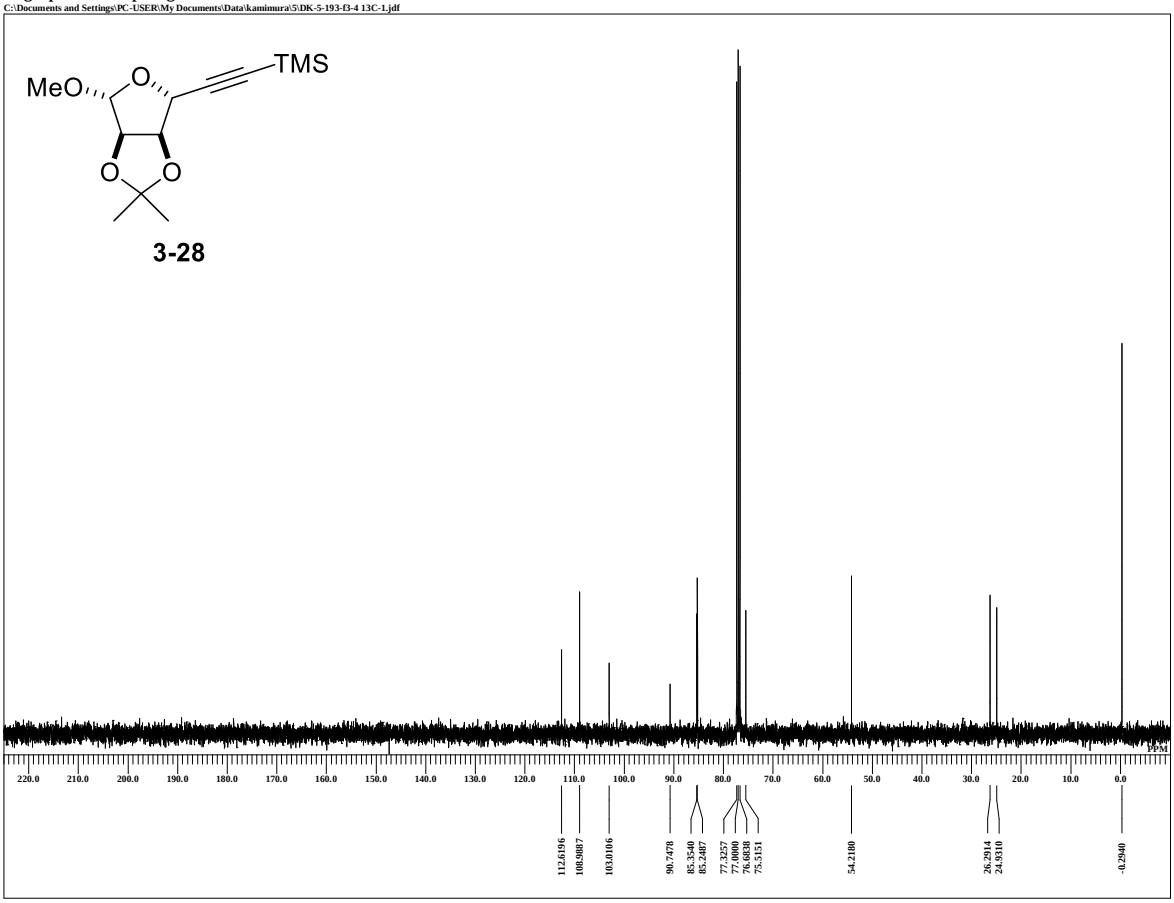
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-193-f3-4-1.jdf



DFILE	DK-5-193-f3-4-1.jdf
COMNT	single_pulse
DATIM	18-11-2013 15:48:17
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	38
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-193-f3-4-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	460.0 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-193-f3-4 13C-1.jdf

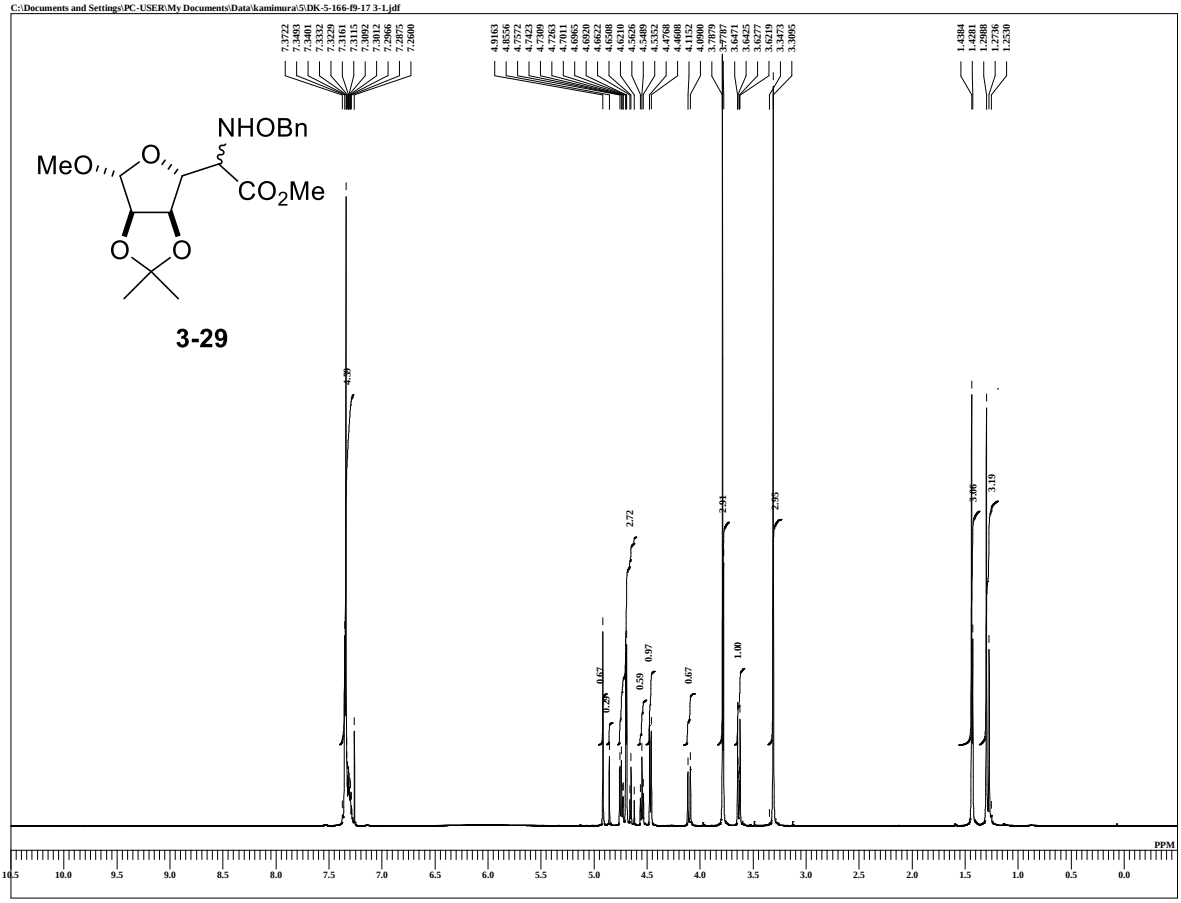


DFILE	DK-5-193-f3-4 13C-1.jdf
COMNT	single pulse decoupled gat
DATIM	18-11-2013 15:54:00
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	101
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	101
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-193-f3-4 13C-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	460.0 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

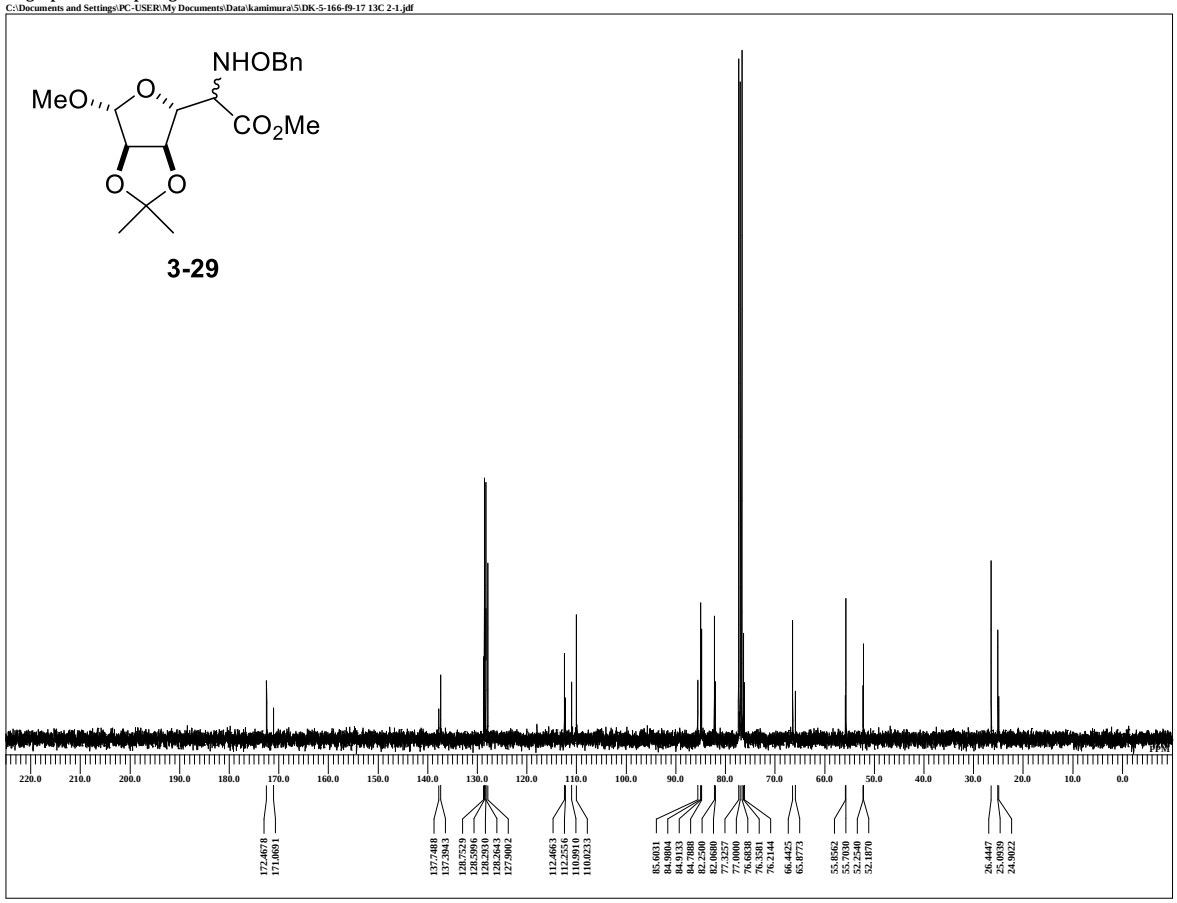
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-166-f9-17 3-1.jdf




```
DFILE   DK-5-166-f9-17 3-1.jdf
COMNT   single_pulse
DATIM   24-01-2014 15:03:32
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.44 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       16384
SPO         16384
TIMES           8
DUMMY           1
FREQU    7422.80 Hz
FLT        30000 Hz
DELAY      16.68 usec
ACQTM      2.2073 sec
PD         2.0000 sec
SCANS           8
ADBIT          16
RGAIN          34
BF          0.10 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW        115 usec
IRATN           79
DFILE   DK-5-166-f9-17 3-1.jdf
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED        0 Hz
FILDC
FILDF
CTEMP       21.2 c
SLVNT   CDCL3
EXREF       7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-166-f9-17 13C 2-1.jdf

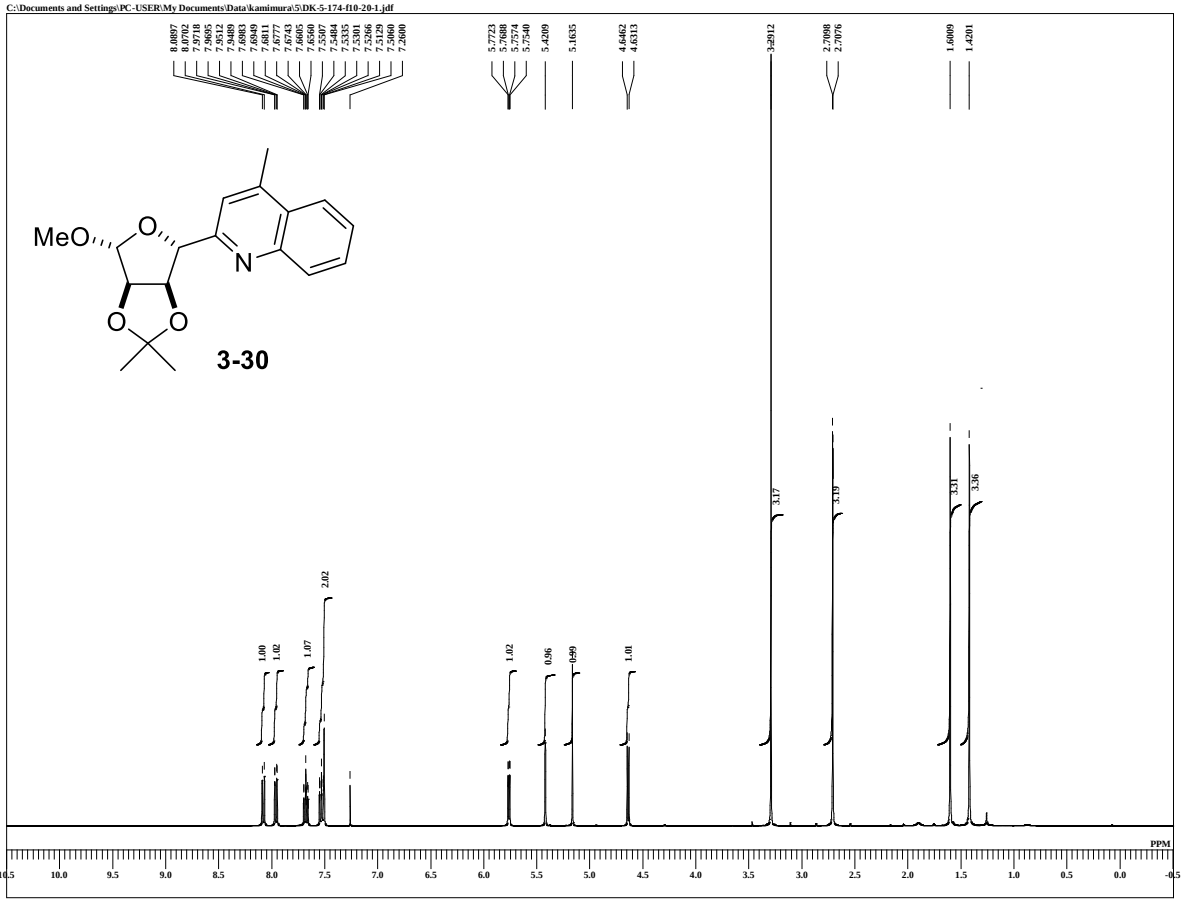



```
DFILE   DK-5-166-f9-17 13C 2-1.j
COMNT   single pulse decoupled gat
DATIM   24-01-2014 17:01:56
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET       5.13 KHz
OBFIN       0.98 Hz
PW1         3.03 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       32768
SPO         32768
TIMES         201
DUMMY           4
FREQU   31250.00 Hz
FLT       125000 Hz
DELAY      20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS         201
ADBIT          16
RGAIN          60
BF          1.00 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW        115 usec
IRATN           79
DFILE   DK-5-166-f9-17 13C 2-1.j
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV           0
LGAIN           0
LKPHS           0
LKSIG           0
CSPED        0 Hz
FILDC
FILDF
CTEMP       21.5 c
SLVNT   CDCL3
EXREF       77.00 ppm
```

スペクトルデータ

single_pulse

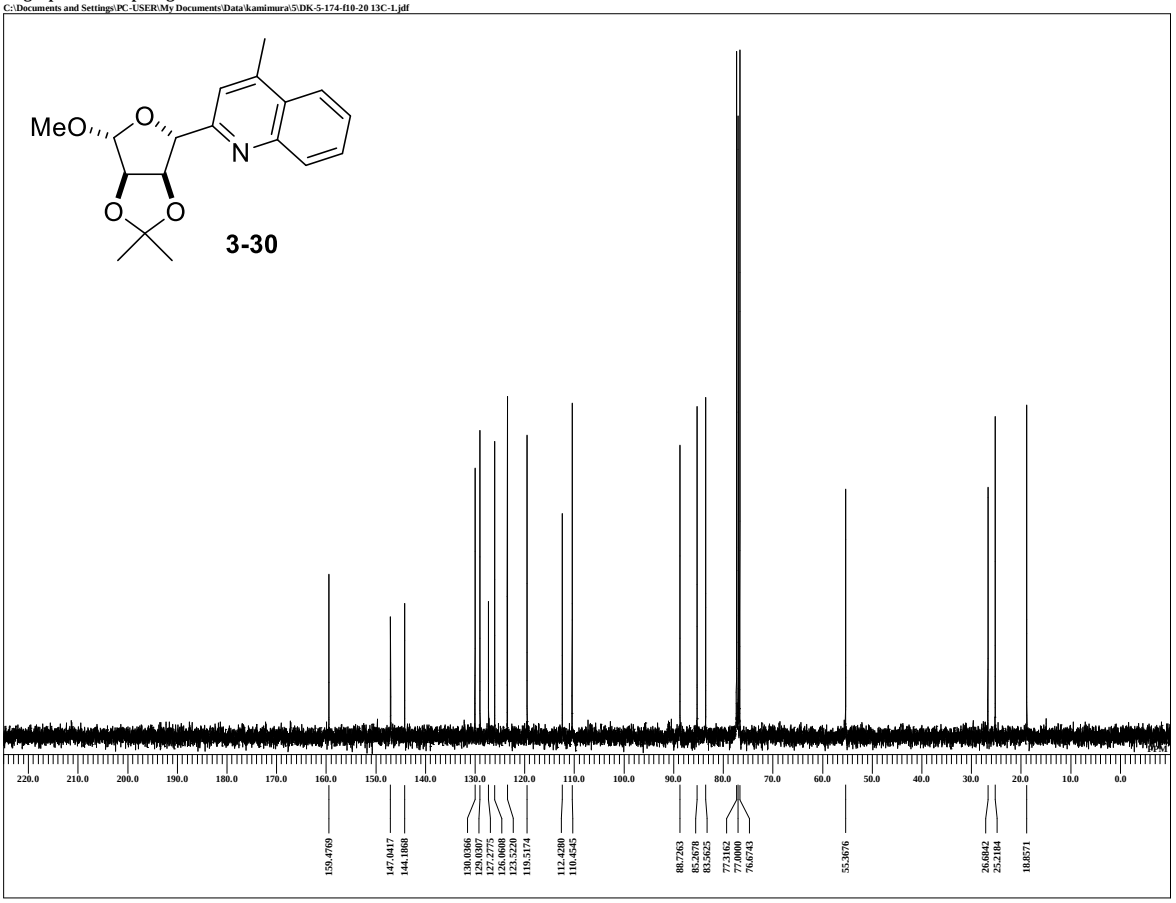
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-174-f10-20-1.jdf



DFILE DK-5-174-f10-20-1.jdf
COMNT single_pulse
DATIM 08-11-2013 12:43:46
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-174-f10-20-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.3 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-174-f10-20 13C-1.jdf

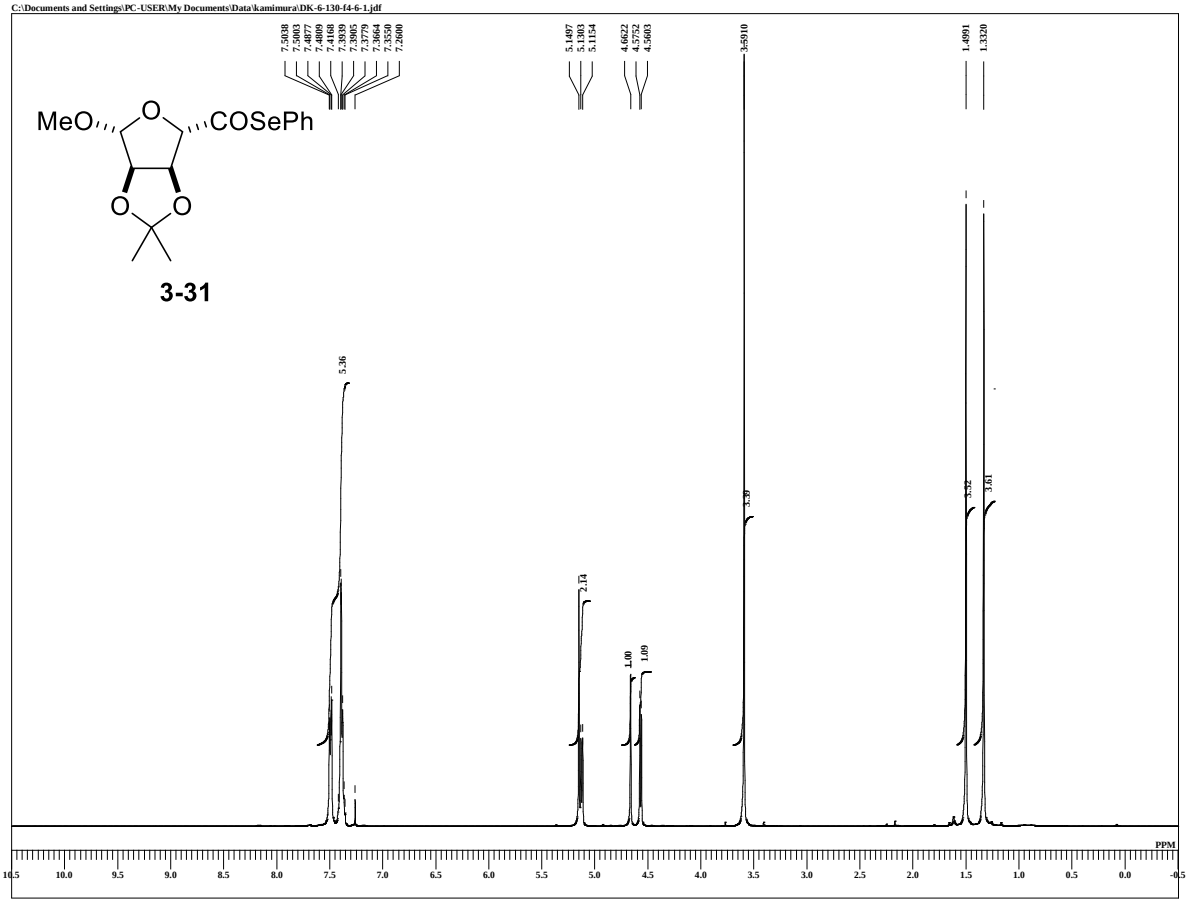


DFILE DK-5-174-f10-20 13C-1.jd
COMNT single pulse decoupled gat
DATIM 08-11-2013 12:49:03
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 93
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 93
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-174-f10-20 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

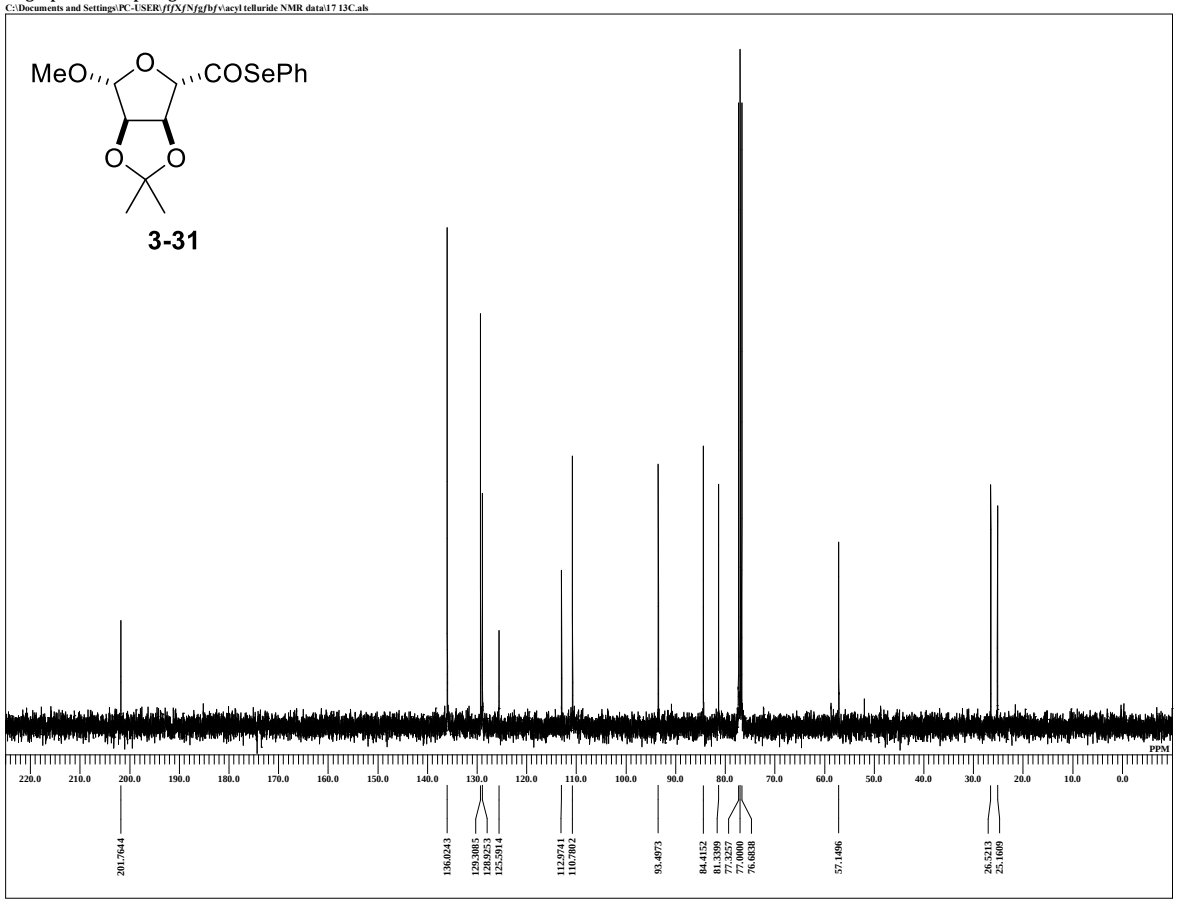
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-130-f4-6-1.jdf



DFILE	DK-6-130-f4-6-1.jdf
COMNT	single_pulse
DATIM	05-03-2014 19:04:08
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-130-f4-6-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER*ttfXfNfgfbf*\acyl telluride NMR data\17 13C.als

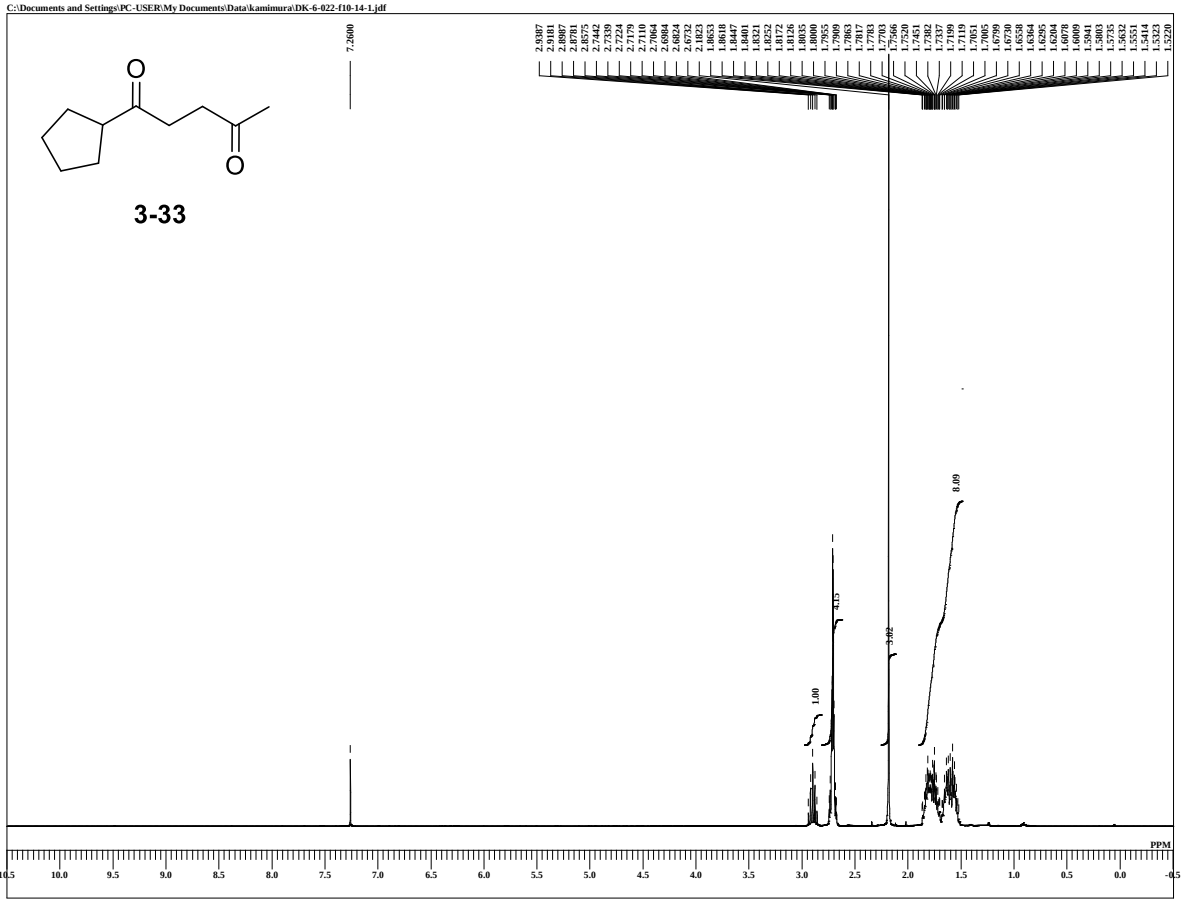


DFILE	17 13C.als
COMNT	single pulse decoupled gat
DATIM	02-11-2013 18:00:18
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	26214
SPO	26214
TIMES	77
DUMMY	4
FREQU	24999.62 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	77
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	17 13C.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	26.7 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-022-f10-14-1.jdf

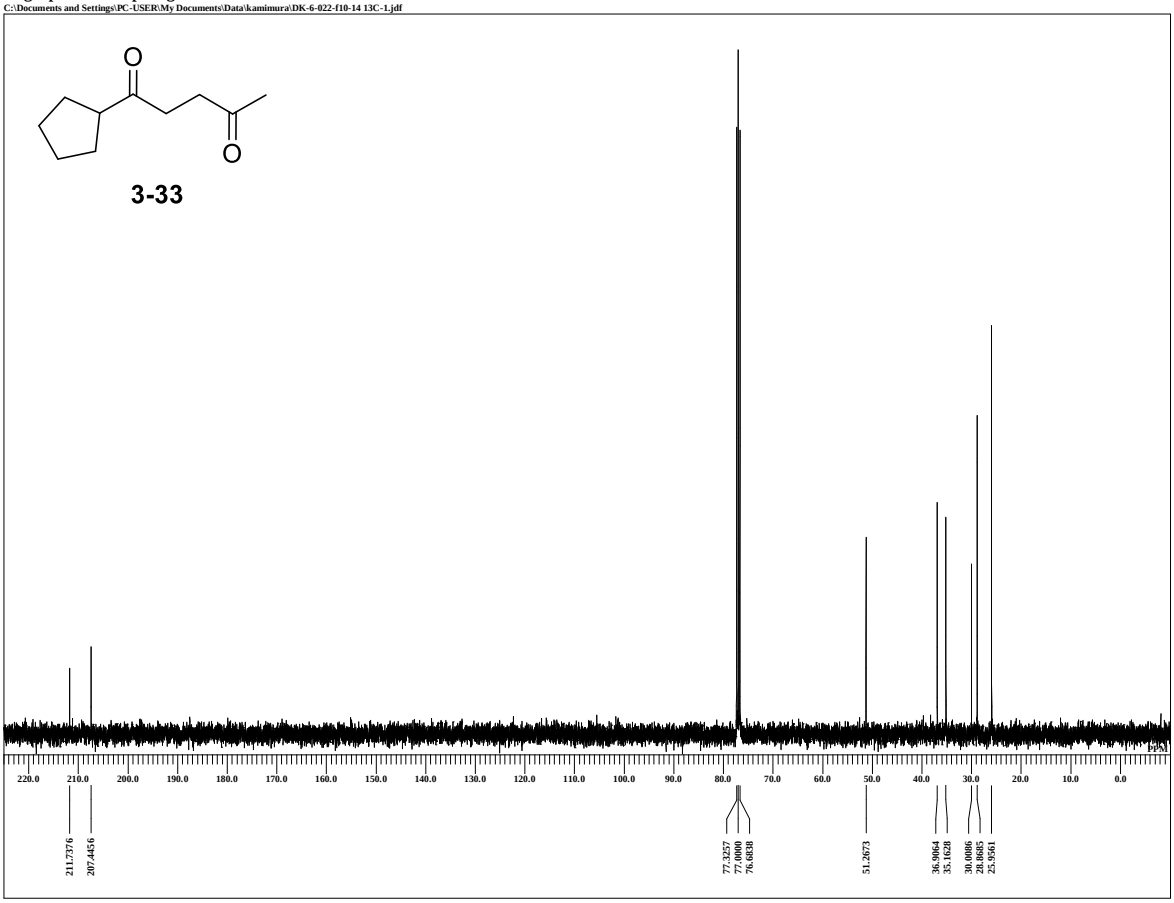


3-33

DFILE	DK-6-022-f10-14-1.jdf
COMNT	single_pulse
DATIM	10-02-2014 11:24:42
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	36
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-022-f10-14-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-022-f10-14 13C-1.jdf



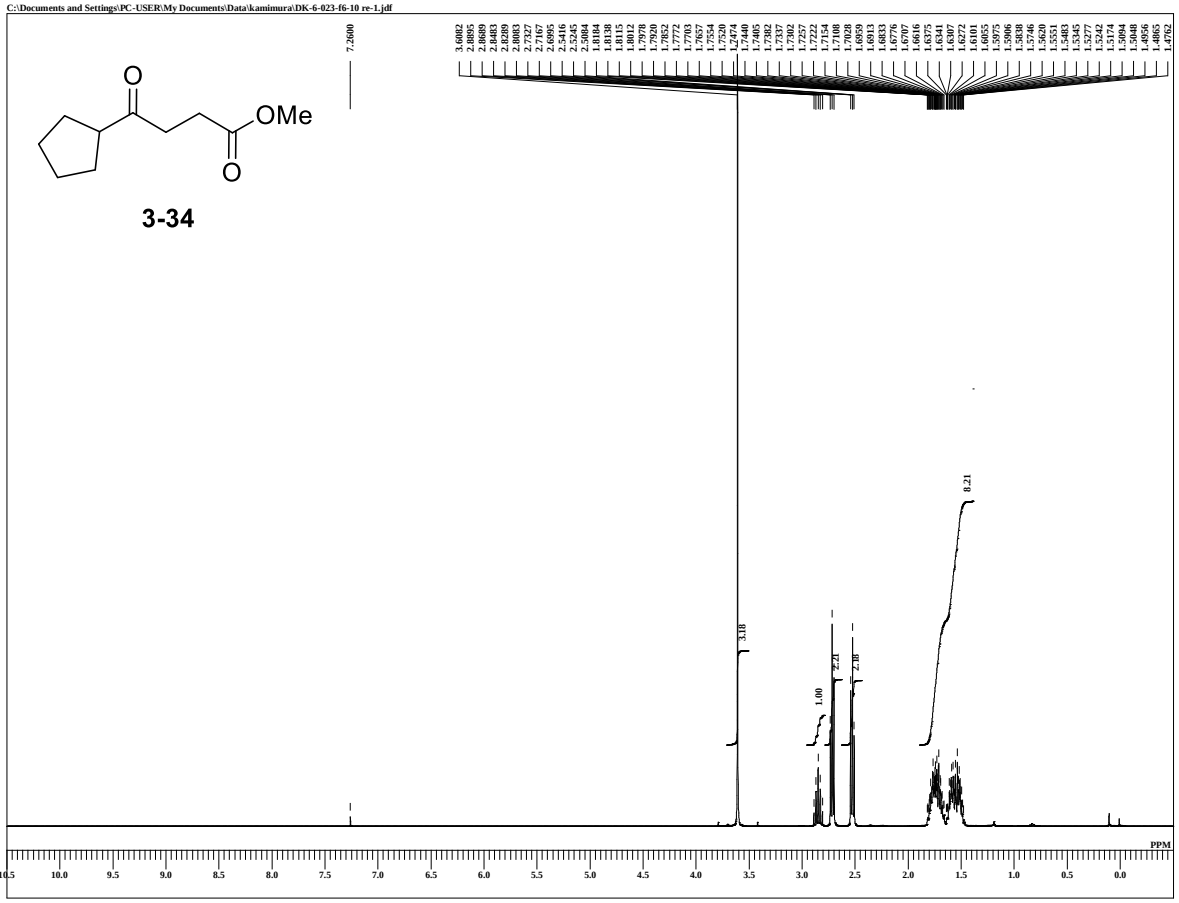
3-33

DFILE	DK-6-022-f10-14 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	10-02-2014 11:30:30
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	101
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	101
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-022-f10-14 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

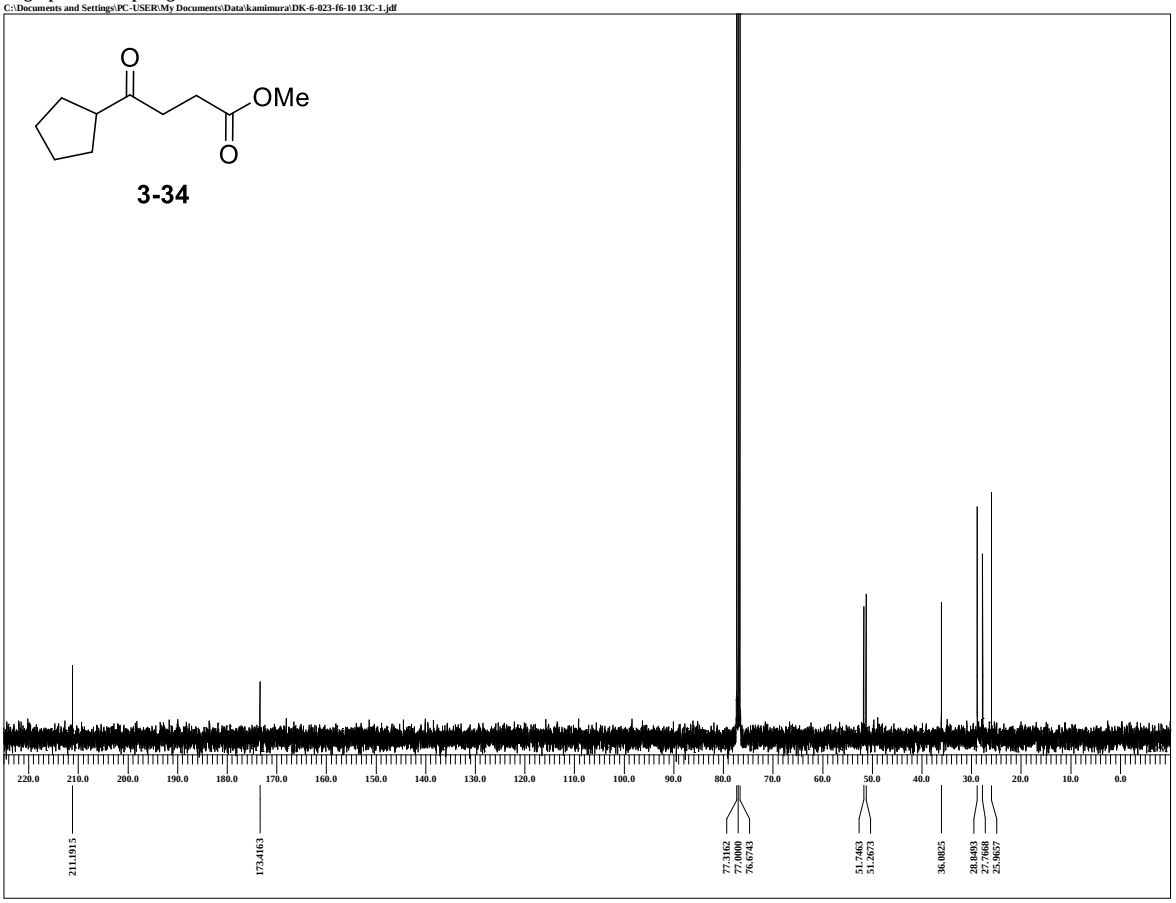
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-023-f6-10 re-1.jdf



DFILE DK-6-023-f6-10 re-1.jdf
COMNT single_pulse
DATIM 17-03-2014 09:26:13
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 20
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-023-f6-10 re-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.8 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-023-f6-10 13C-1.jdf

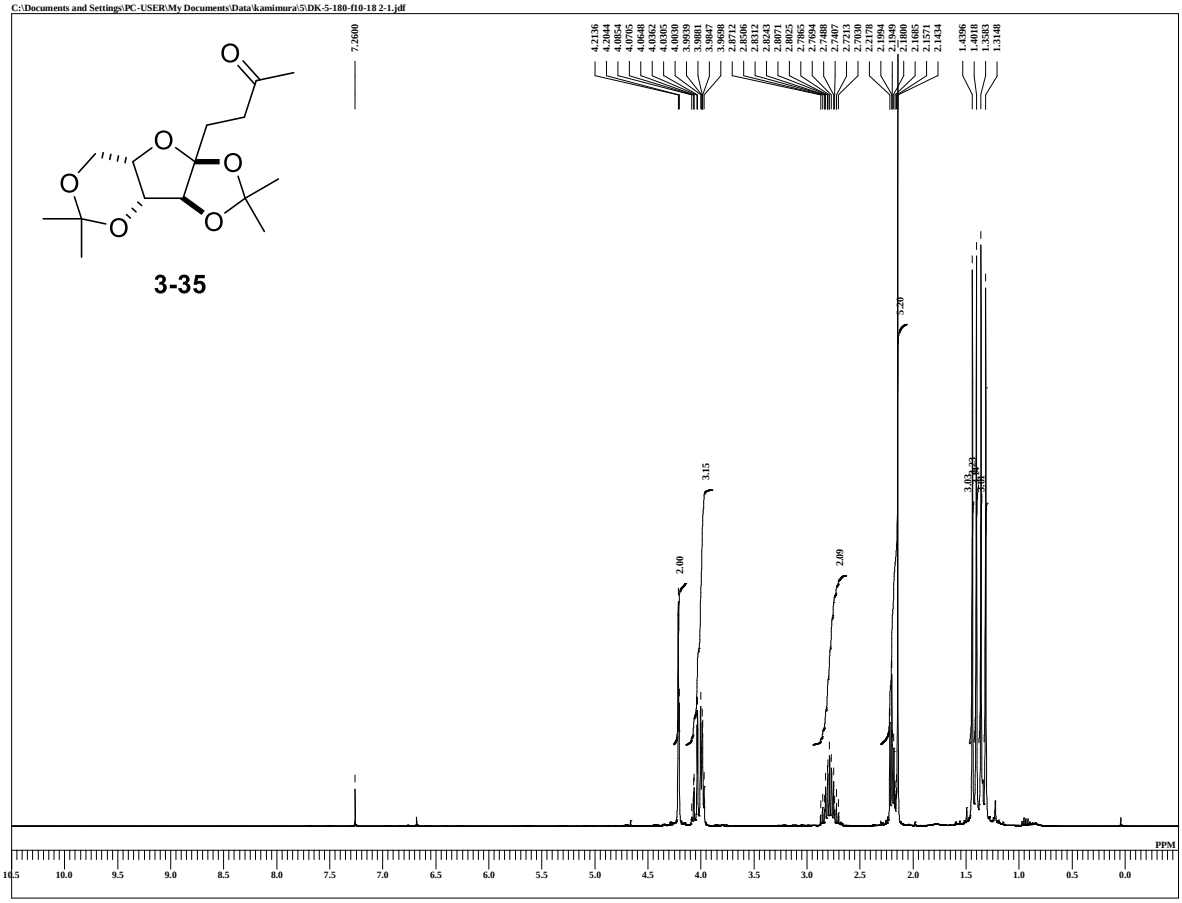


DFILE DK-6-023-f6-10 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 14-03-2014 18:07:40
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 151
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 151
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-023-f6-10 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

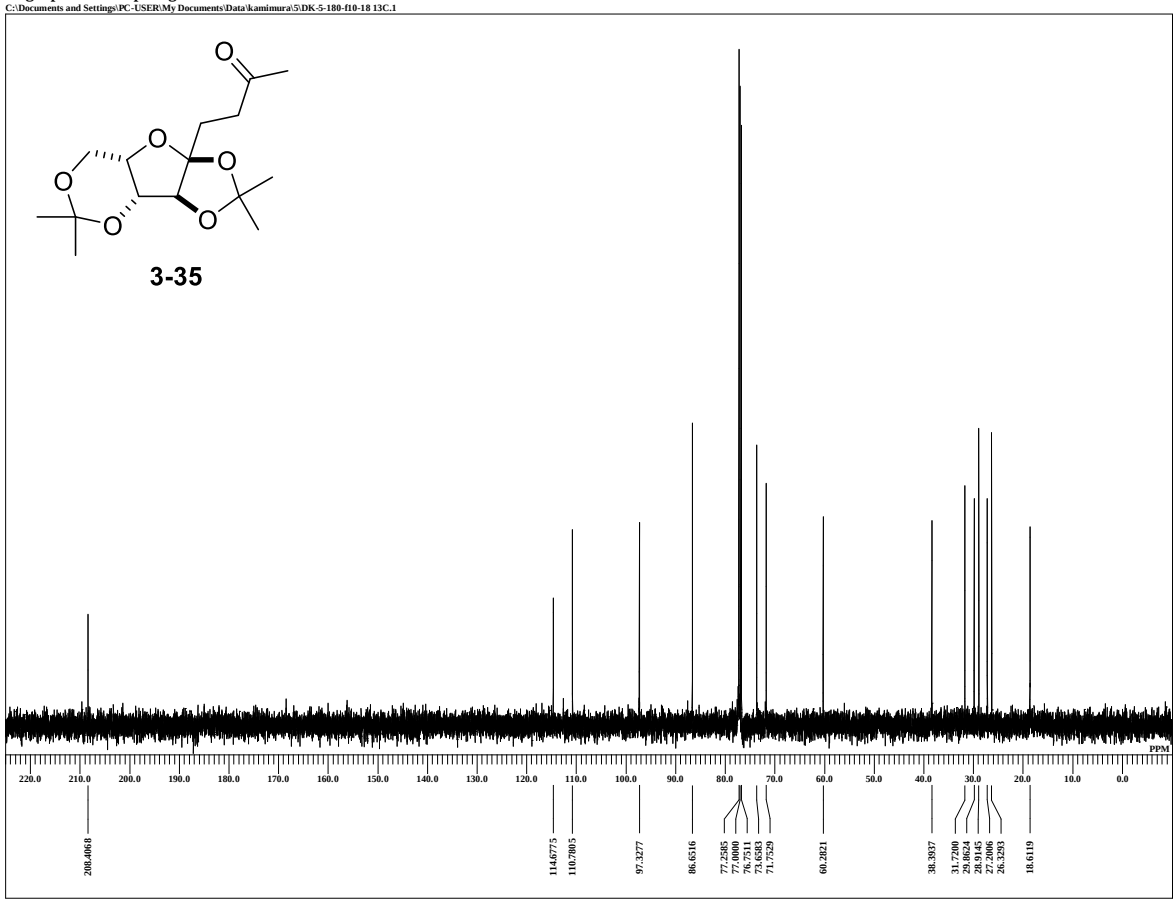
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-180-f10-18 2-1.jdf



DFILE	DK-5-180-f10-18 2-1.jdf
COMNT	single_pulse
DATIM	28-01-2014 09:27:35
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	26
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-180-f10-18 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-180-f10-18 13C.1

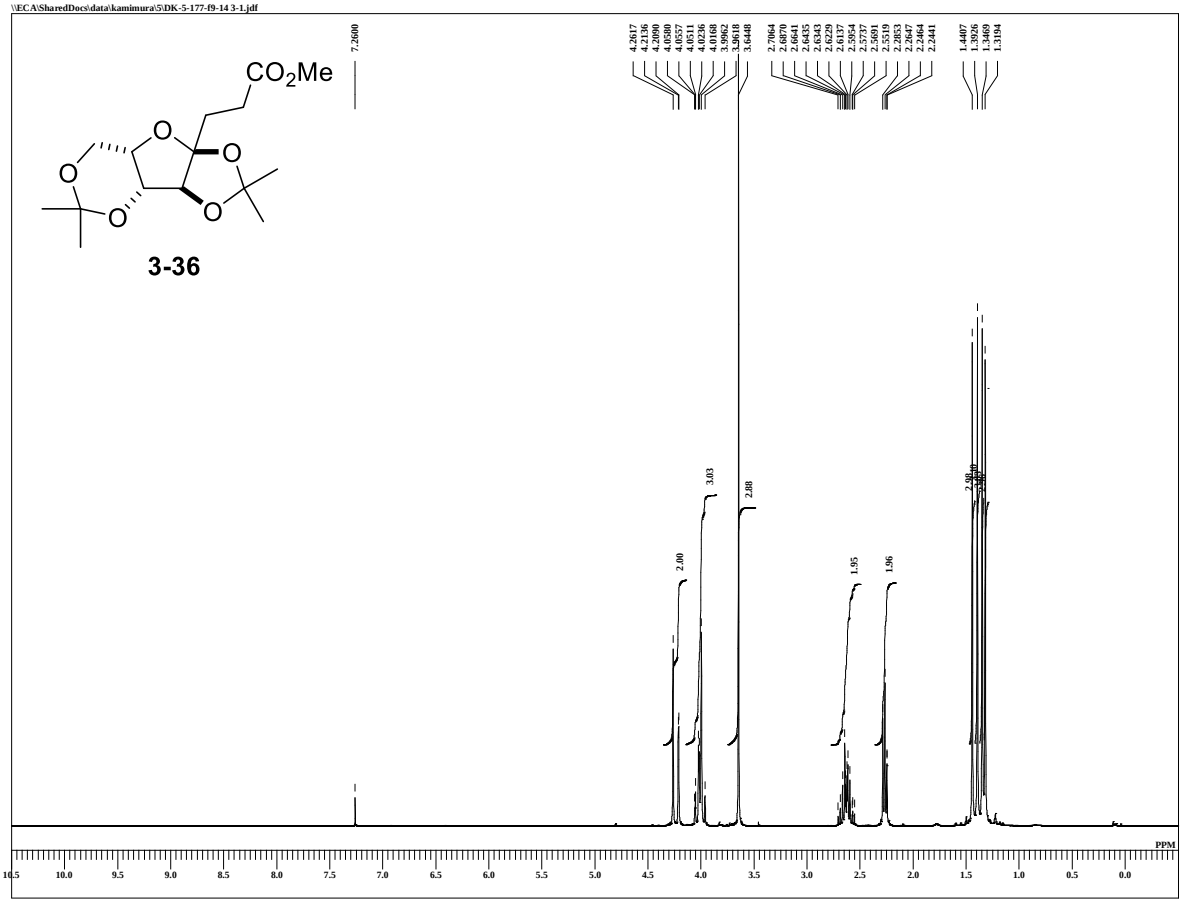


DFILE	DK-5-180-f10-18 13C.1
COMNT	single pulse decoupled gat
DATIM	11-11-2013 09:14:49
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	91
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	91
ADBIT	16
RGAIN	48
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-180-f10-18 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

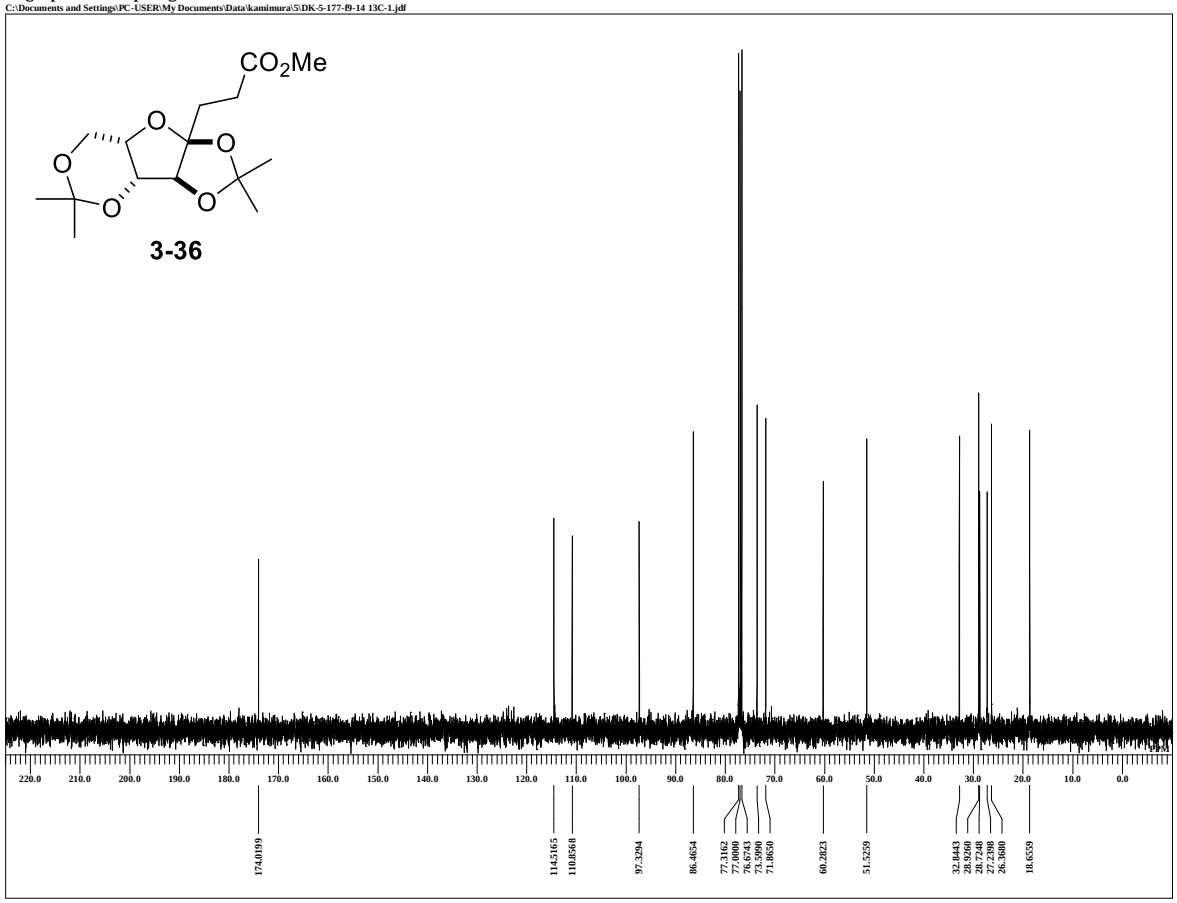
\\ECA\SharedDocs\data\kamimura\5\DK-5-177-f9-14 3-1.jdf



DFILE DK-5-177-f9-14 3-1.jdf
COMNT single_pulse
DATIM 28-01-2014 09:15:22
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 24
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-177-f9-14 3-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-177-f9-14 13C-1.jdf

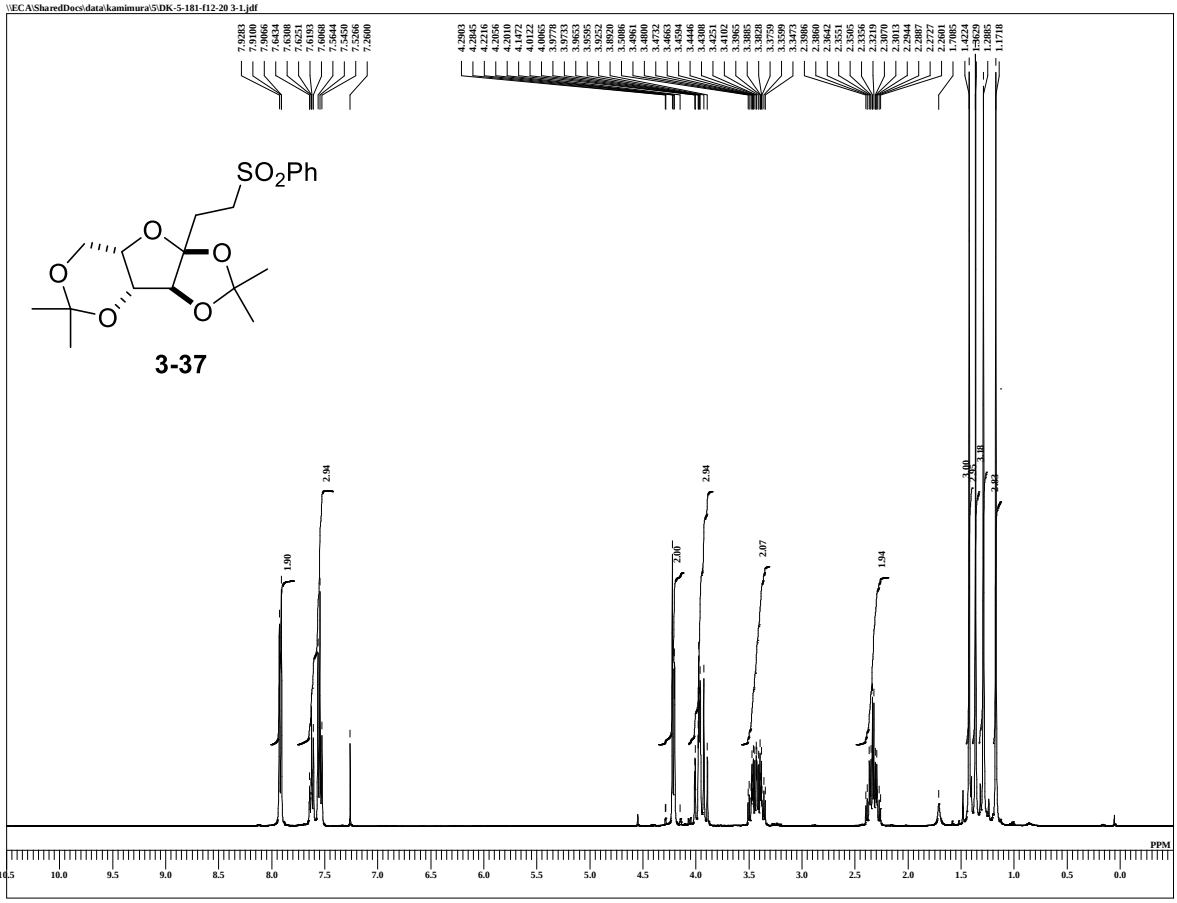


DFILE DK-5-177-f9-14 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 10-11-2013 09:23:15
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 78
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 78
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-177-f9-14 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

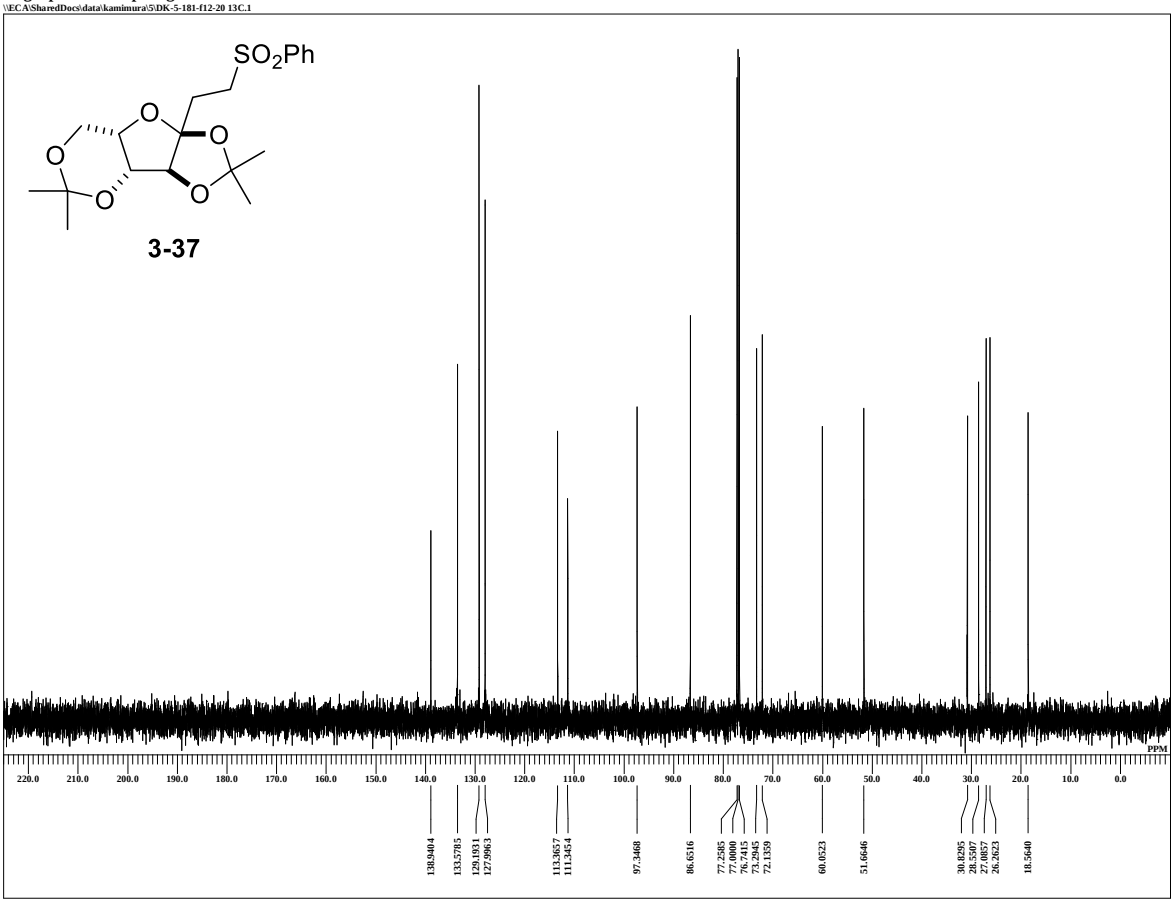
\\ECA\SharedDocs\data\kamimura\5\DK-5-181-f12-20 3-1.jdf



DFILE	DK-5-181-f12-20 3-1.jdf
COMNT	single_pulse
DATIM	11-11-2013 14:50:43
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-181-f12-20 3-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\5\DK-5-181-f12-20 13C.1

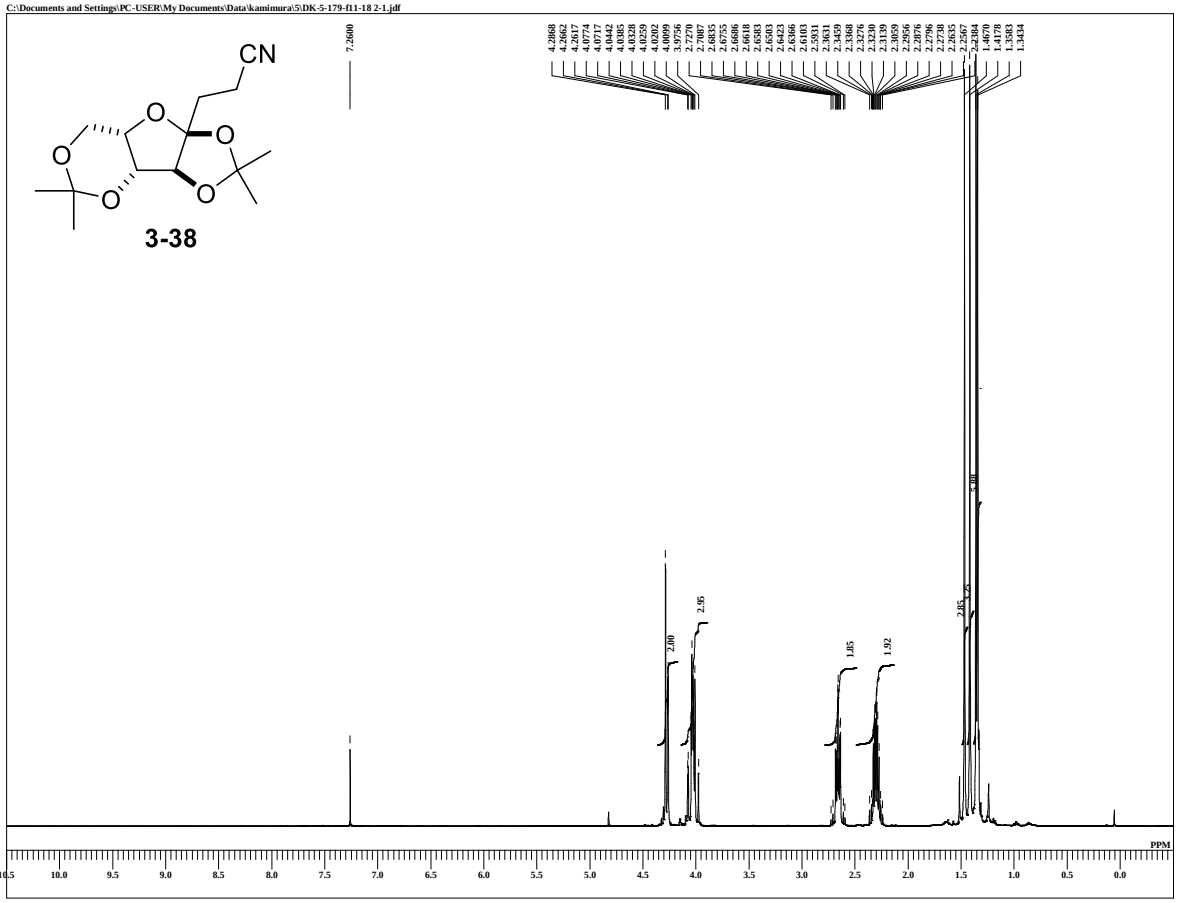


DFILE	DK-5-181-f12-20 13C.1
COMNT	single pulse decoupled gat
DATIM	11-11-2013 09:22:07
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	52
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	52
ADBIT	16
RGAIN	46
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-181-f12-20 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

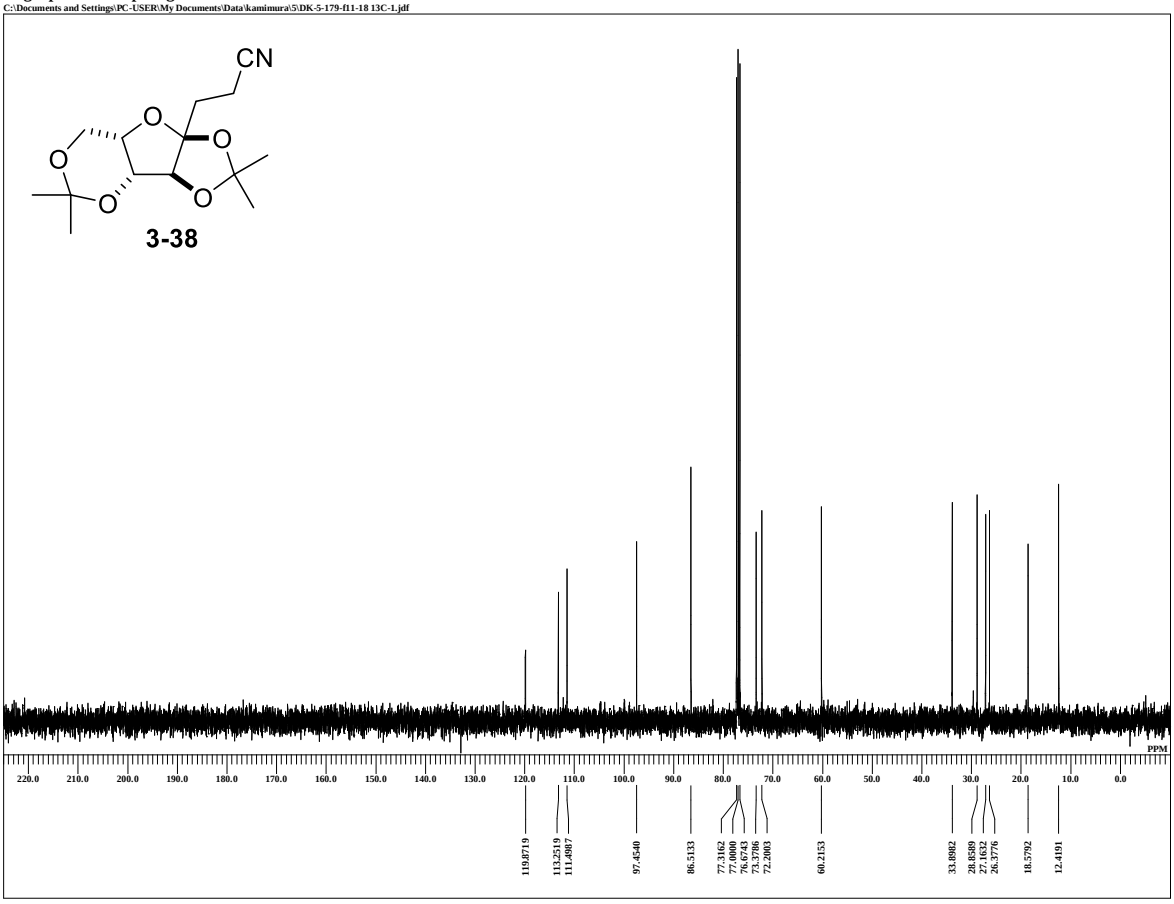
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-179-f11-18 2-1.jdf



DFILE	DK-5-179-f11-18 2-1.jdf
COMNT	single_pulse
DATIM	29-01-2014 11:18:58
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-179-f11-18 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-179-f11-18 13C-1.jdf

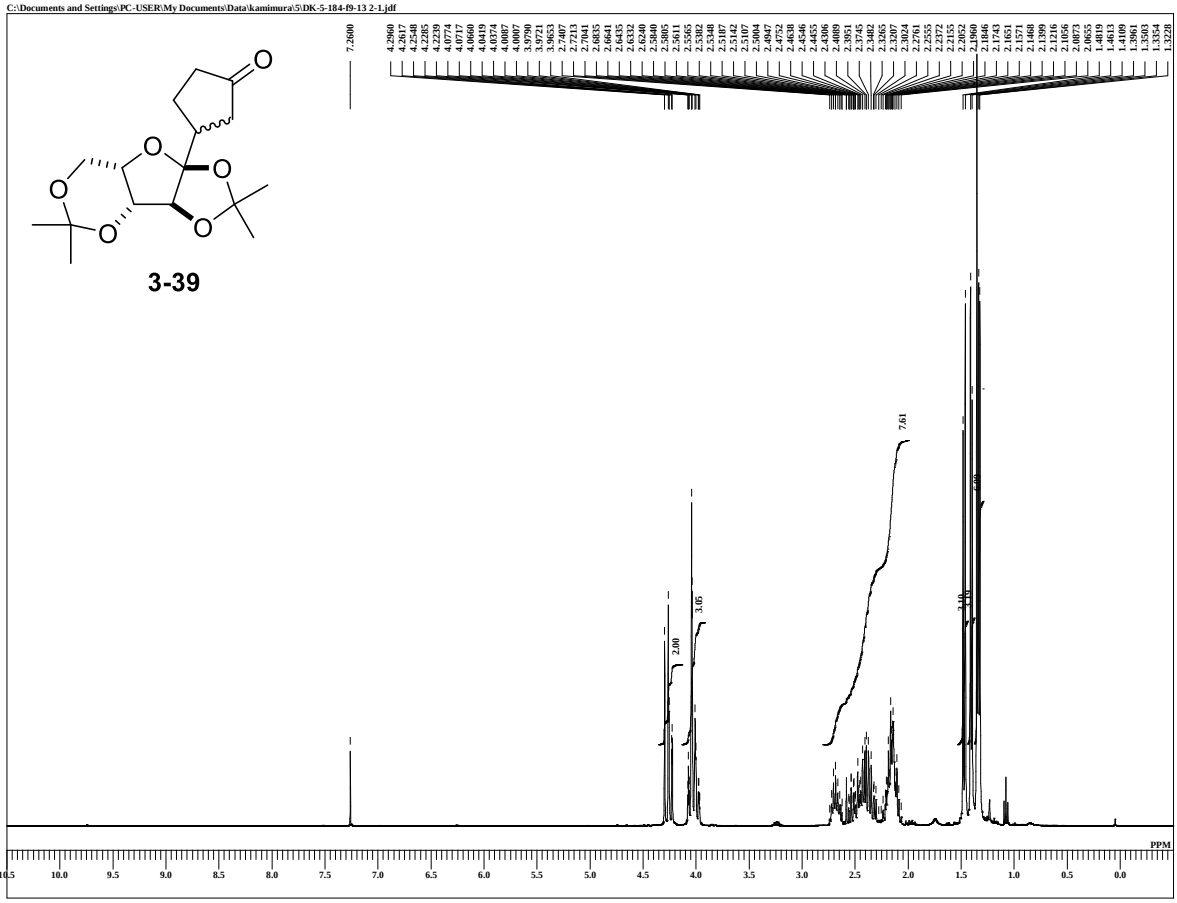


DFILE	DK-5-179-f11-18 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	10-11-2013 09:52:49
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	77
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	77
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-179-f11-18 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

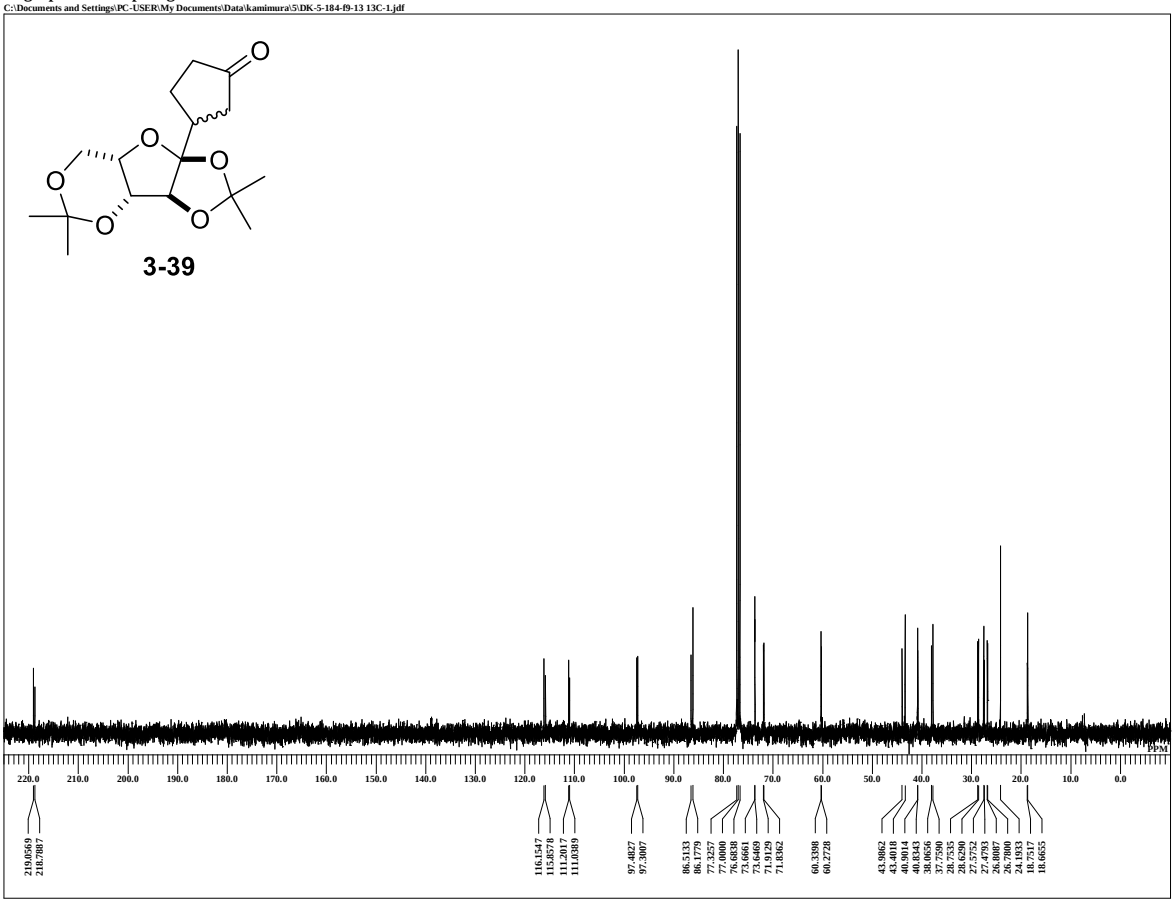
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-184-f9-13 2-1.jdf



DFILE DK-5-184-f9-13 2-1.jdf
COMNT single_pulse
DATIM 29-01-2014 17:07:55
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 28
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-184-f9-13 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-184-f9-13 13C-1.jdf

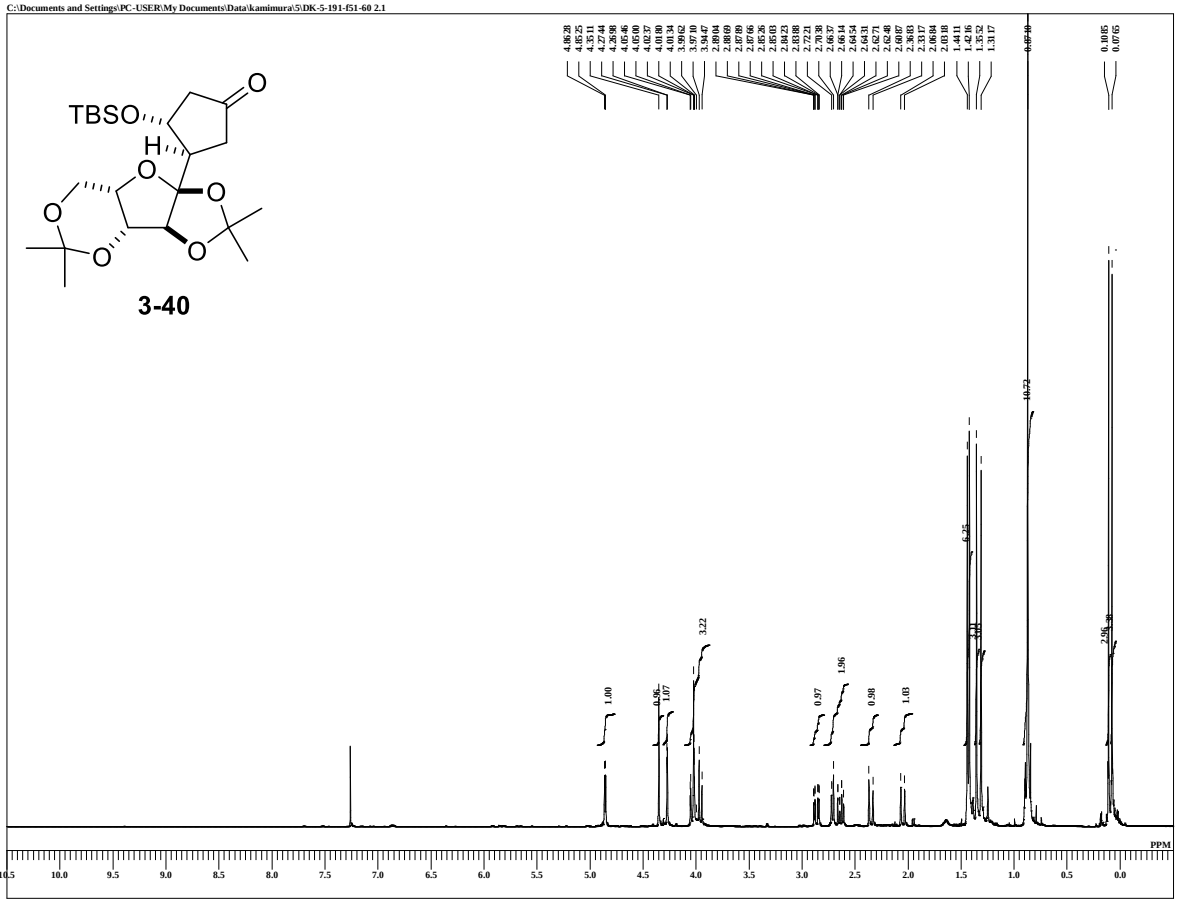


DFILE DK-5-184-f9-13 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 16-11-2013 15:20:15
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 102
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 102
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-184-f9-13 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

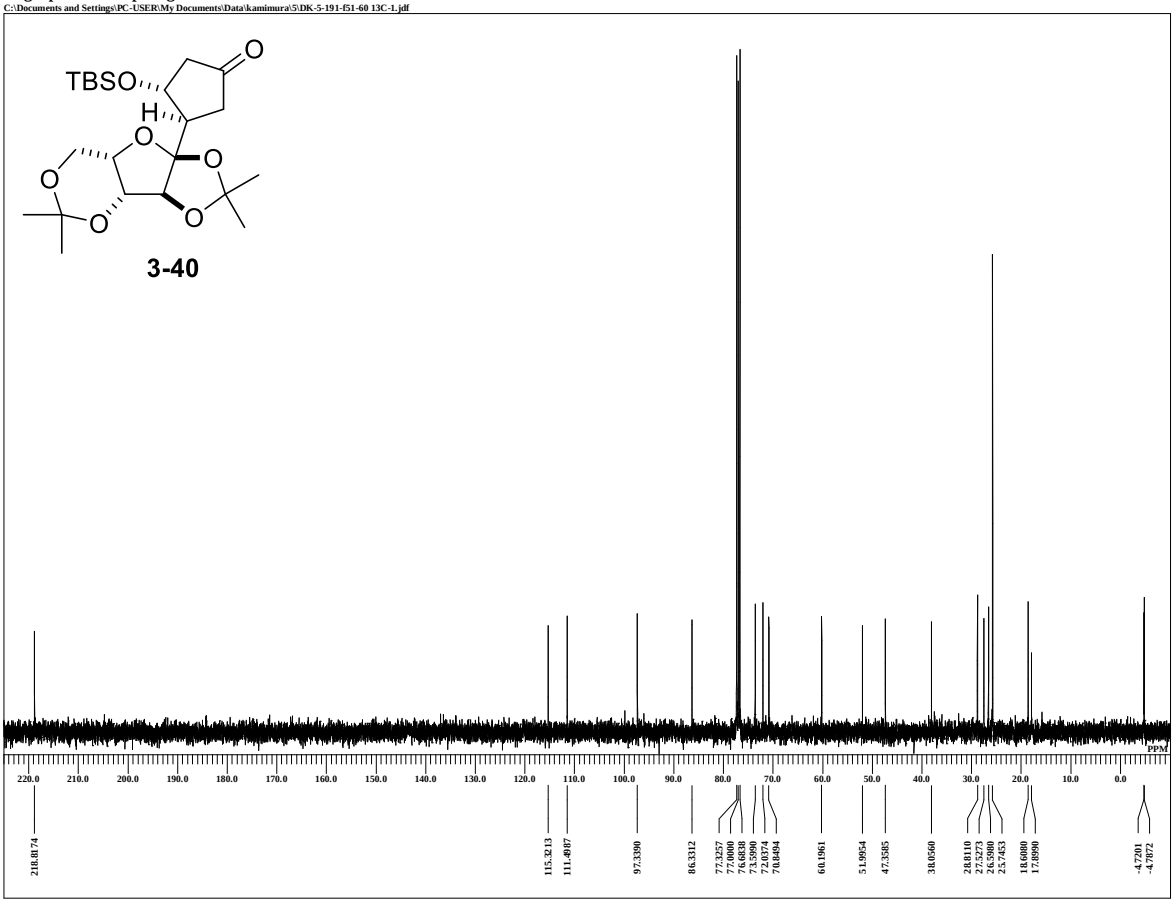
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-191-f51-60 2.1



DFILE	DK-5-191-f51-60 2.1
COMNT	single_pulse
DATIM	15-11-2013 19:46:35
MENUF	
OBNUC	1H
OFR	495.13 MHz
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
PW1	6.00 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	9286.78 Hz
FLT	38000 Hz
DELAY	13.16 usec
ACQTM	1.7642 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	36
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-5-191-f51-60 2.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-191-f51-60 13C-1.jdf

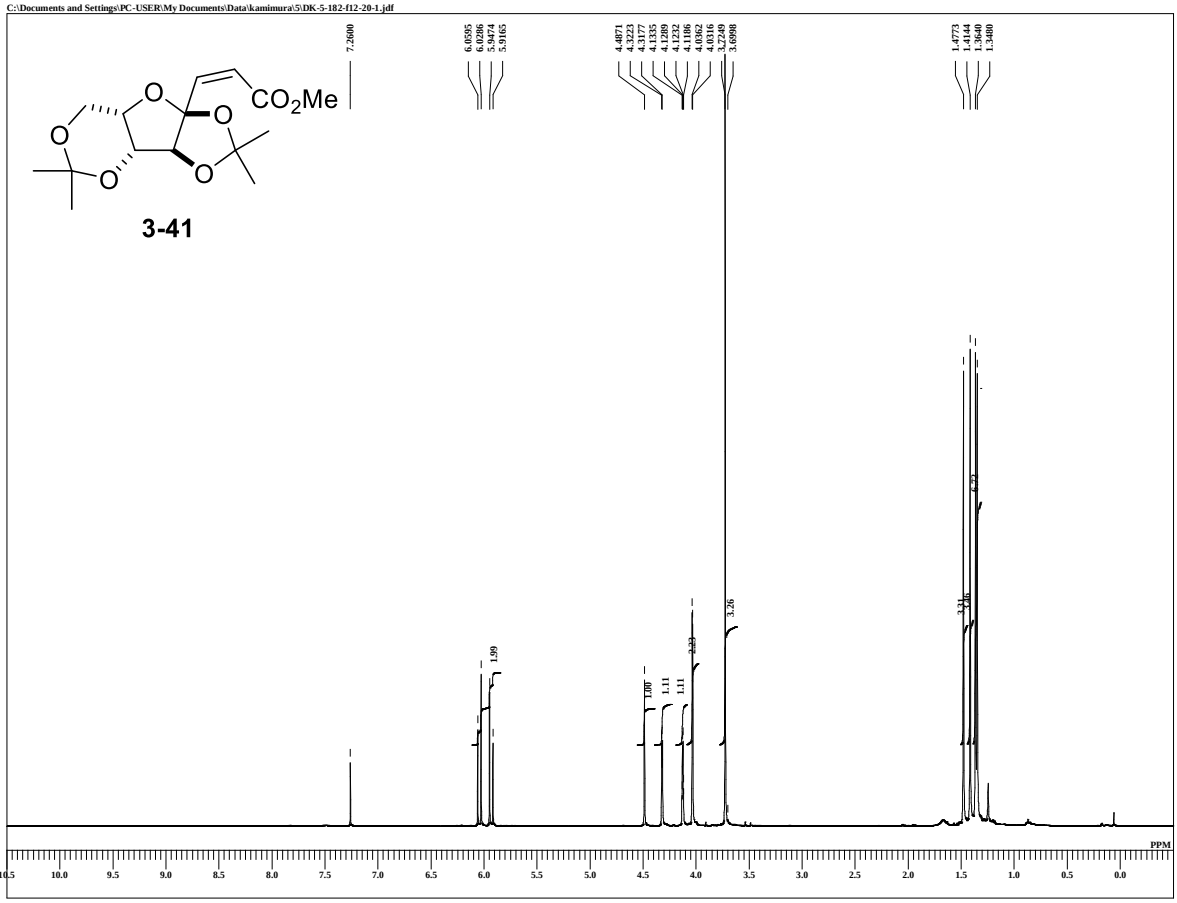


DFILE	DK-5-191-f51-60 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	15-11-2013 15:37:13
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	101
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	101
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-191-f51-60 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

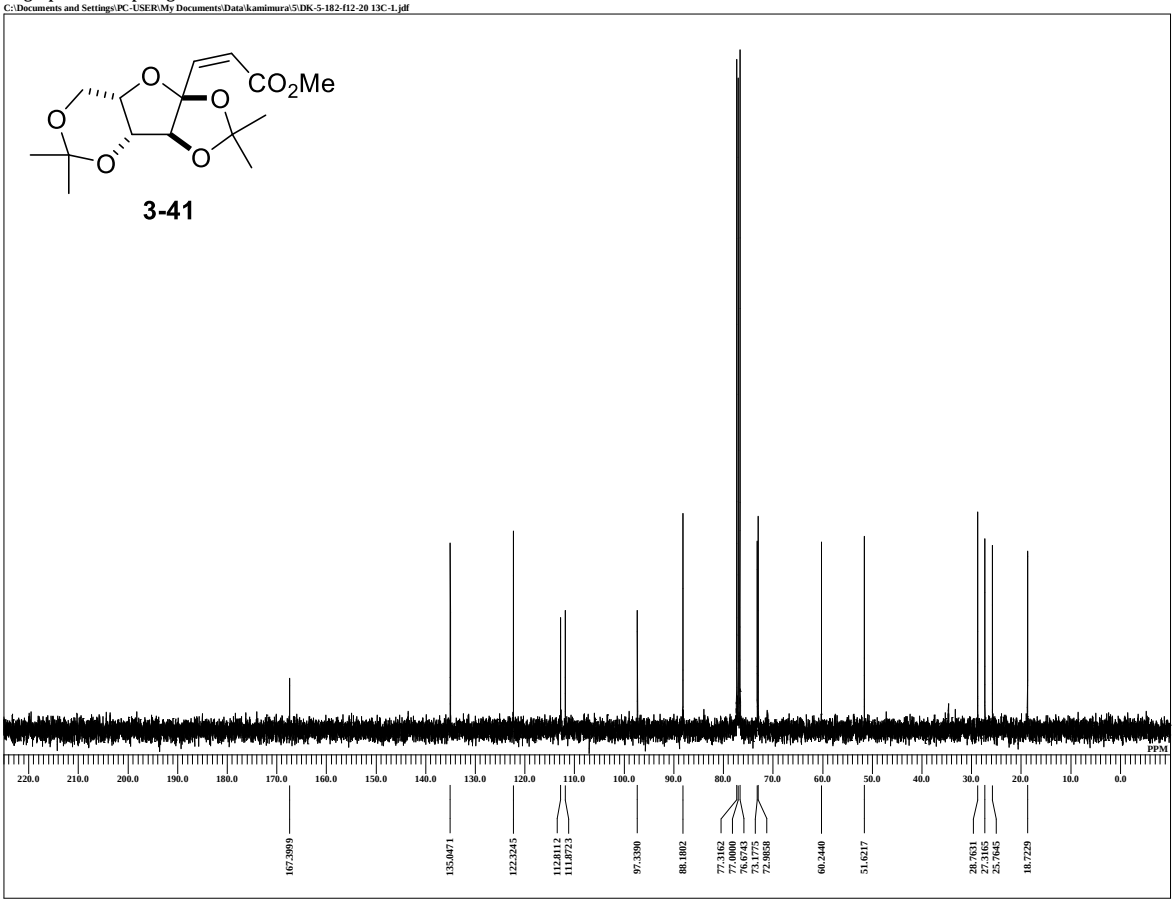
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-182-f12-20-1.jdf




```
DFILE    DK-5-182-f12-20-1.jdf
COMNT    single_pulse
DATIM    11-11-2013 14:15:43
MENUF
OBNUC    1H
OFR        395.88 MHz
OBFRQ      395.88 MHz
OBSET        6.28 KHz
OBFIN        0.87 Hz
PW1          6.44 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        16384
SPO          16384
TIMES            8
DUMMY            1
FREQU        7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM        2.2073 sec
PD           2.0000 sec
SCANS            8
ADBIT           16
RGAIN           34
BF           0.10 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN            79
DFILE    DK-5-182-f12-20-1.jdf
SF
LKSET        13.20 KHz
LKFIN        75.7 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED            0 Hz
FILDC
FILDF
CTEMP        23.2 c
SLVNT    CDCL3
EXREF        7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-182-f12-20 13C-1.jdf

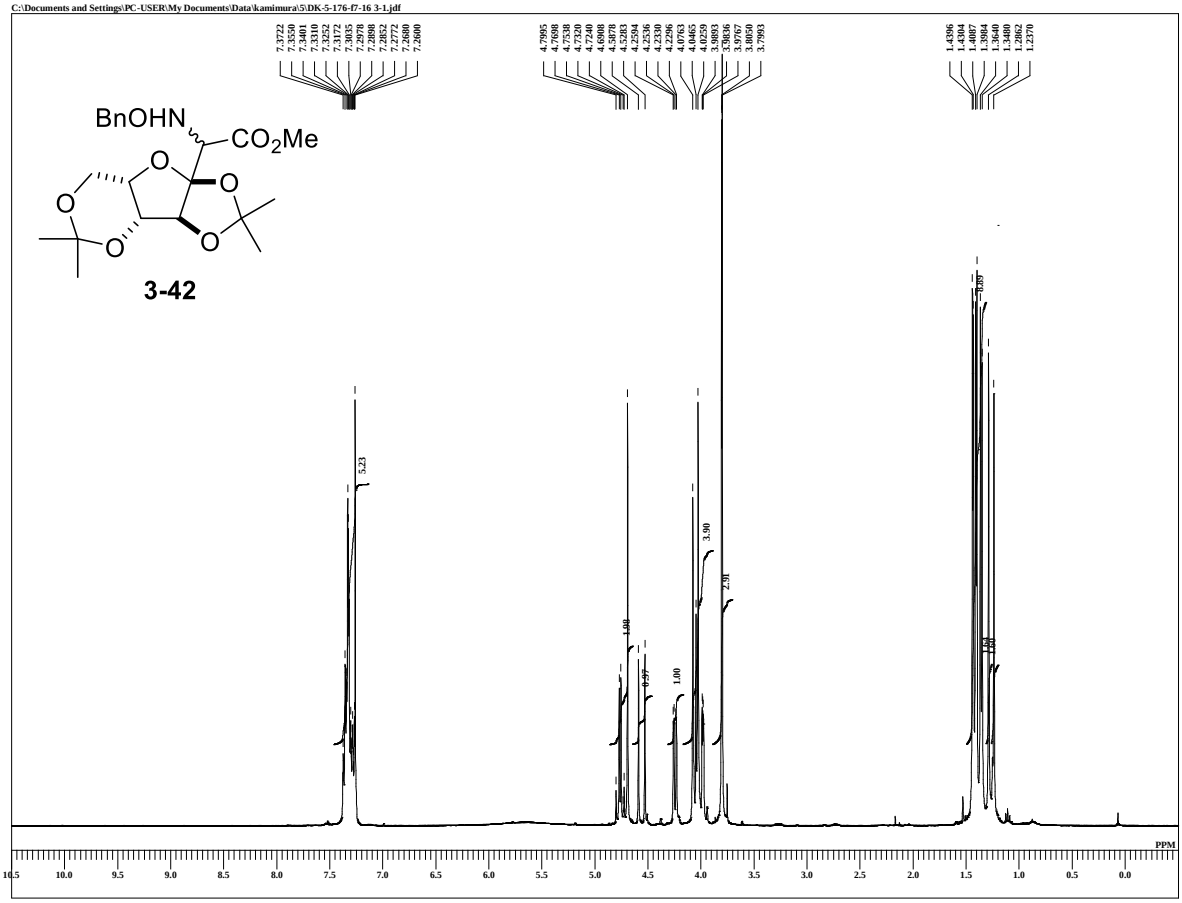



```
DFILE    DK-5-182-f12-20 13C-1.jd
COMNT    single pulse decoupled gat
DATIM    11-11-2013 14:19:30
MENUF
OBNUC    13C
OFR        99.55 MHz
OBFRQ      99.55 MHz
OBSET        5.13 KHz
OBFIN        0.98 Hz
PW1          3.03 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        32768
SPO          32768
TIMES           63
DUMMY            4
FREQU        31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM        1.0486 sec
PD           2.0000 sec
SCANS           63
ADBIT           16
RGAIN           60
BF           1.00 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN            79
DFILE    DK-5-182-f12-20 13C-1.jd
SF
LKSET        13.20 KHz
LKFIN        75.7 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED            0 Hz
FILDC
FILDF
CTEMP        23.4 c
SLVNT    CDCL3
EXREF        77.00 ppm
```

スペクトルデータ

single_pulse

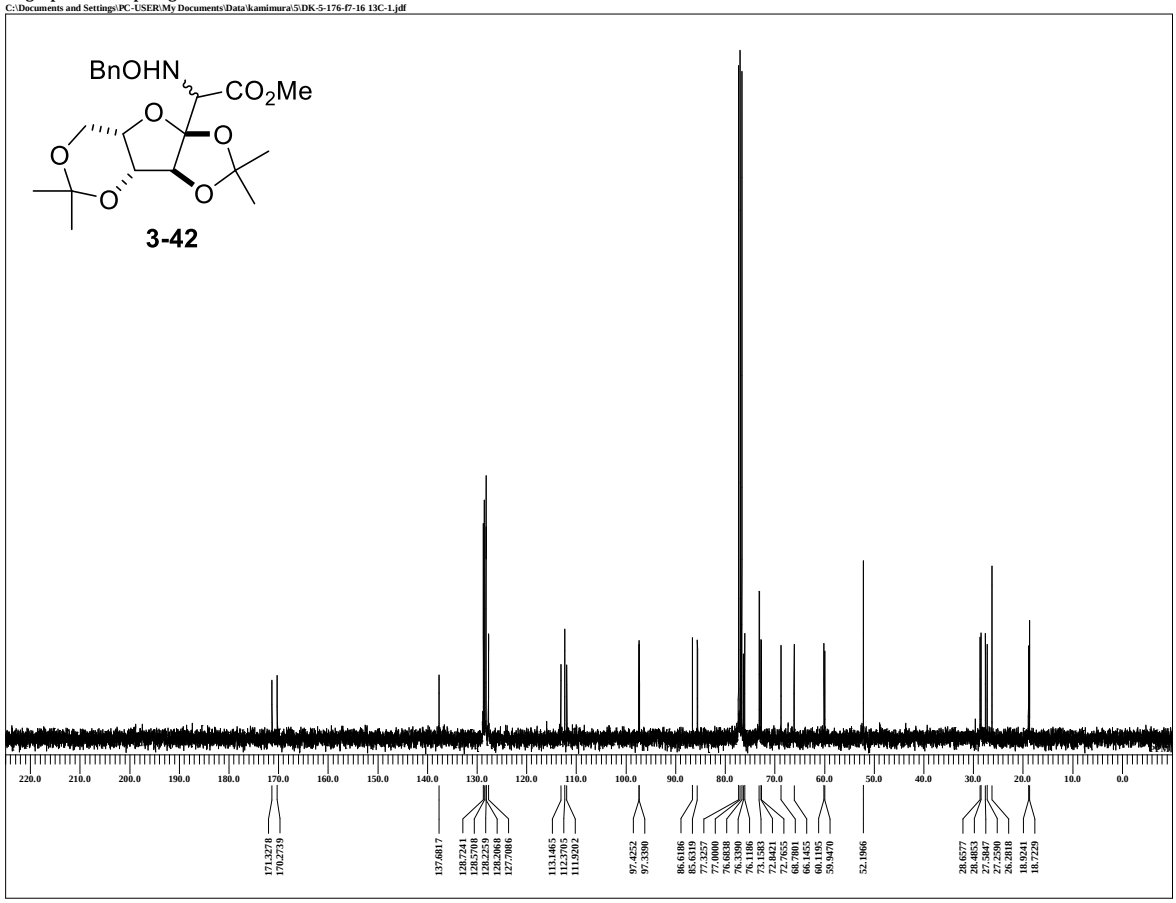
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-176-f7-16 3-1.jdf




```
DFILE     DK-5-176-f7-16 3-1.jdf
COMNT     single_pulse
DATIM     27-01-2014 18:21:20
MENUF
OBNUC     1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET     6.28 KHz
OBFIN     0.87 Hz
PW1       6.44 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     16384
SPO       16384
TIMES     8
DUMMY     1
FREQU     7422.80 Hz
FLT       30000 Hz
DELAY     16.68 usec
ACQTM     2.2073 sec
PD        2.0000 sec
SCANS     8
ADBIT     16
RGAIN     32
BF        0.10 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     DK-5-176-f7-16 3-1.jdf
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     20.9 c
SLVNT     CDCL3
EXREF     7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-176-f7-16 13C-1.jdf

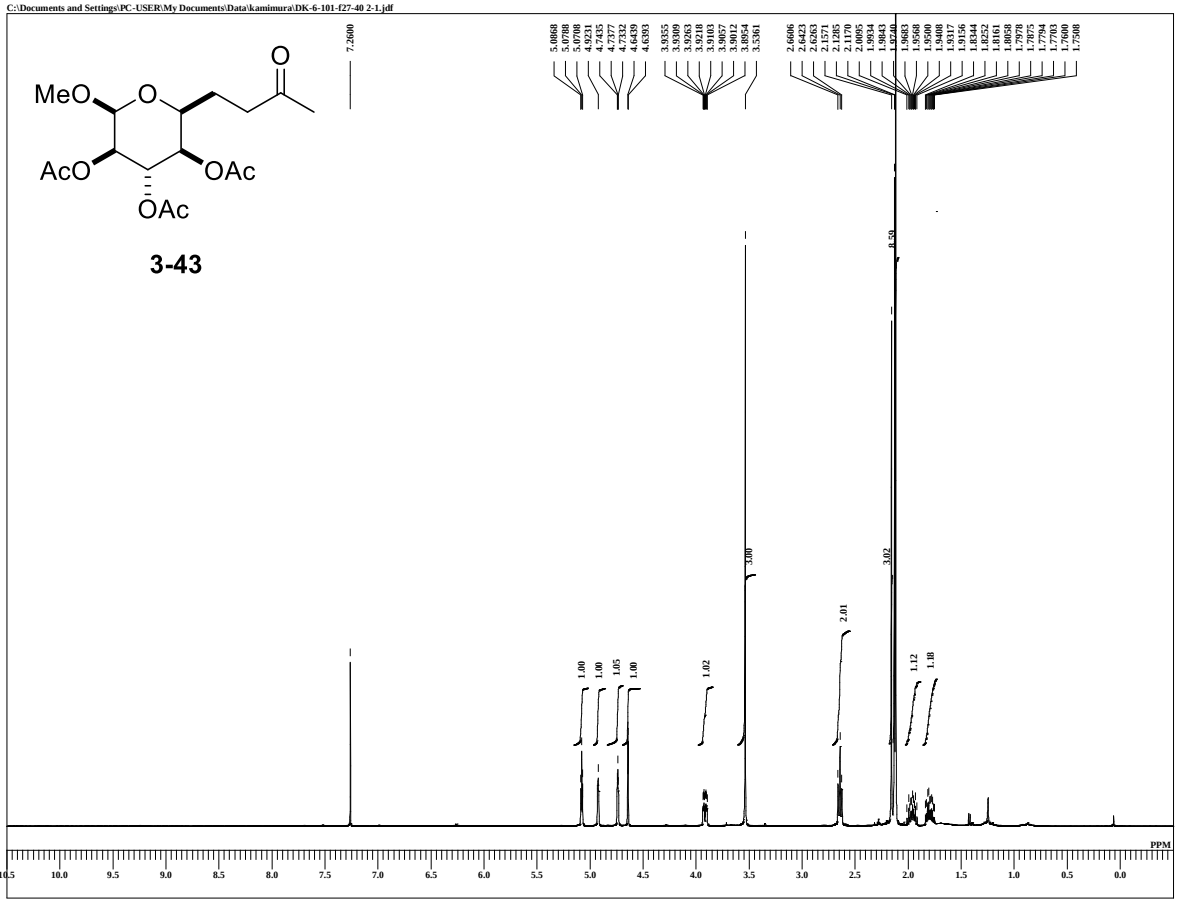



```
DFILE     DK-5-176-f7-16 13C-1.jdf
COMNT     single pulse decoupled gat
DATIM     08-11-2013 18:10:46
MENUF
OBNUC     13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET     5.13 KHz
OBFIN     0.98 Hz
PW1       3.03 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     32768
SPO       32768
TIMES     109
DUMMY     4
FREQU     31250.00 Hz
FLT       125000 Hz
DELAY     20.50 usec
ACQTM     1.0486 sec
PD        2.0000 sec
SCANS     109
ADBIT     16
RGAIN     60
BF        1.00 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     DK-5-176-f7-16 13C-1.jdf
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     23.4 c
SLVNT     CDCL3
EXREF     77.00 ppm
```

スペクトルデータ

single_pulse

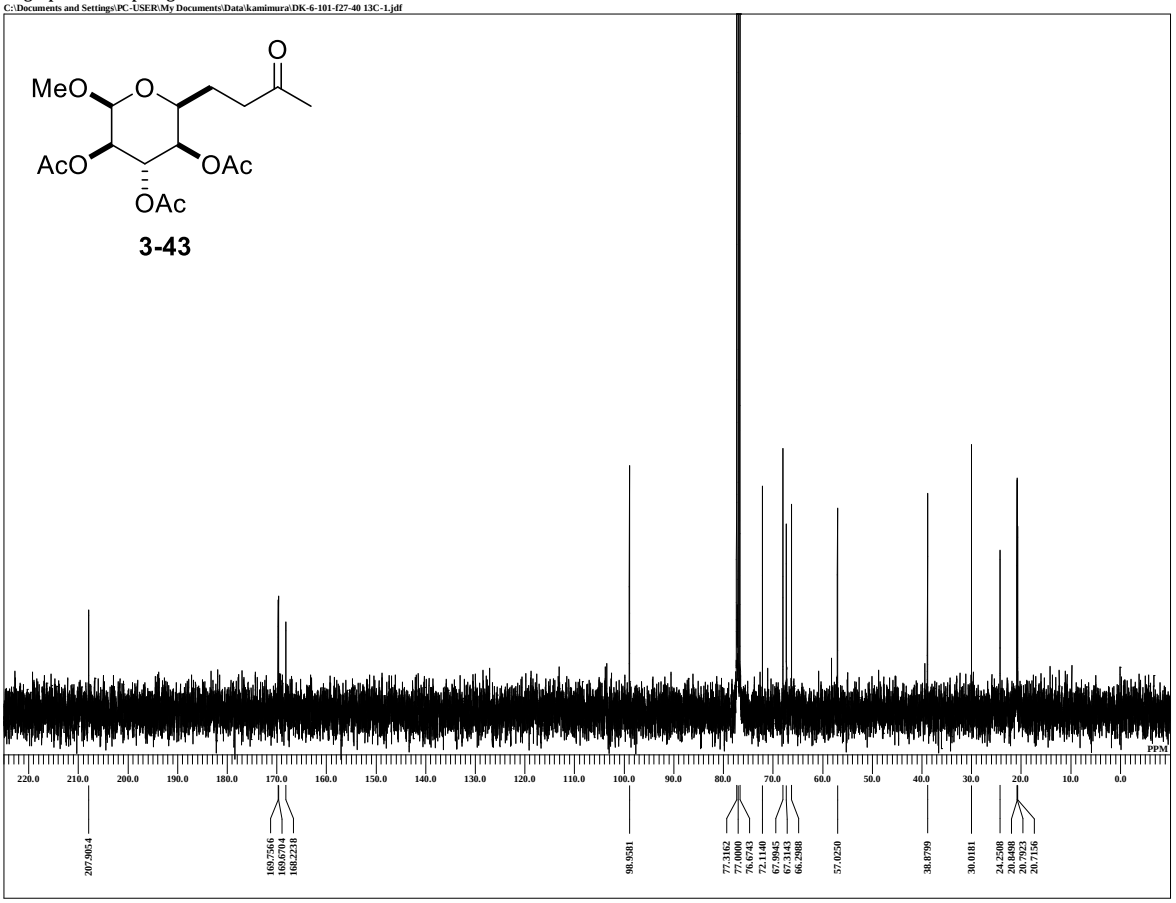
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-101-f27-40 2-1.jdf



DFILE DK-6-101-f27-40 2-1.jdf
COMNT single_pulse
DATIM 04-02-2014 15:21:37
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-6-101-f27-40 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-101-f27-40 13C-1.jdf

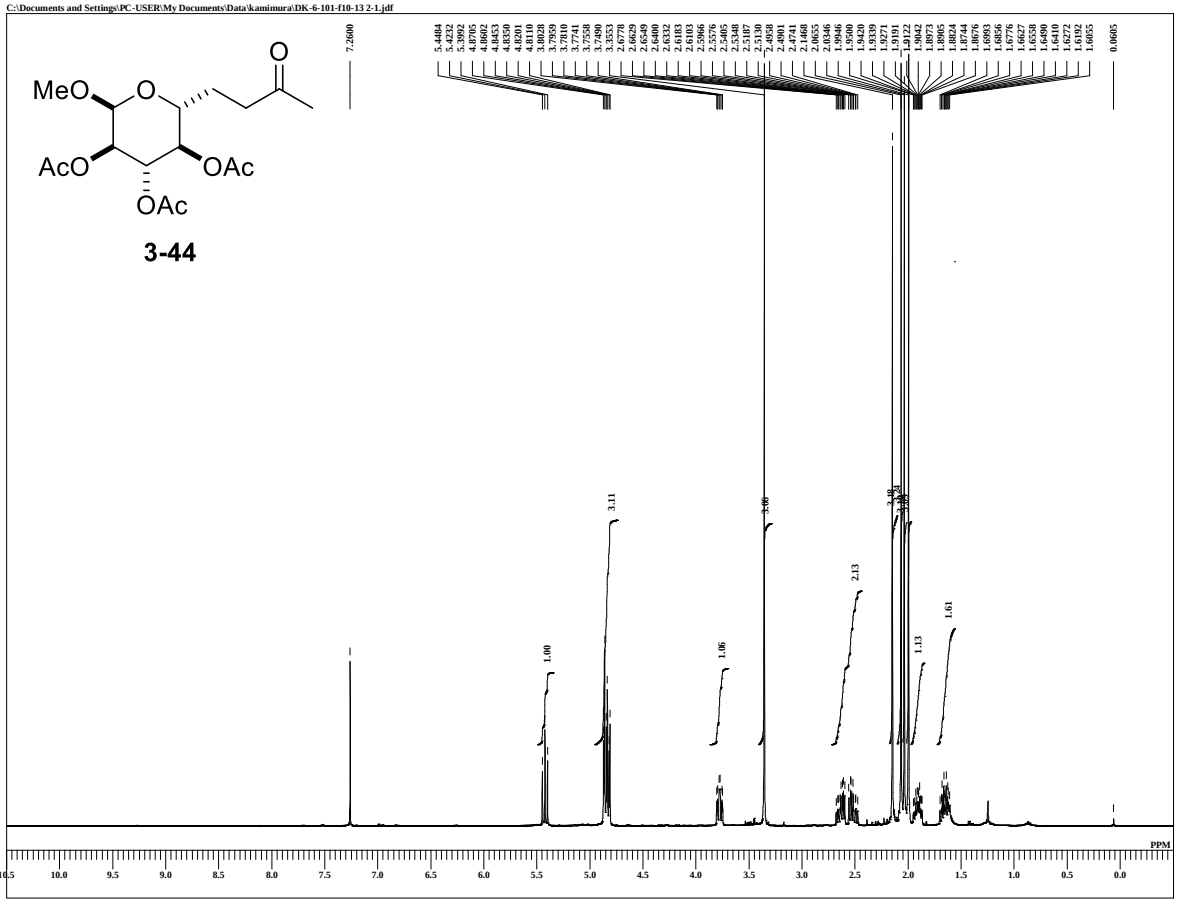


DFILE DK-6-101-f27-40 13C-1.jd
COMNT single pulse decoupled gat
DATIM 04-02-2014 09:13:21
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 181
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 181
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-101-f27-40 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

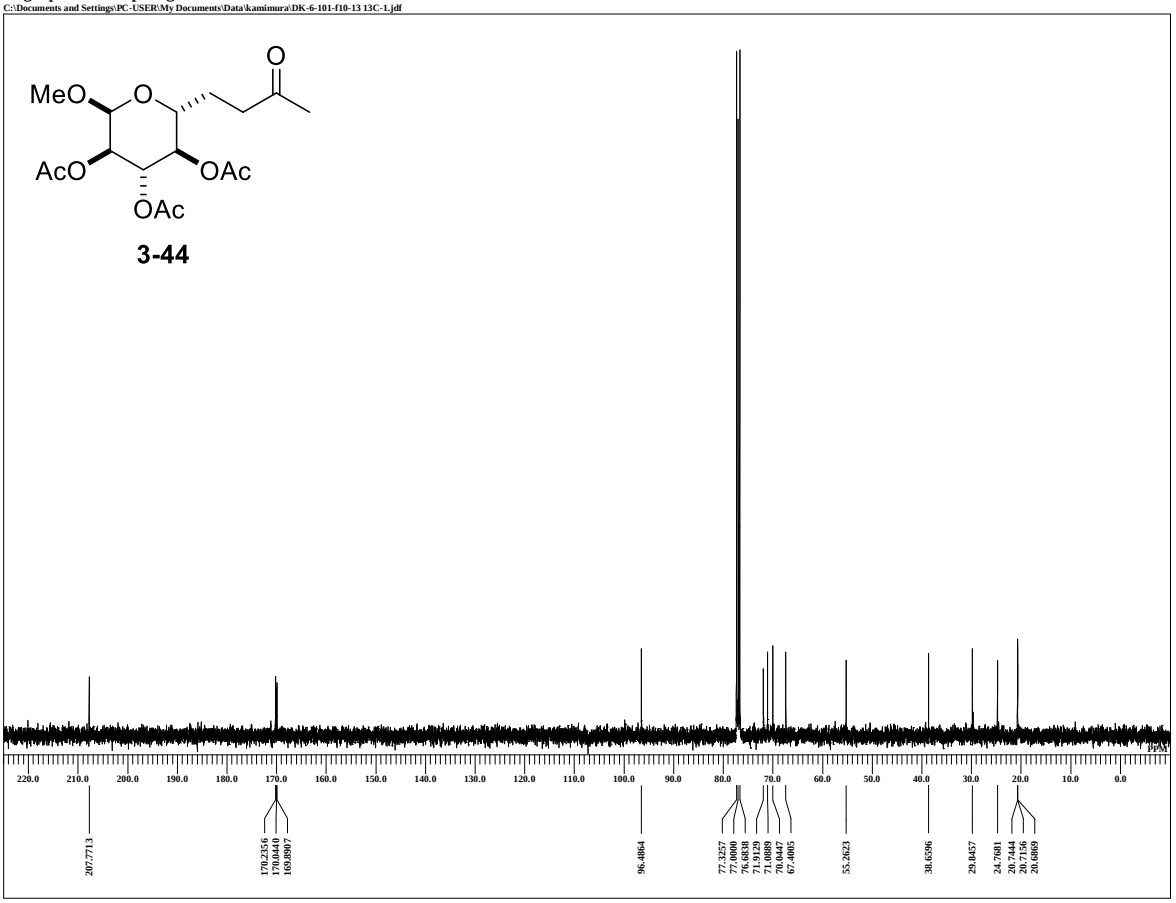
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-101-f10-13 2-1.jdf



DFILE DK-6-101-f10-13 2-1.jdf
COMNT single_pulse
DATIM 04-02-2014 15:16:40
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-6-101-f10-13 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.6 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-101-f10-13 13C-1.jdf

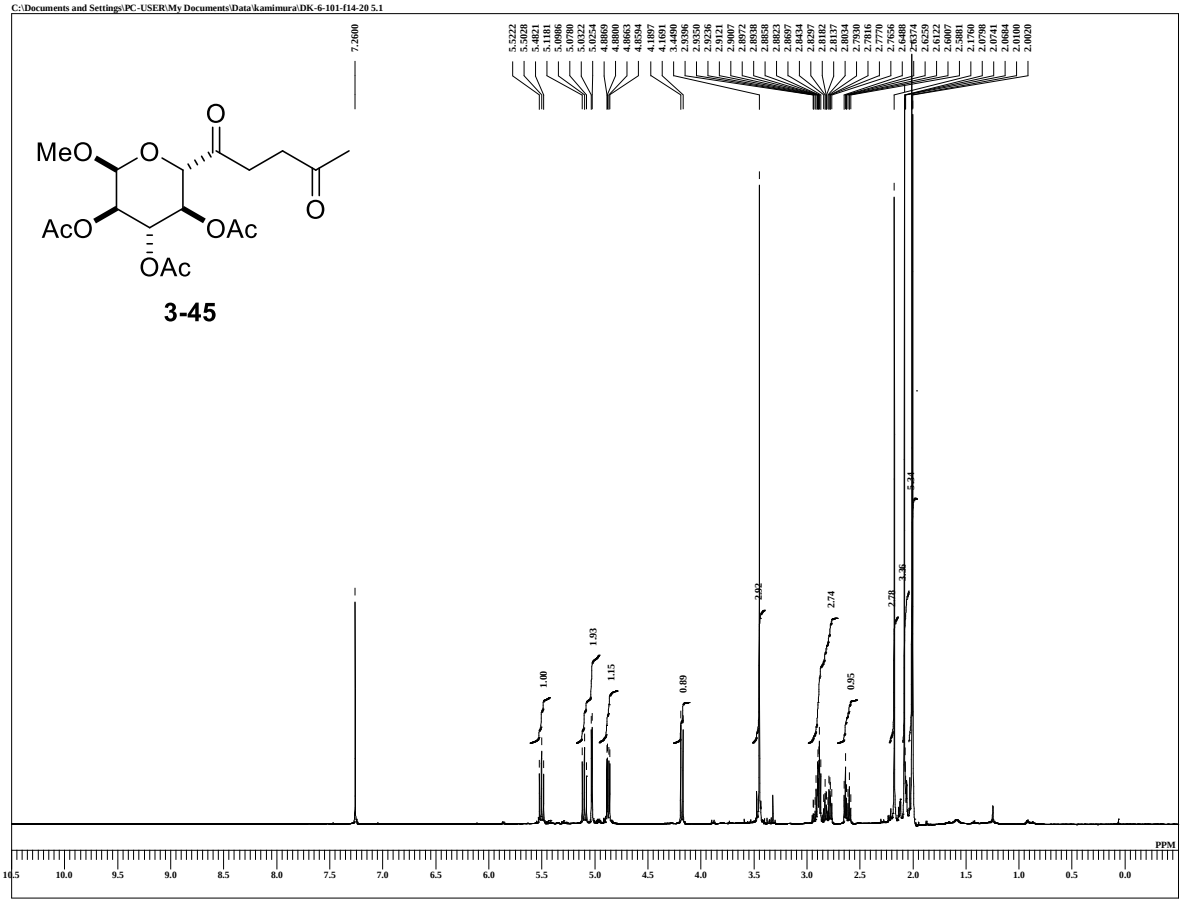


DFILE DK-6-101-f10-13 13C-1.jd
COMNT single pulse decoupled gat
DATIM 04-02-2014 11:46:44
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 151
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 151
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-101-f10-13 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

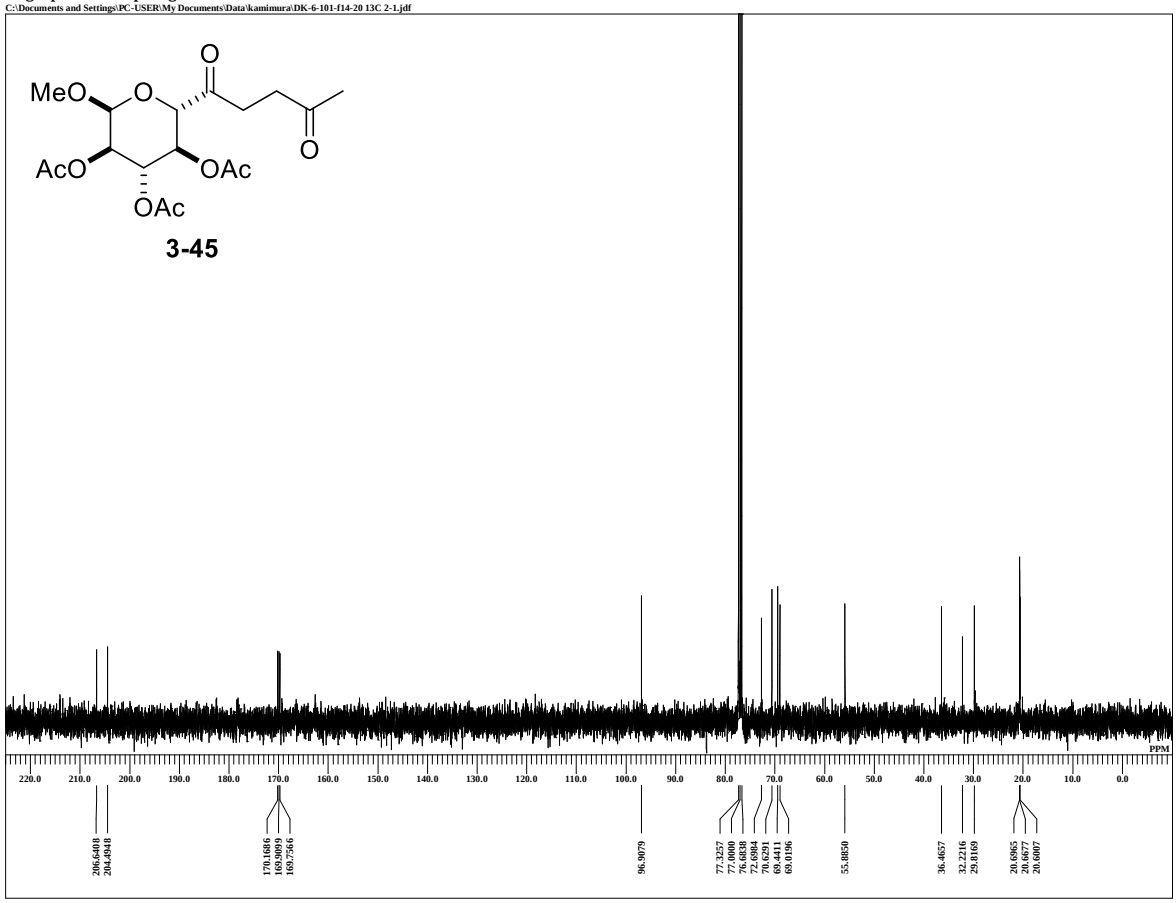
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-101-f14-20 5.1



DFILE DK-6-101-f14-20 5.1
COMNT single_pulse
DATIM 07-02-2014 13:29:59
MENUF
OBNUC 1H
OFR 495.13 MHz
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
PW1 6.00 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 9286.78 Hz
FLT 38000 Hz
DELAY 13.16 usec
ACQTM 1.7642 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 48
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE DK-6-101-f14-20 5.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.8 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-101-f14-20 13C 2-1.jdf

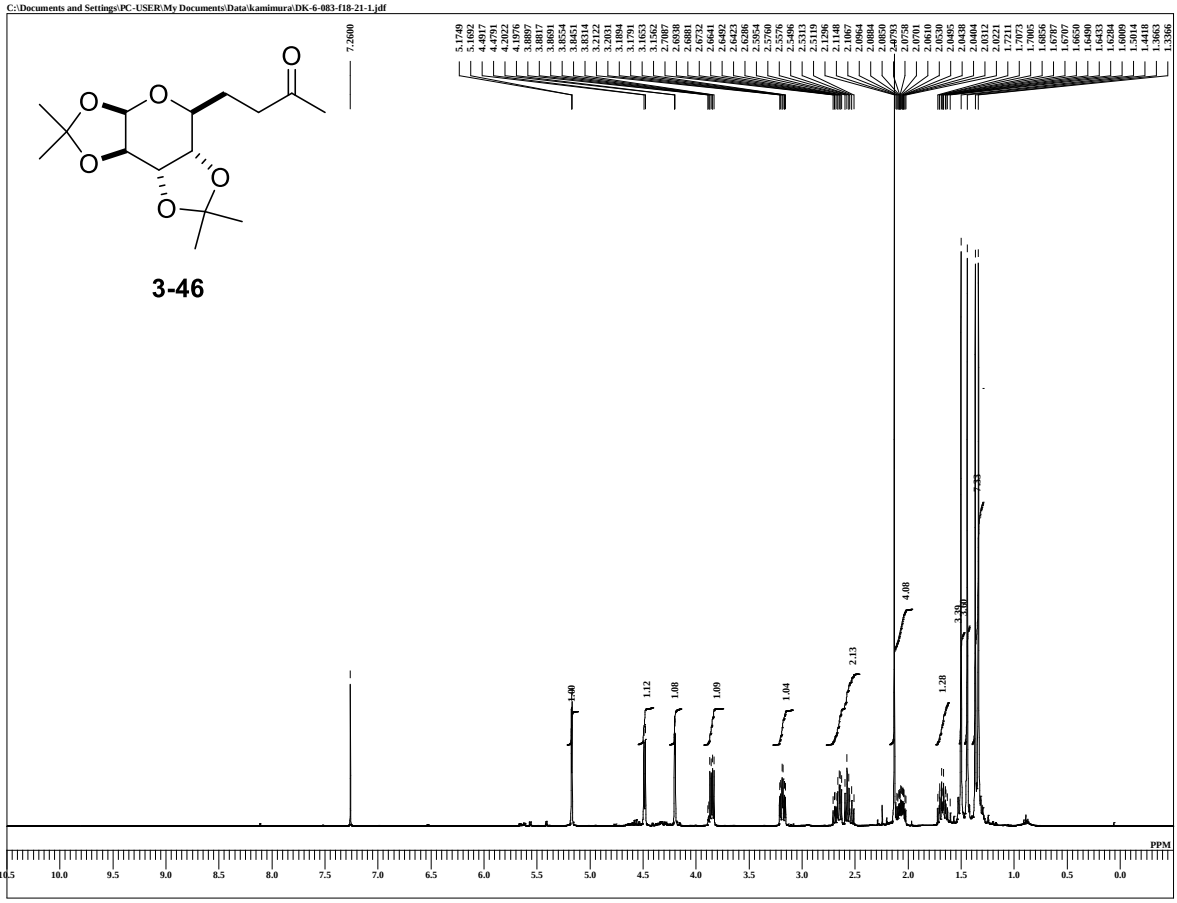


DFILE DK-6-101-f14-20 13C 2-1.
COMNT single pulse decoupled gat
DATIM 06-02-2014 18:12:03
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 158
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 158
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-101-f14-20 13C 2-1.
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

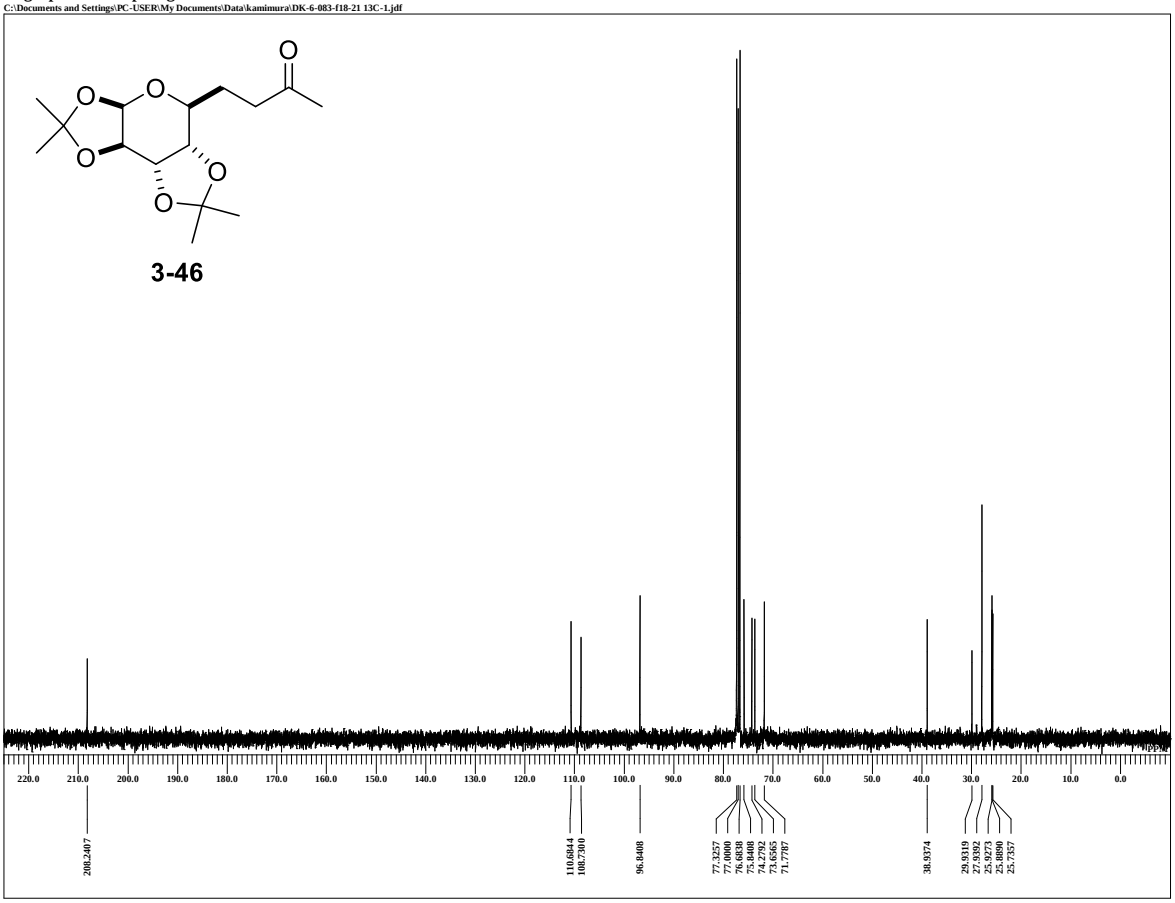
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-083-f18-21-1.jdf



DFILE DK-6-083-f18-21-1.jdf
COMNT single_pulse
DATIM 15-01-2014 14:32:34
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-083-f18-21-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-083-f18-21 13C-1.jdf

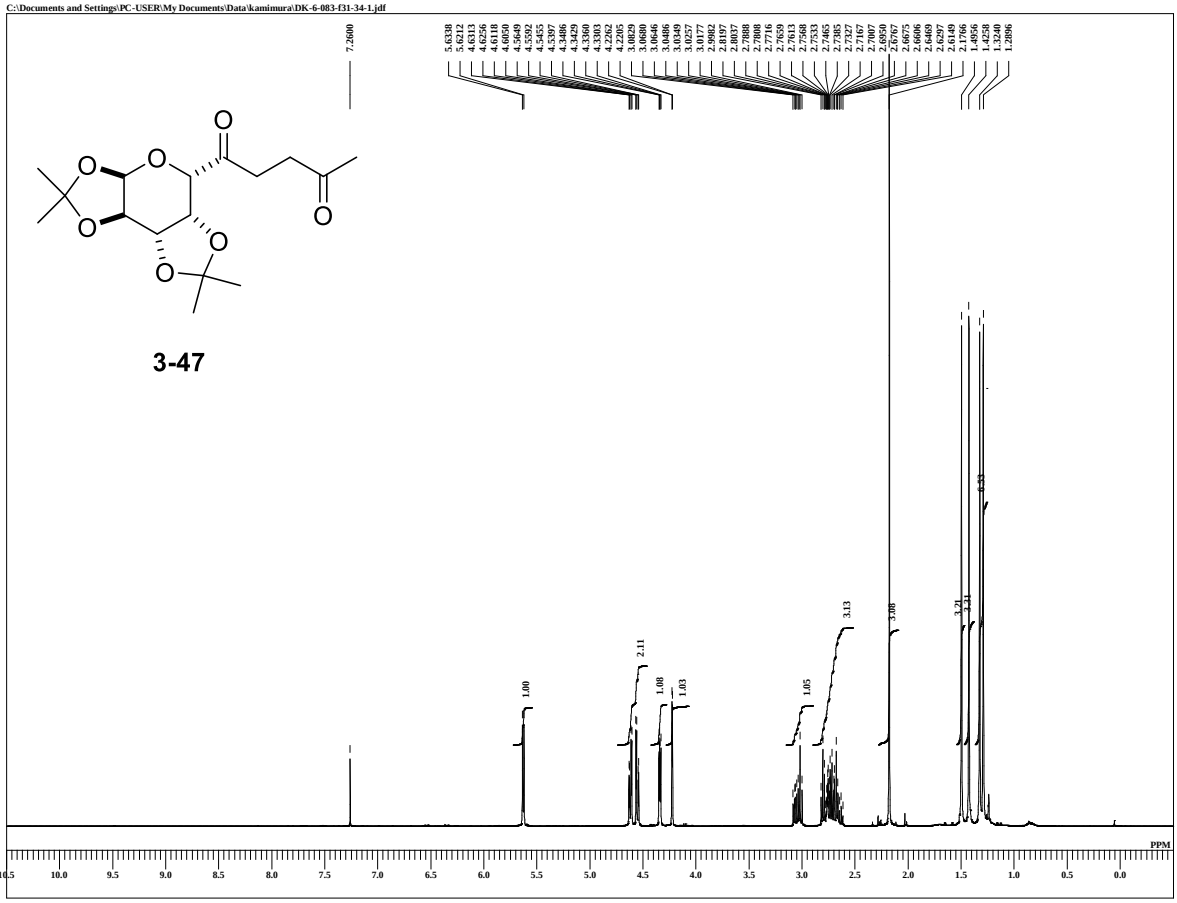


DFILE DK-6-083-f18-21 13C-1.jd
COMNT single pulse decoupled gat
DATIM 15-01-2014 14:41:28
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 164
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 164
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-083-f18-21 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

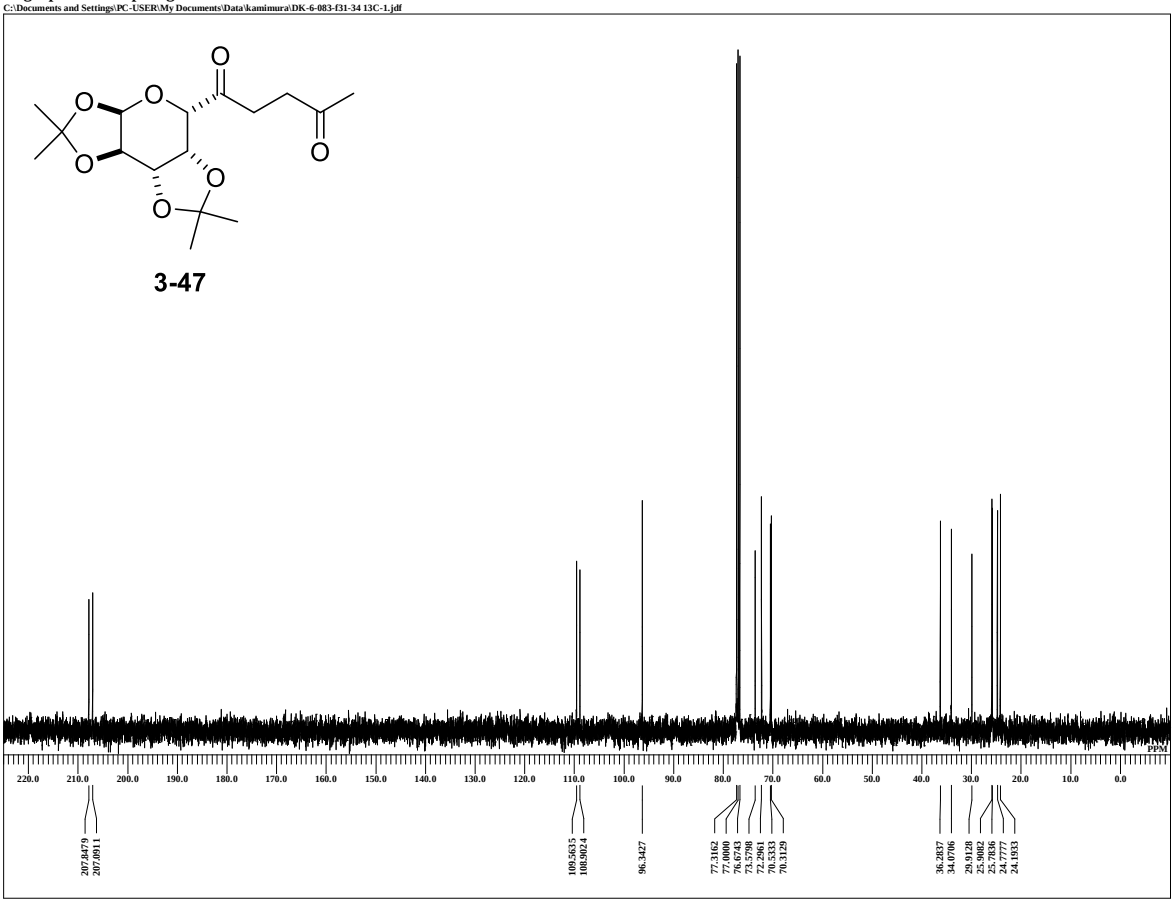
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-083-f31-34-1.jdf



DFILE	DK-6-083-f31-34-1.jdf
COMNT	single_pulse
DATIM	17-01-2014 11:49:29
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	147 usec
IRATN	79
DFILE	DK-6-083-f31-34-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-083-f31-34 13C-1.jdf

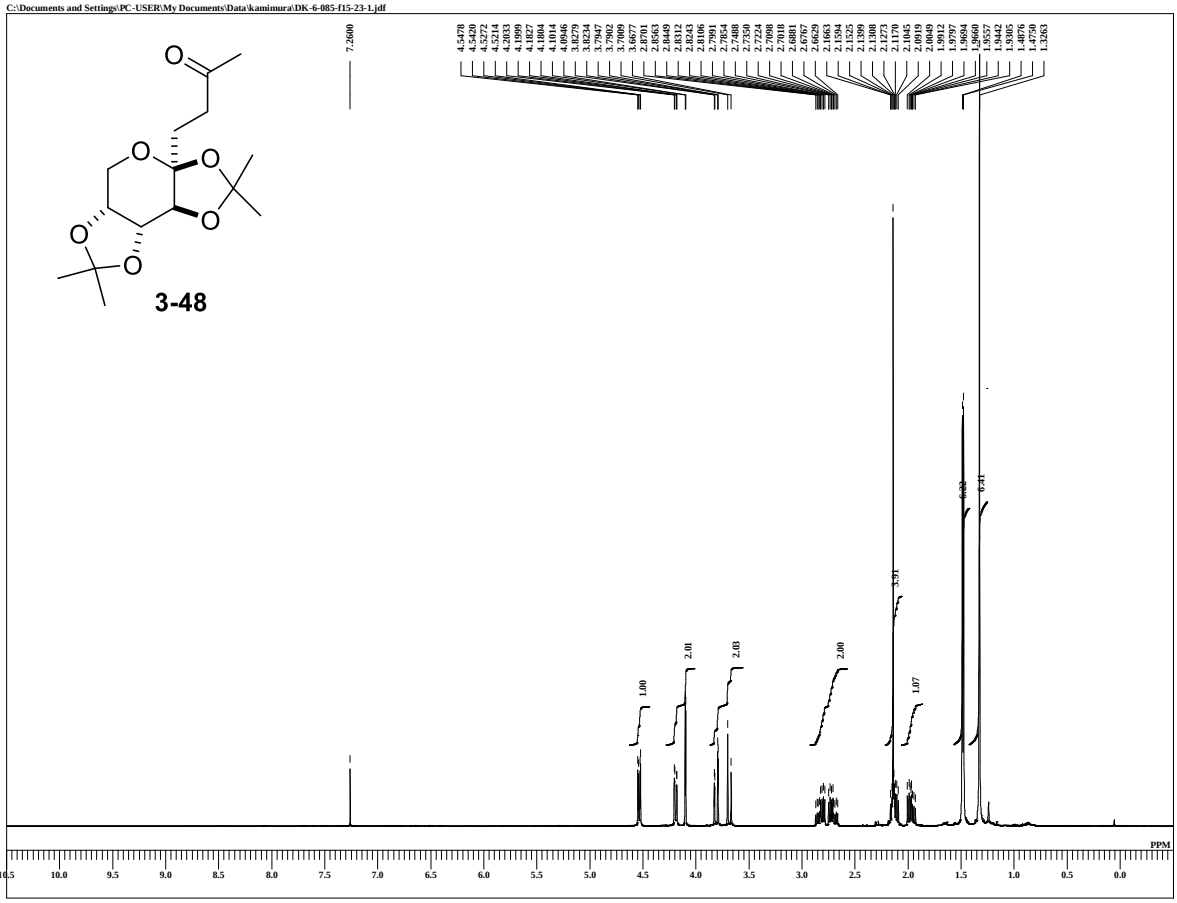


DFILE	DK-6-083-f31-34 13C-1.jc
COMNT	single pulse decoupled gat
DATIM	17-01-2014 11:53:13
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	62
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	62
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-083-f31-34 13C-1.jc
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

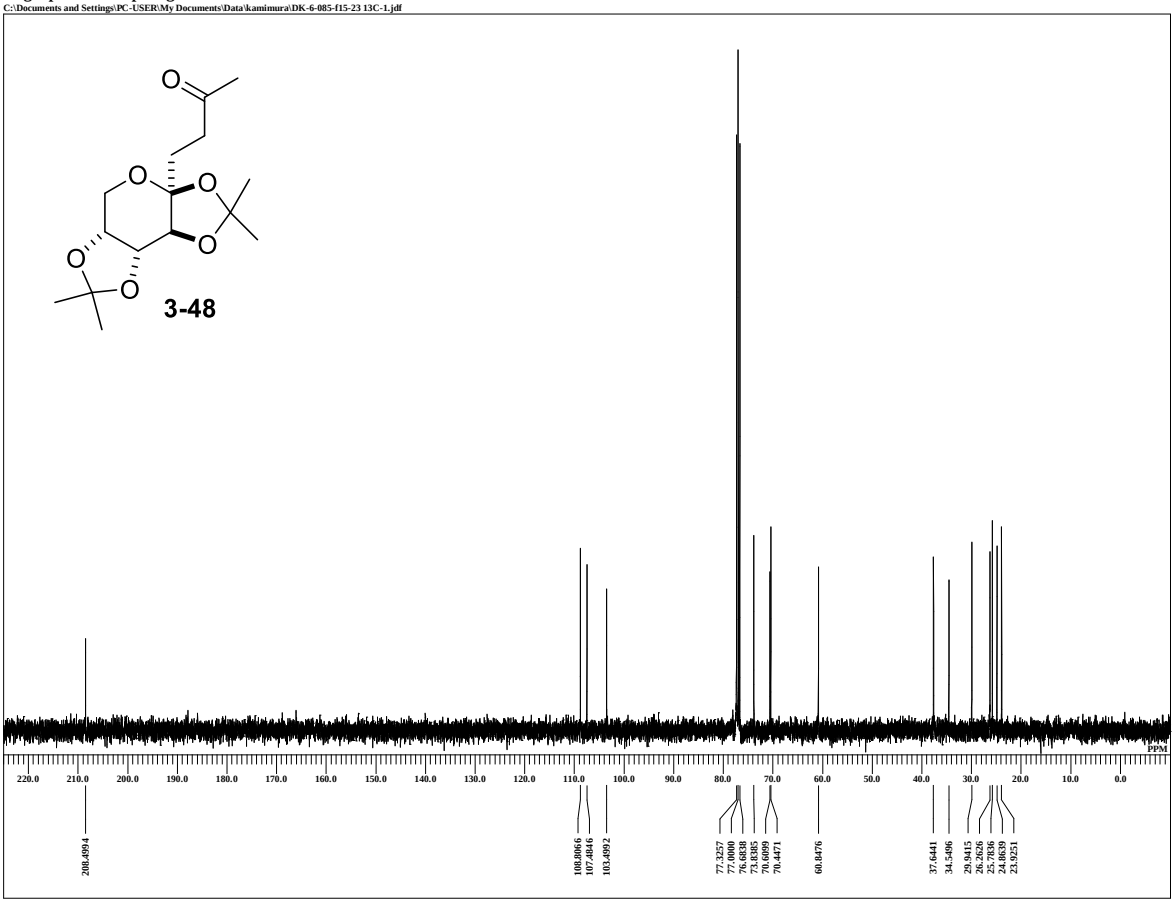
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-085-f15-23-1.jdf



DFILE DK-6-085-f15-23-1.jdf
COMNT single_pulse
DATIM 17-01-2014 15:45:01
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-085-f15-23-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-085-f15-23 13C-1.jdf

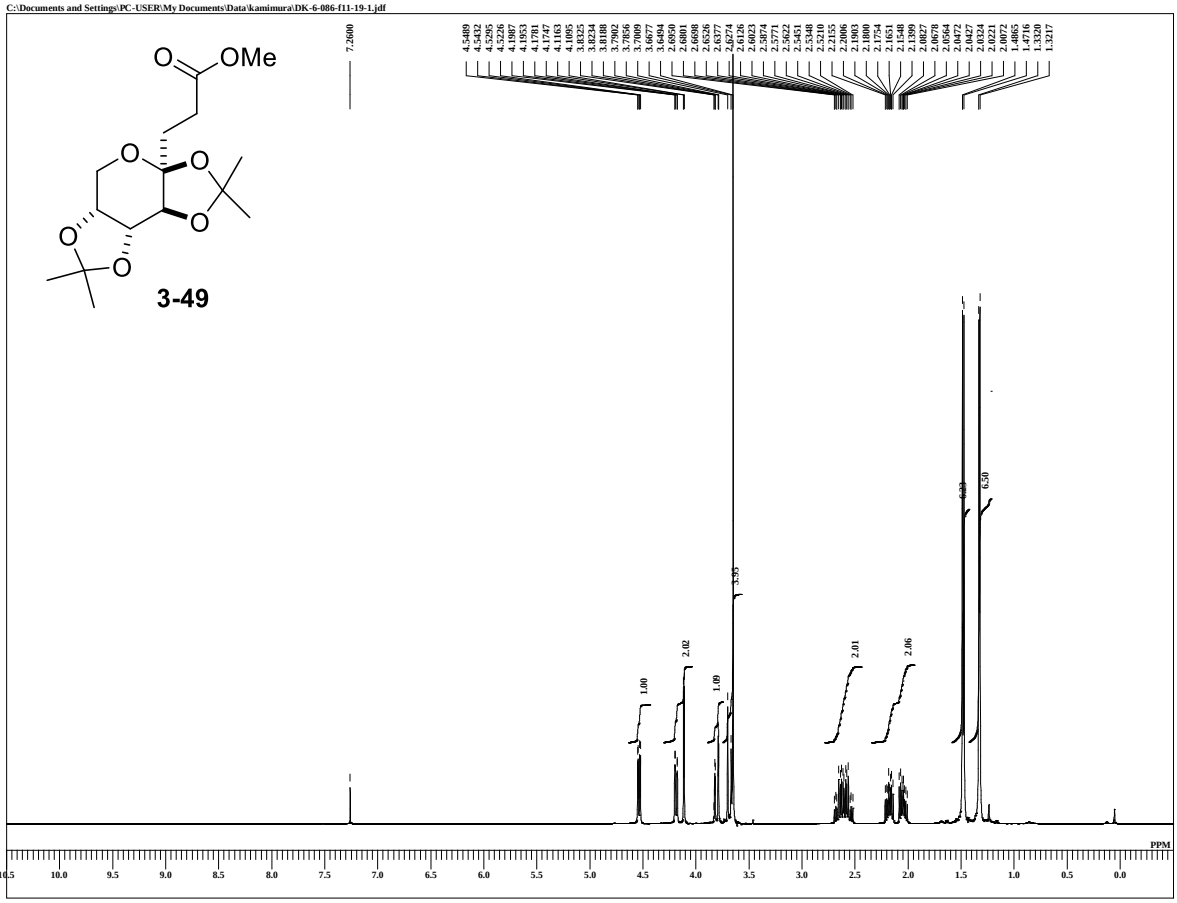


DFILE DK-6-085-f15-23 13C-1.jd
COMNT single pulse decoupled gat
DATIM 17-01-2014 15:49:48
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 83
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 83
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-085-f15-23 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

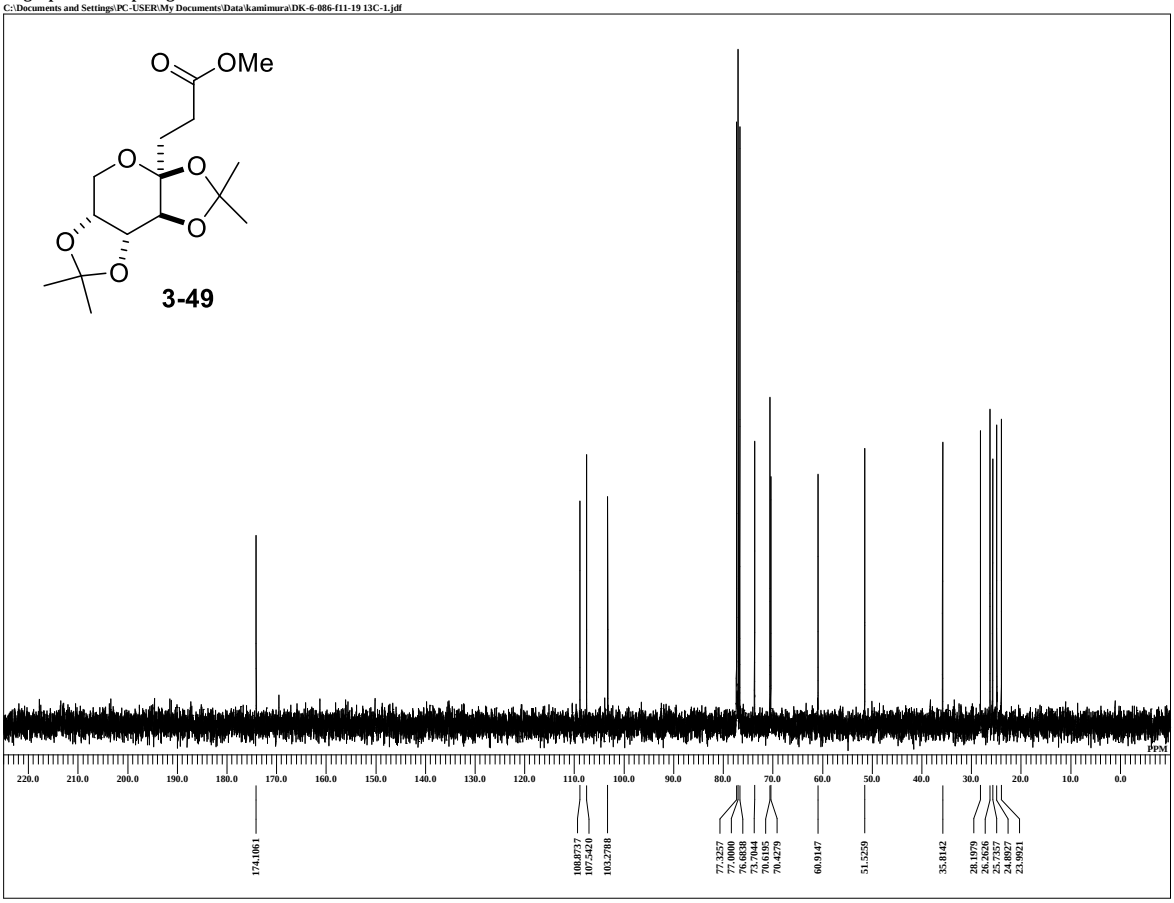
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-086-f11-19-1.jdf



DFILE DK-6-086-f11-19-1.jdf
COMNT single_pulse
DATIM 17-01-2014 18:13:32
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 28
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-086-f11-19-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-086-f11-19 13C-1.jdf

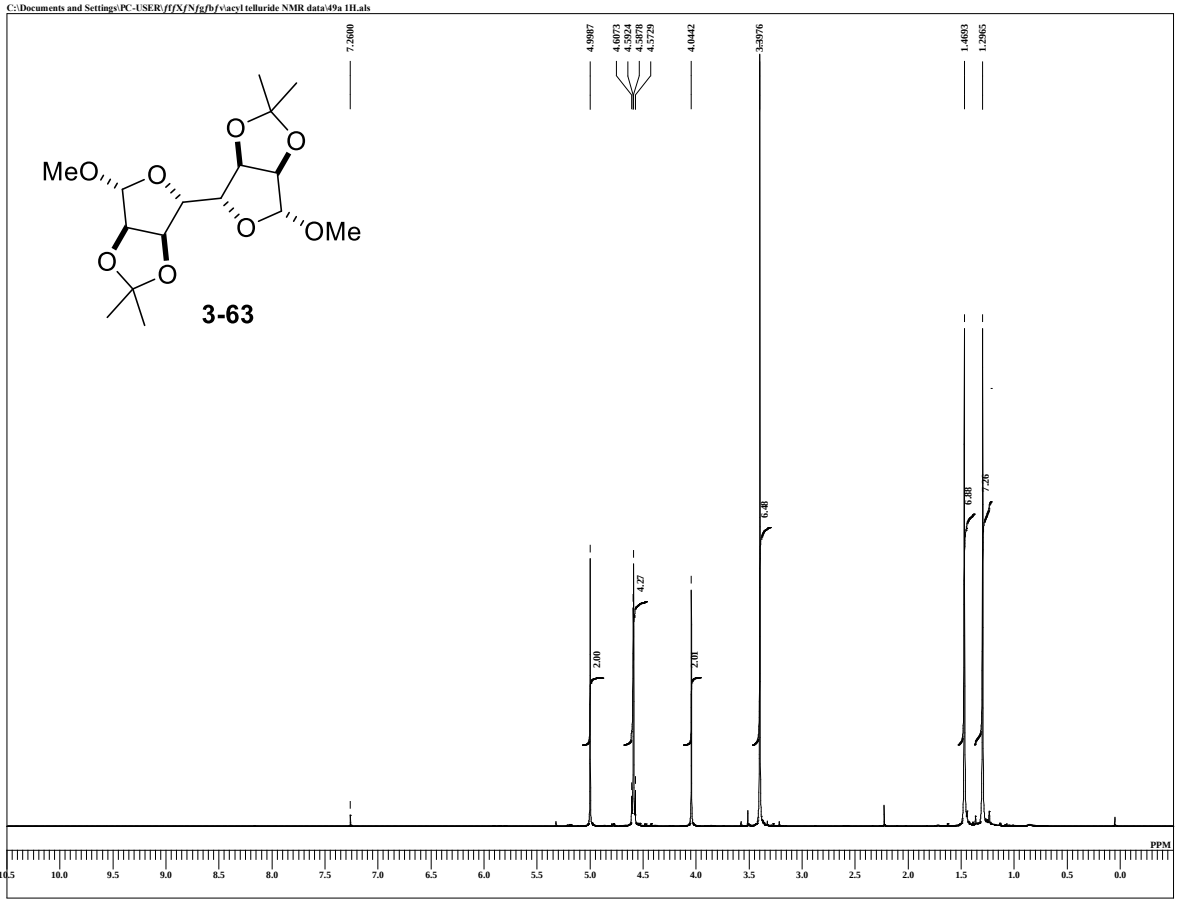


DFILE DK-6-086-f11-19 13C-1.jd
COMNT single pulse decoupled gat
DATIM 17-01-2014 18:16:11
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 41
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 41
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-086-f11-19 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

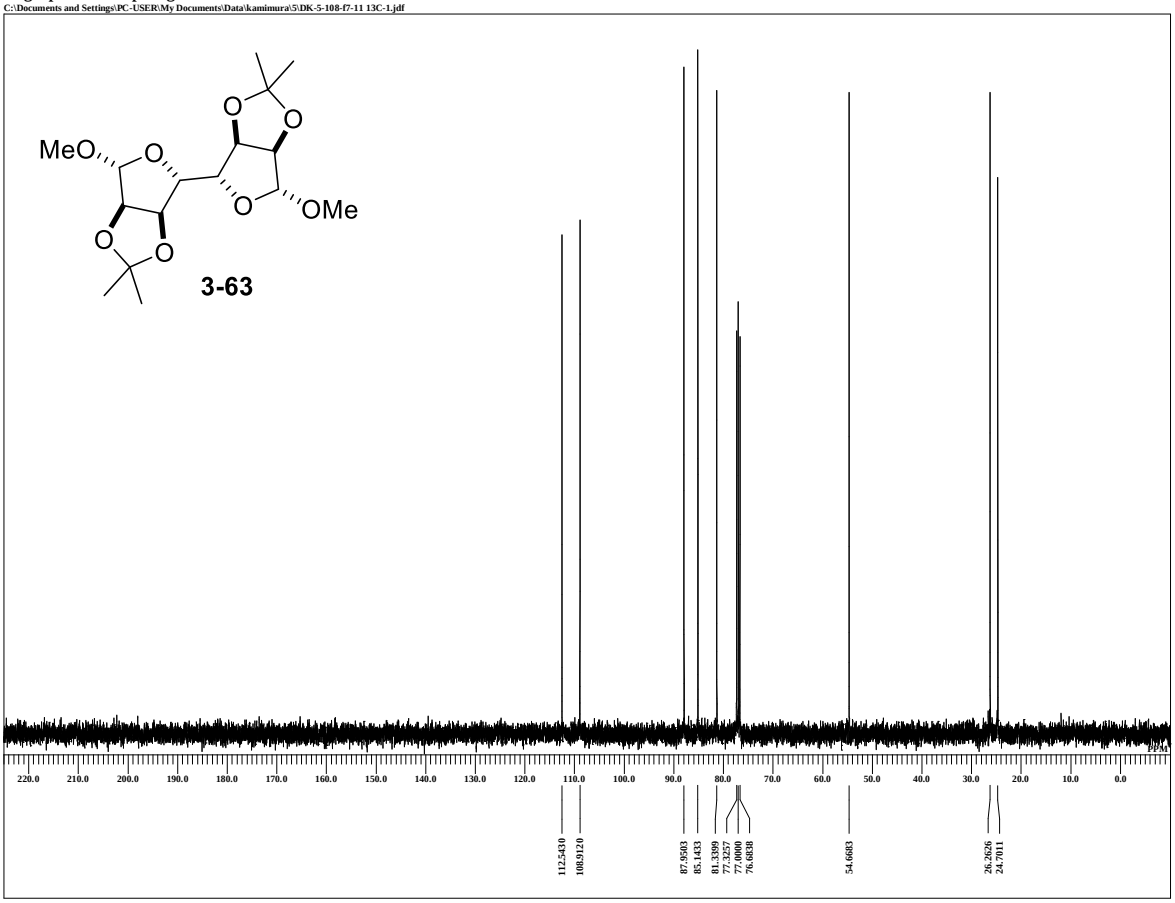
C:\Documents and Settings\PC-USER\ftfXfNfgfbf v\acyl telluride NMR data\49a 1H.als



DFILE 49a 1H.als
COMNT single_pulse
DATIM 10-01-2014 08:13:41
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 26
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 49a 1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-108-f7-11 13C-1.jdf

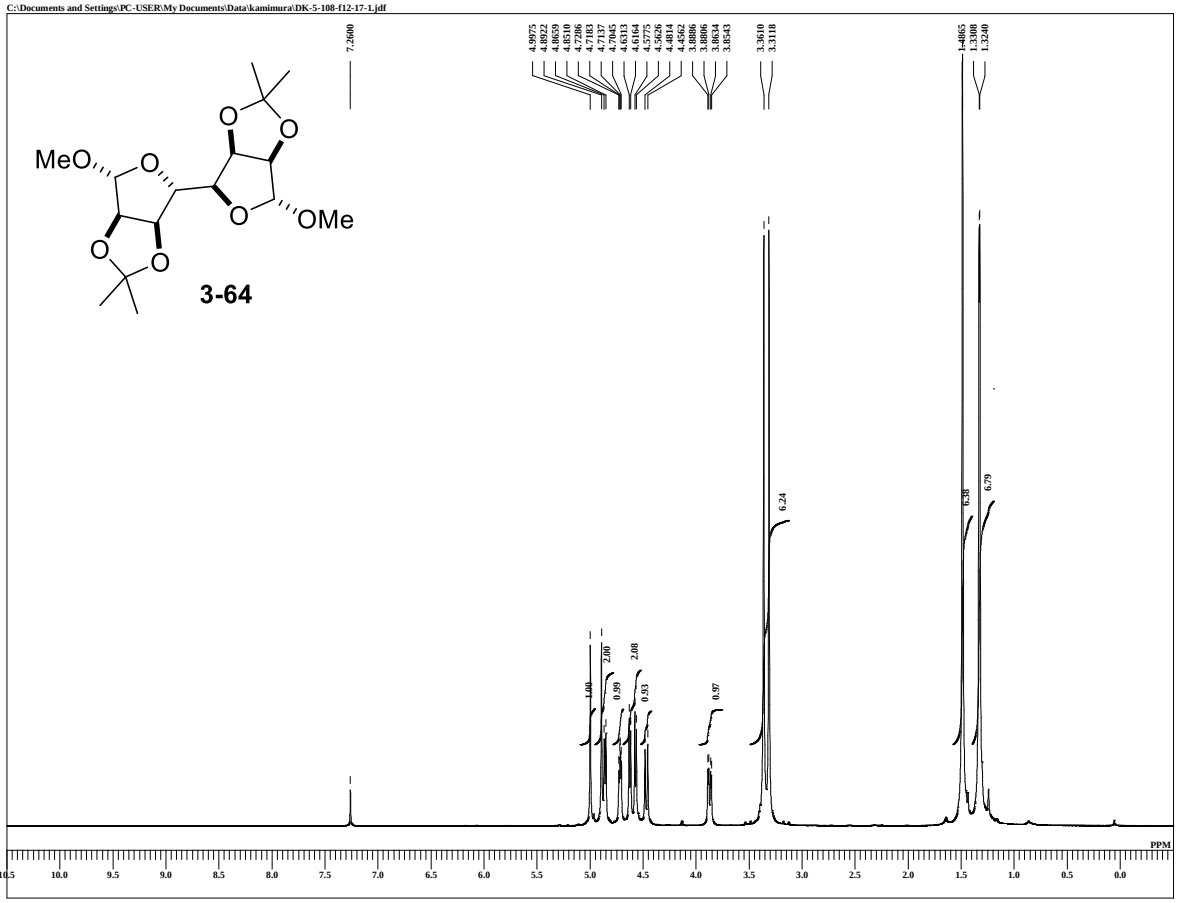


DFILE DK-5-108-f7-11 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 10-01-2014 08:16:21
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 39
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 39
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-108-f7-11 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

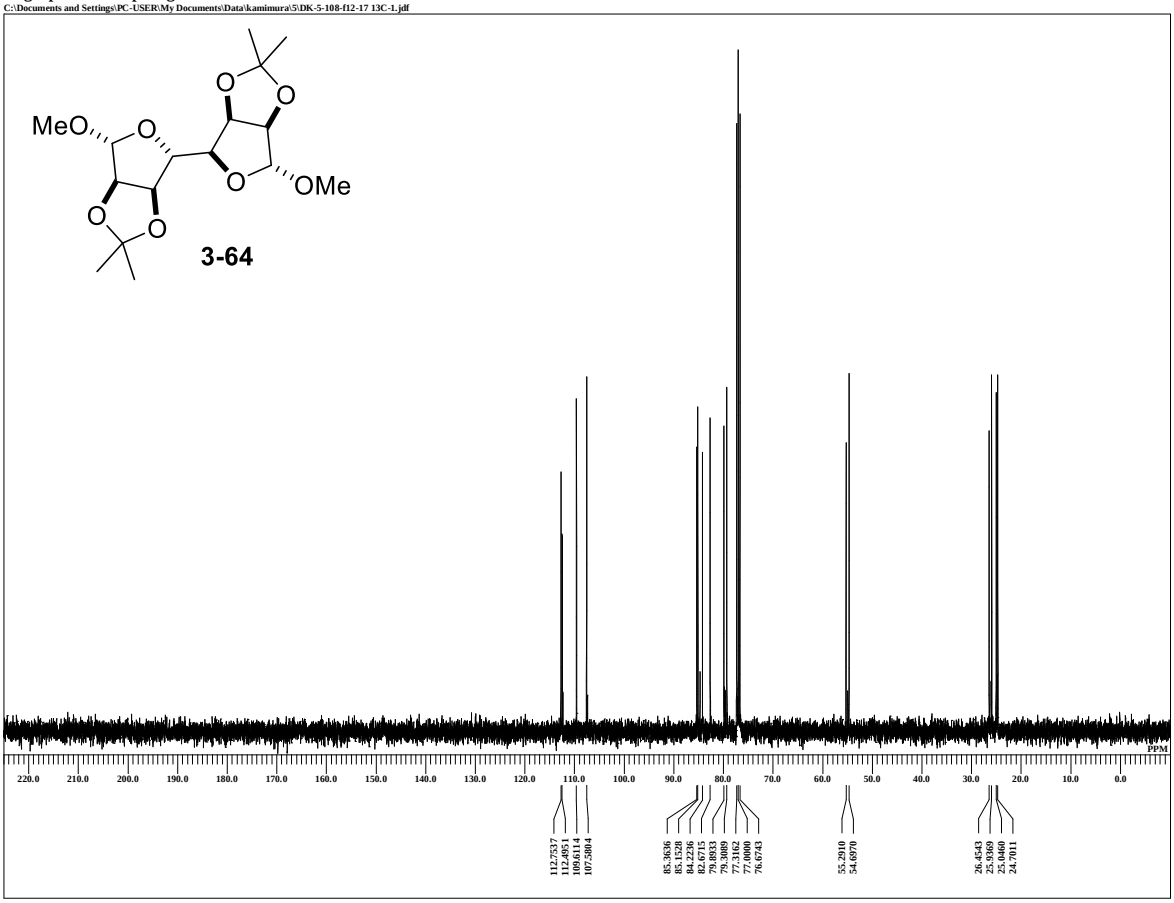
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-5-108-f12-17-1.jdf



DFILE	DK-5-108-f12-17-1.jdf
COMNT	single_pulse
DATIM	17-02-2014 16:41:33
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	30
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-108-f12-17-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-108-f12-17 13C-1.jdf

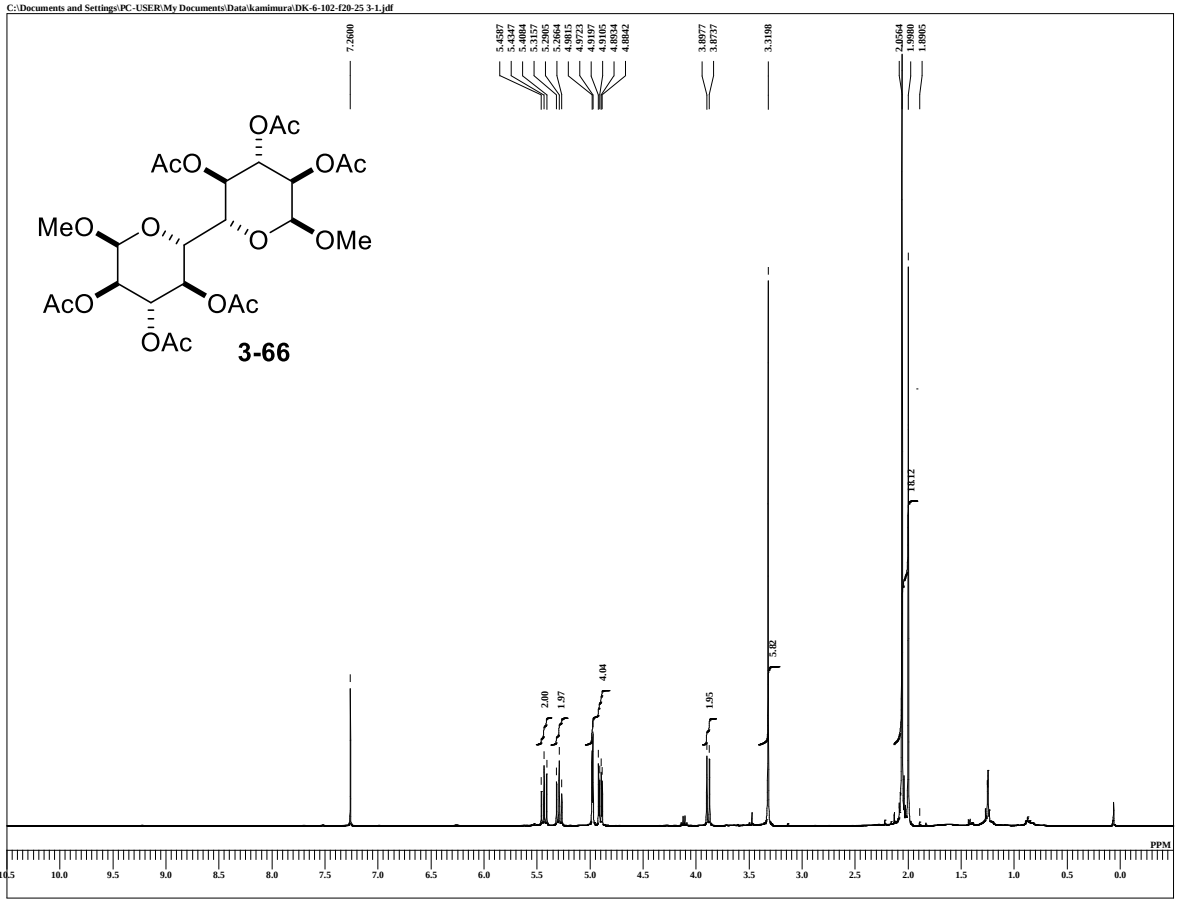


DFILE	DK-5-108-f12-17 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	10-01-2014 08:44:52
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	72
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	72
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-108-f12-17 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-102-f20-25 3-1.jdf

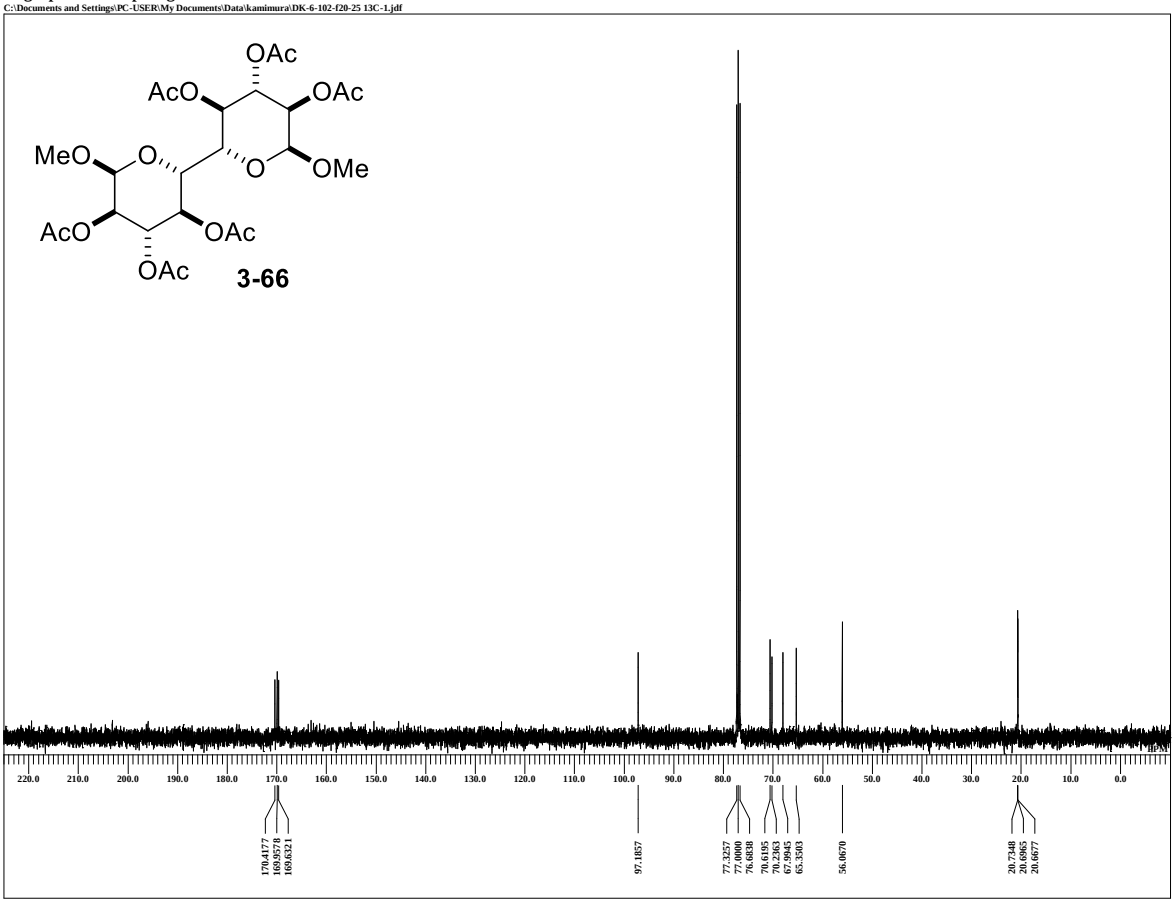


3-66

```
DFILE   DK-6-102-f20-25 3-1.jdf
COMNT   single_pulse
DATIM   05-02-2014 19:07:06
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.44 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT      16384
SPO        16384
TIMES          8
DUMMY          1
FREQU    7422.80 Hz
FLT       30000 Hz
DELAY      16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS          8
ADBIT         16
RGAIN         38
BF          0.10 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       115 usec
IRATN          79
DFILE   DK-6-102-f20-25 3-1.jdf
SF
LKSET      13.20 KHz
LKFIN       75.7 Hz
LKLEV          0
LGAIN          0
LKPHS          0
LKSIG          0
CSPED          0 Hz
FILDC
FILDF
CTEMP       21.3 c
SLVNT   CDCL3
EXREF       7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-102-f20-25 13C-1.jdf



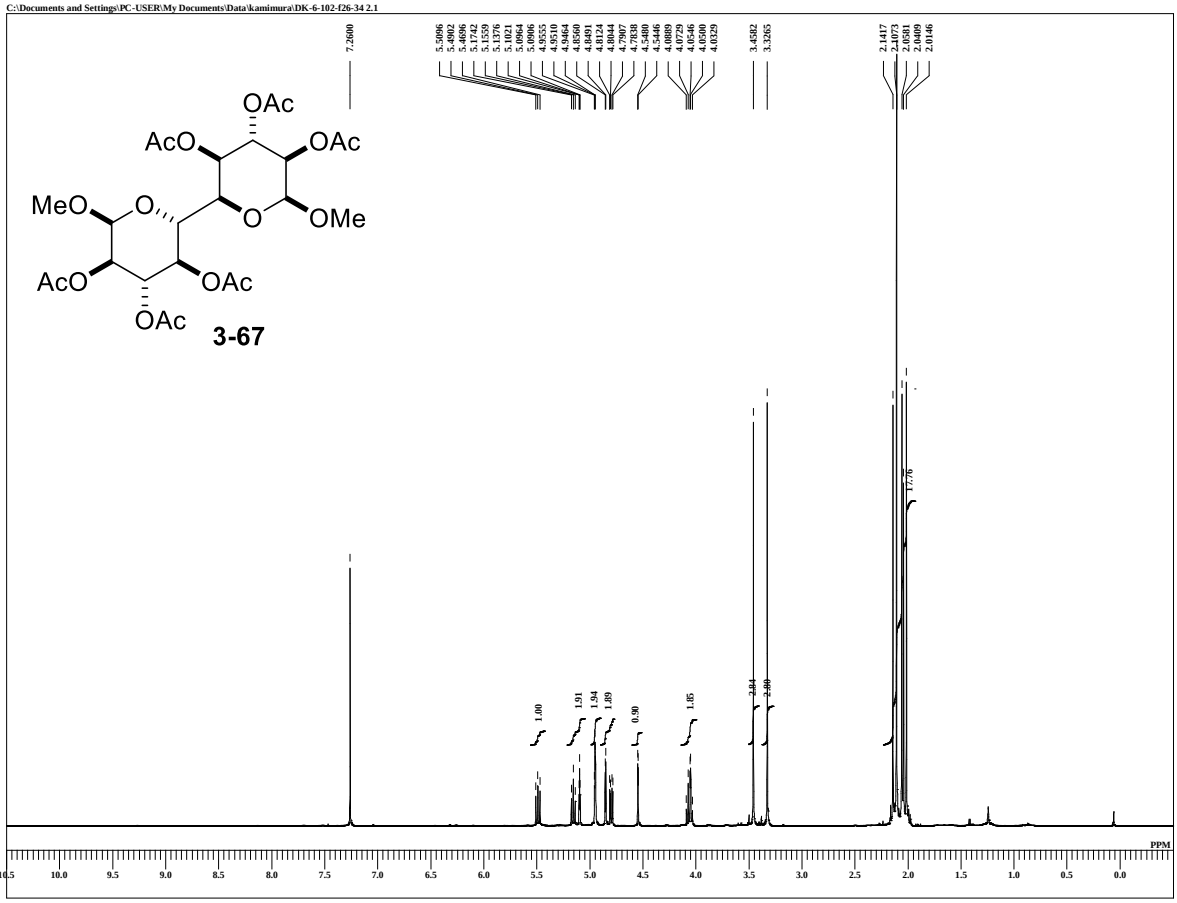
3-66

```
DFILE   DK-6-102-f20-25 13C-1.jc
COMNT   single pulse decoupled gat
DATIM   05-02-2014 15:37:59
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
PW1        3.03 usec
DEADT      0.00 usec
PREDL      0.00000 msec
IWT        1.0000 sec
POINT     32768
SPO       32768
TIMES       137
DUMMY         4
FREQU   31250.00 Hz
FLT      125000 Hz
DELAY     20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS       137
ADBIT        16
RGAIN        60
BF         1.00 Hz
T1         0.00
T2         0.00
T3       100.00
T4       100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET      6.28 KHz
IRFIN      0.87 Hz
IRRPW      115 usec
IRATN         79
DFILE   DK-6-102-f20-25 13C-1.jc
SF
LKSET     13.20 KHz
LKFIN      75.7 Hz
LKLEV         0
LGAIN         0
LKPHS         0
LKSIG         0
CSPED         0 Hz
FILDC
FILDF
CTEMP      21.2 c
SLVNT   CDCL3
EXREF      77.00 ppm
```

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-102-f26-34 2.1



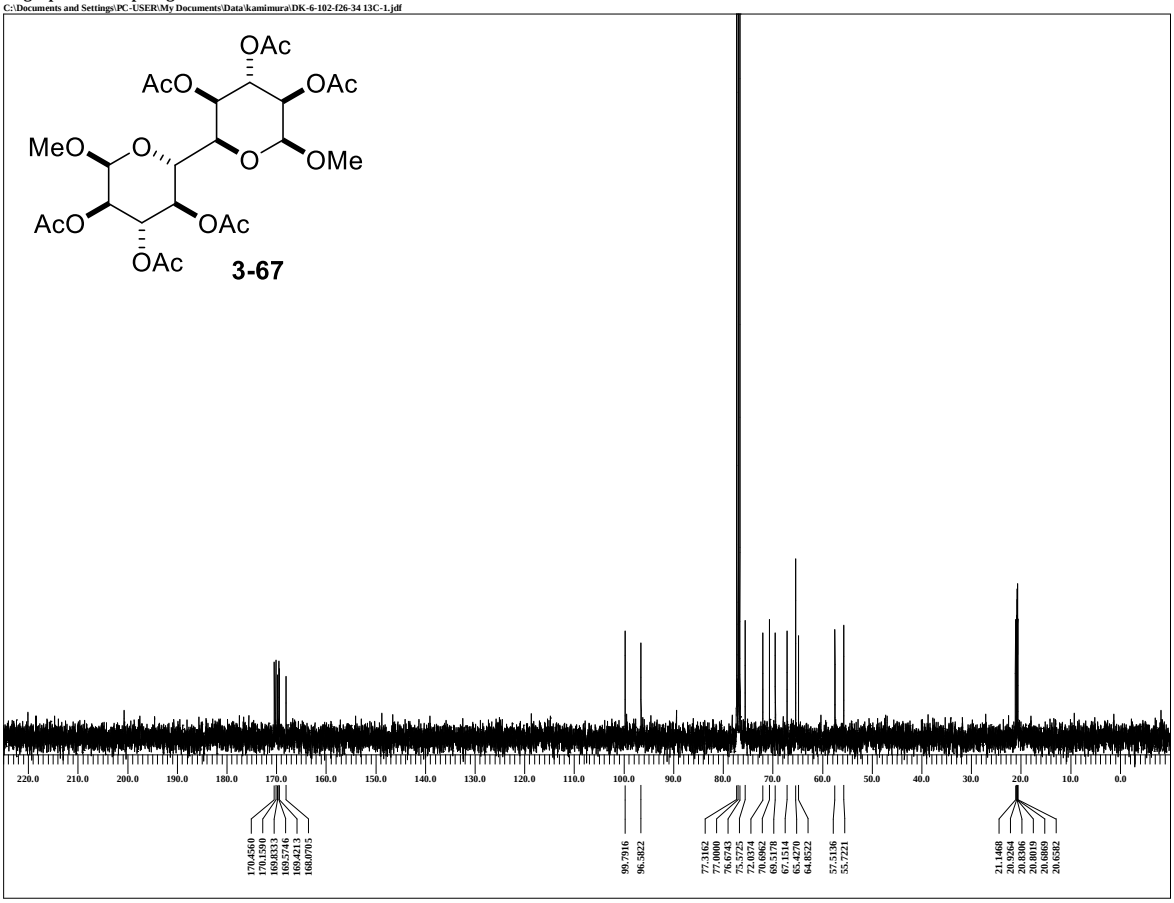
OAc
AcO
OAc
MeO
O
O
OMe
AcO
OAc
OAc

3-67

DFILE	DK-6-102-f26-34 2.1
COMNT	single_pulse
DATIM	05-02-2014 14:18:45
MENUF	
OBNUC	1H
OFR	495.13 MHz
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
PW1	6.00 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	9286.78 Hz
FLT	38000 Hz
DELAY	13.16 usec
ACQTM	1.7642 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	48
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-6-102-f26-34 2.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-102-f26-34 13C-1.jdf



OAc
AcO
OAc
MeO
O
O
OMe
AcO
OAc
OAc

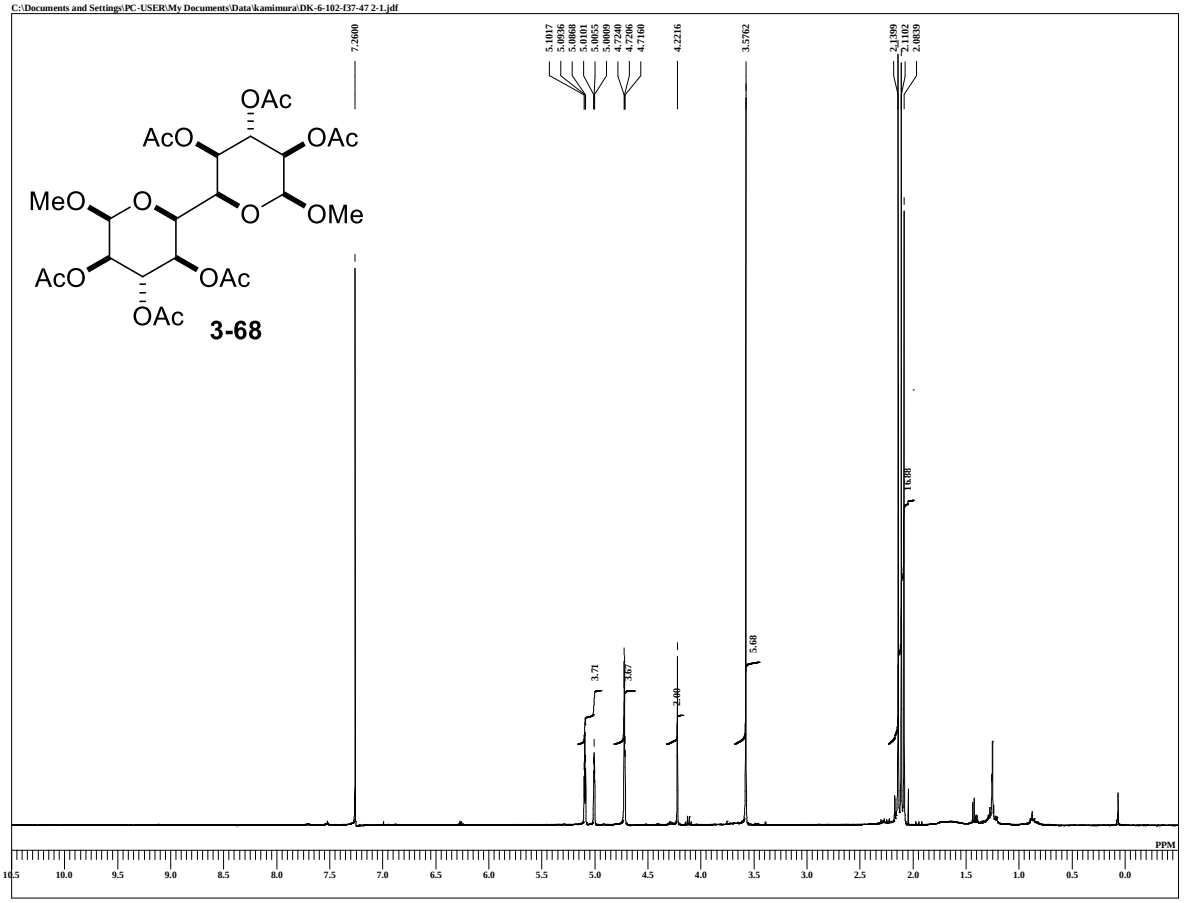
3-67

DFILE	DK-6-102-f26-34 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	05-02-2014 11:50:50
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	164
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	164
ADBIT	16
RGAIN	58
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-102-f26-34 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

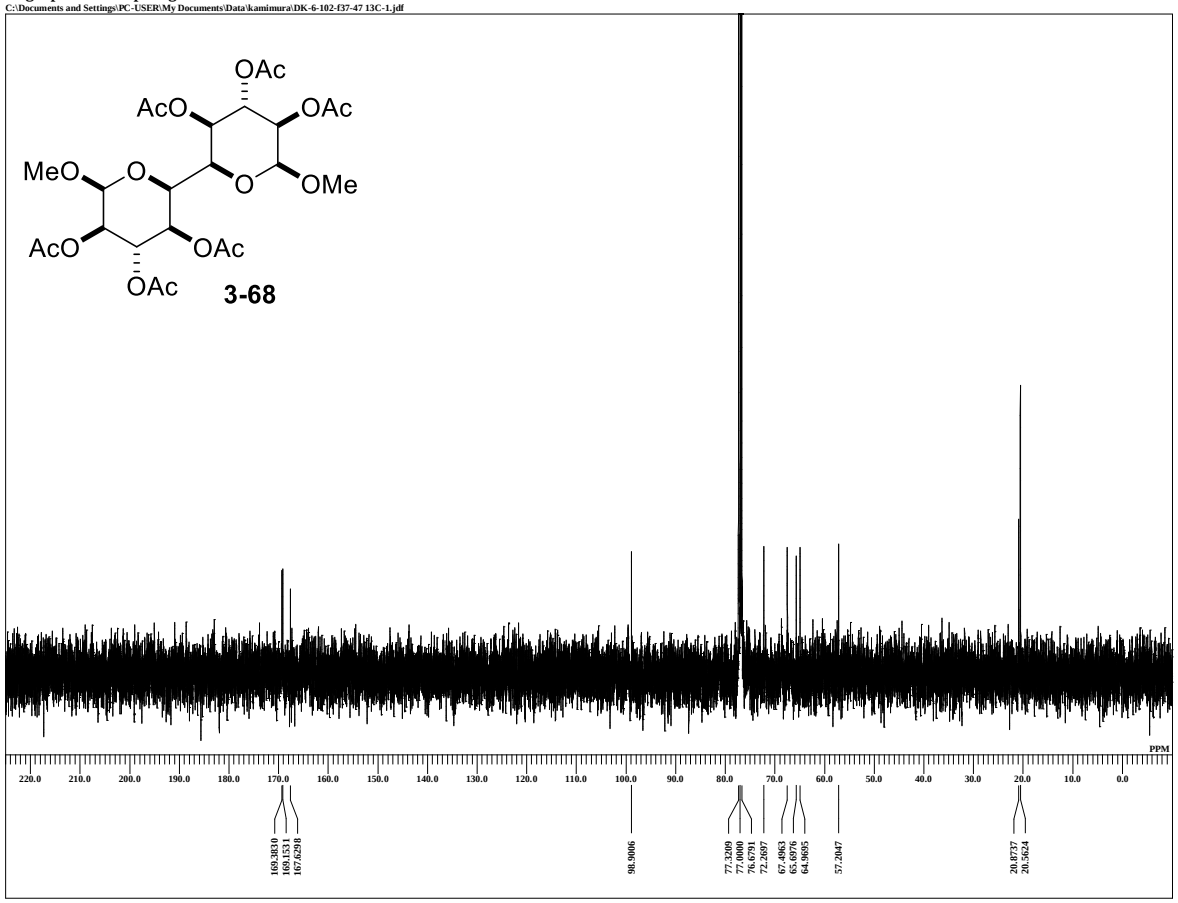
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-102-f37-47 2-1.jdf




```
DFILE    DK-6-102-f37-47 2-1.jdf
COMNT    single_pulse
DATIM    06-02-2014 09:55:41
MENUF
OBNUC    1H
OFR        395.88 MHz
OBFRQ      395.88 MHz
OBSET        6.28 KHz
OBFIN        0.87 Hz
PW1          6.44 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        16384
SPO          16384
TIMES            8
DUMMY            1
FREQU     7422.80 Hz
FLT         30000 Hz
DELAY       16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS            8
ADBIT           16
RGAIN           46
BF           0.10 Hz
T1           0.00
T2           0.00
T3         100.00
T4         100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW       115 usec
IRATN           79
DFILE    DK-6-102-f37-47 2-1.jdf
SF
LKSET       13.20 KHz
LKFIN        75.7 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED        0 Hz
FILDC
FILDF
CTEMP       21.1 c
SLVNT    CDCL3
EXREF       7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-102-f37-47 13C-1.jdf

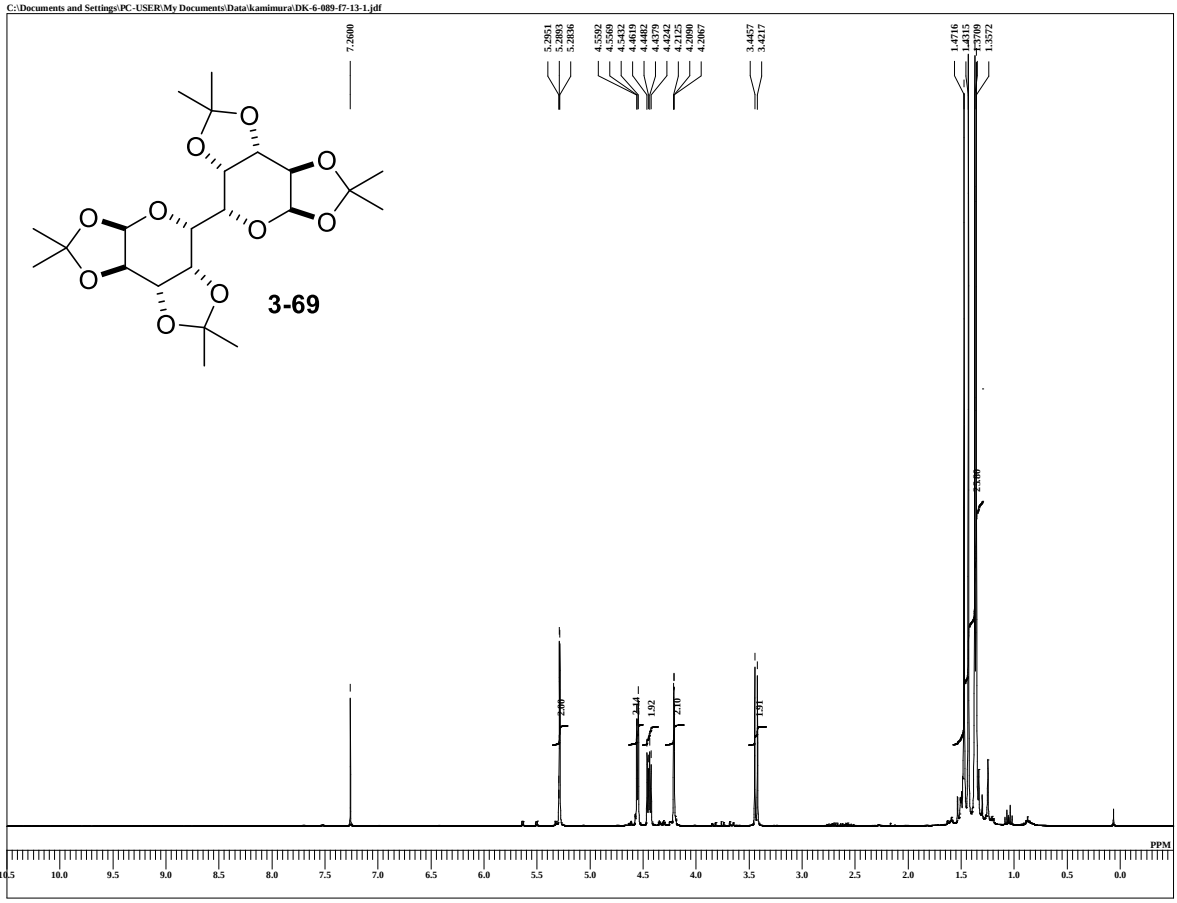



```
DFILE    DK-6-102-f37-47 13C-1.jd
COMNT    single pulse decoupled gat
DATIM    06-02-2014 10:07:04
MENUF
OBNUC    13C
OFR        99.55 MHz
OBFRQ      99.55 MHz
OBSET        5.13 KHz
OBFIN        0.98 Hz
PW1          3.03 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT      131072
SPO        131072
TIMES          212
DUMMY            4
FREQU     31250.00 Hz
FLT        125000 Hz
DELAY       20.50 usec
ACQTM       1.0486 sec
PD          2.0000 sec
SCANS          212
ADBIT           16
RGAIN           60
BF           1.00 Hz
T1           0.00
T2           0.00
T3         100.00
T4         100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW       115 usec
IRATN           79
DFILE    DK-6-102-f37-47 13C-1.jd
SF
LKSET       13.20 KHz
LKFIN        75.7 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED        0 Hz
FILDC
FILDF
CTEMP       21.2 c
SLVNT    CDCL3
EXREF       77.00 ppm
```

スペクトルデータ

single_pulse

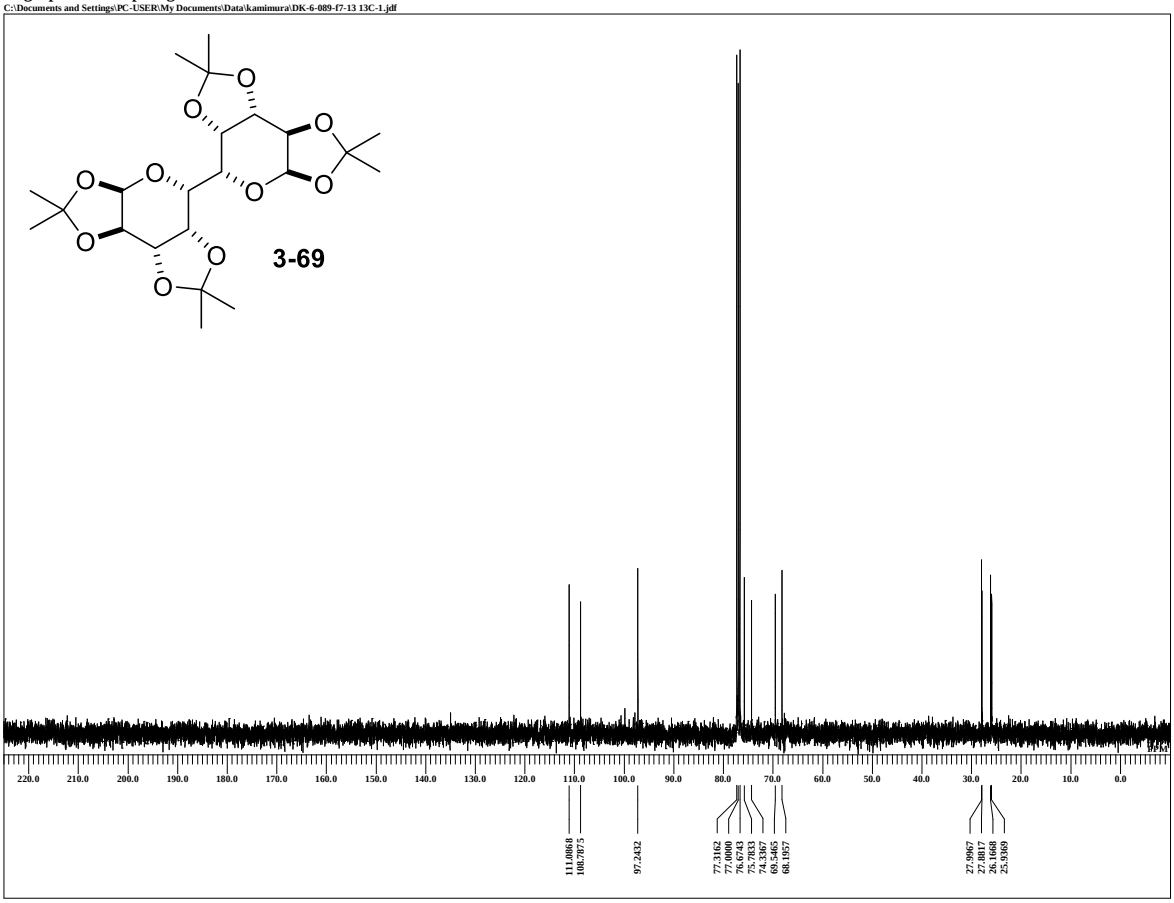
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-089-f7-13-1.jdf



DFILE	DK-6-089-f7-13-1.jdf
COMNT	single_pulse
DATIM	22-01-2014 11:12:28
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-089-f7-13-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-089-f7-13 13C-1.jdf

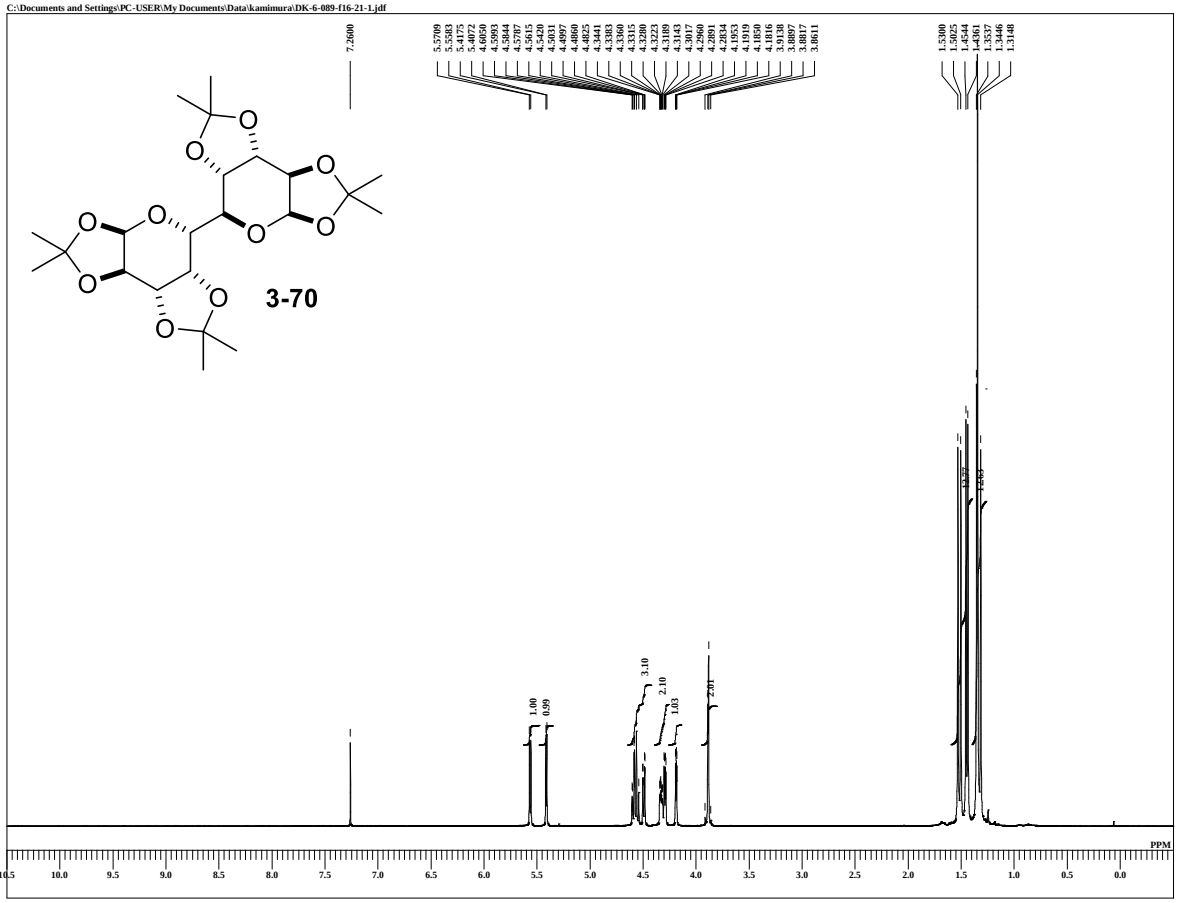


DFILE	DK-6-089-f7-13 13C-1.jdf
COMNT	single pulse decoupled gat
DATIM	22-01-2014 11:17:26
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	86
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	86
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-089-f7-13 13C-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

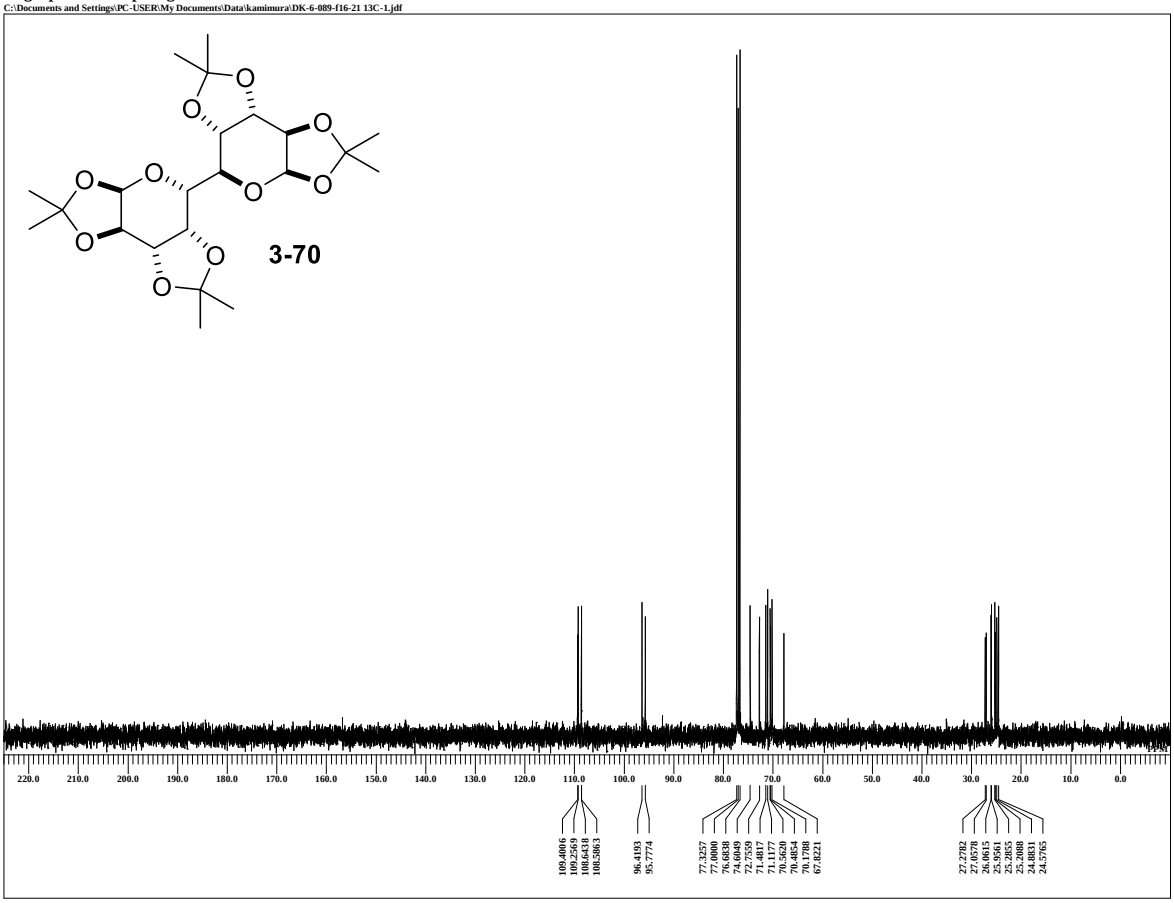
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-089-f16-21-1.jdf



DFILE DK-6-089-f16-21-1.jdf
COMNT single_pulse
DATIM 22-01-2014 10:06:38
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 32
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-6-089-f16-21-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-089-f16-21 13C-1.jdf

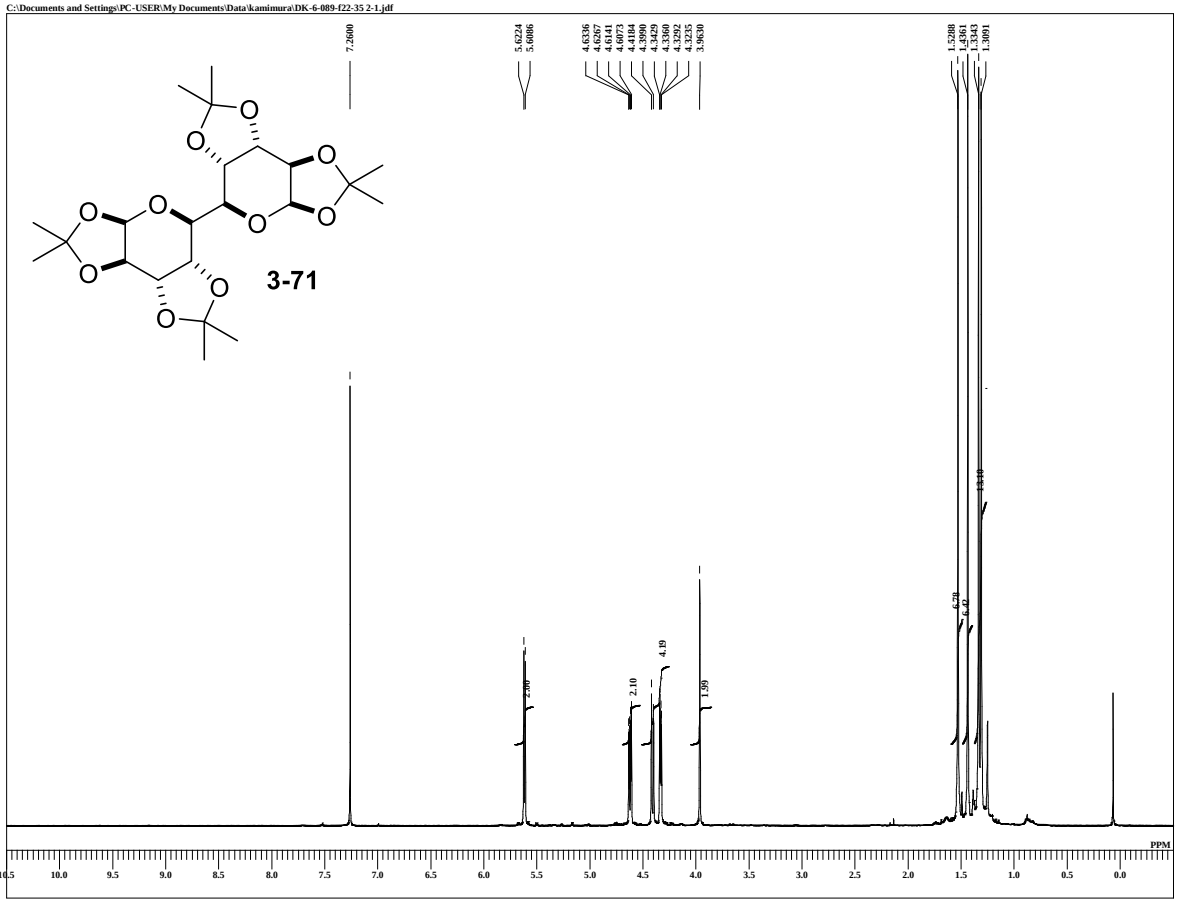


DFILE DK-6-089-f16-21 13C-1.jd
COMNT single pulse decoupled gat
DATIM 22-01-2014 10:12:21
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 101
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 101
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-089-f16-21 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

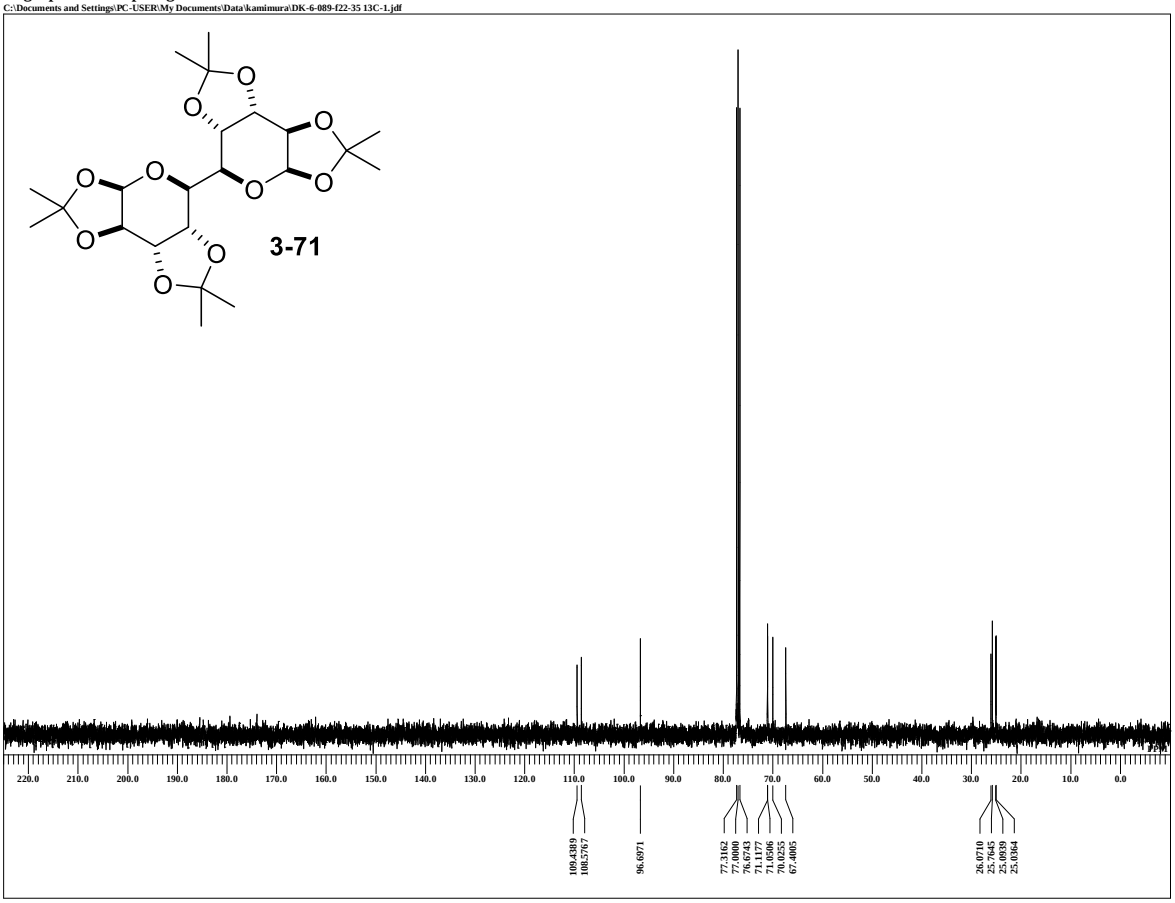
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-089-f22-35 2-1.jdf



DFILE	DK-6-089-f22-35 2-1.jdf
COMNT	single_pulse
DATIM	22-01-2014 15:30:50
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	40
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-089-f22-35 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-089-f22-35 13C-1.jdf

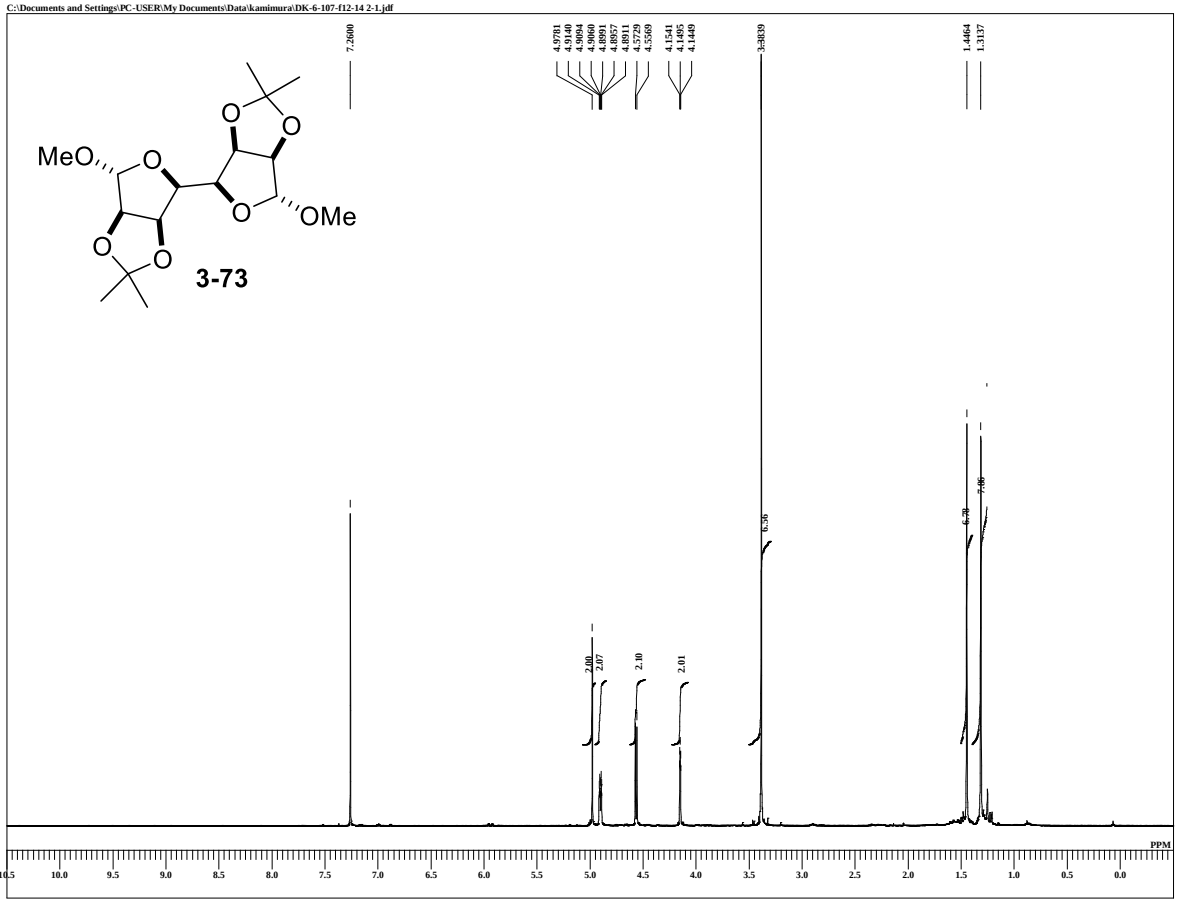


DFILE	DK-6-089-f22-35 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	22-01-2014 13:02:18
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	101
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	101
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-6-089-f22-35 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

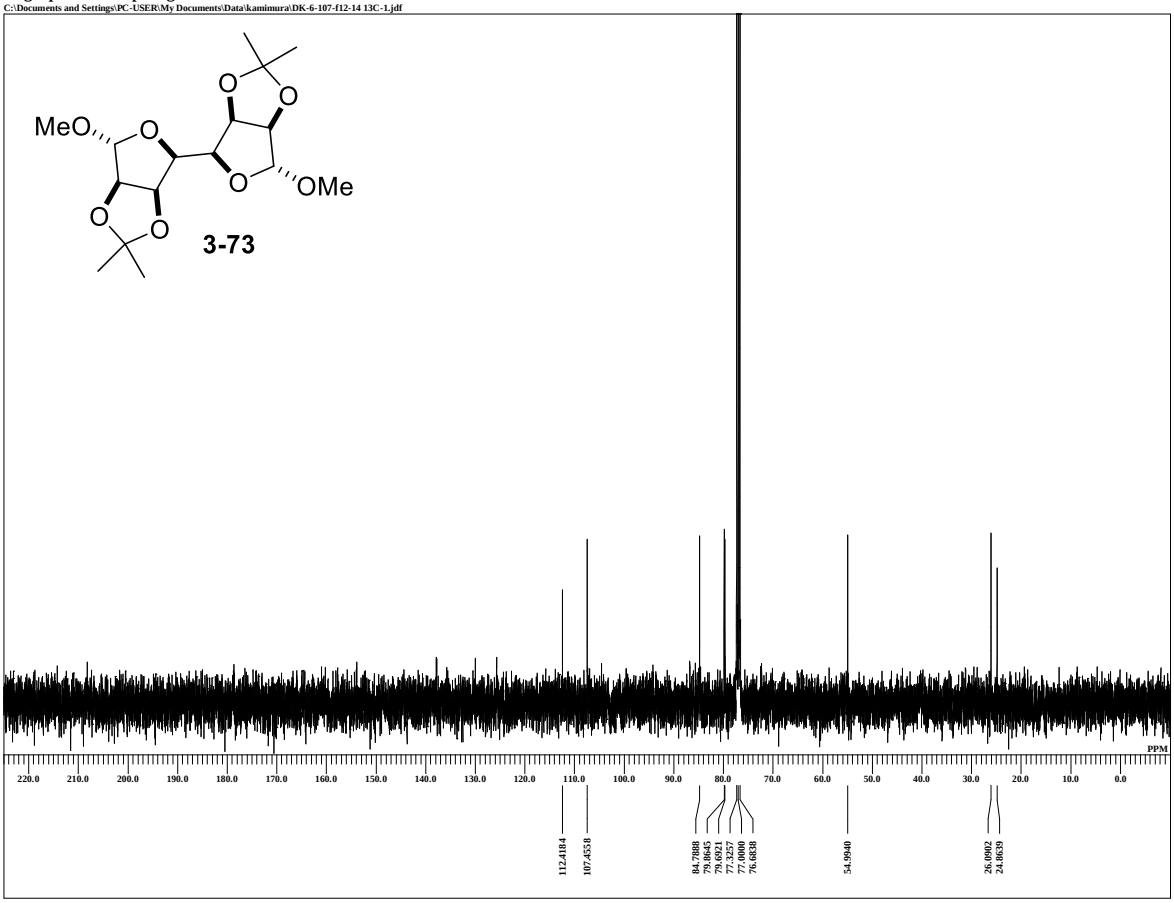
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-107-f12-14 2-1.jdf



DFILE DK-6-107-f12-14 2-1.jdf
COMNT single_pulse
DATIM 17-02-2014 14:51:40
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 48
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-107-f12-14 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.7 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-107-f12-14 13C-1.jdf

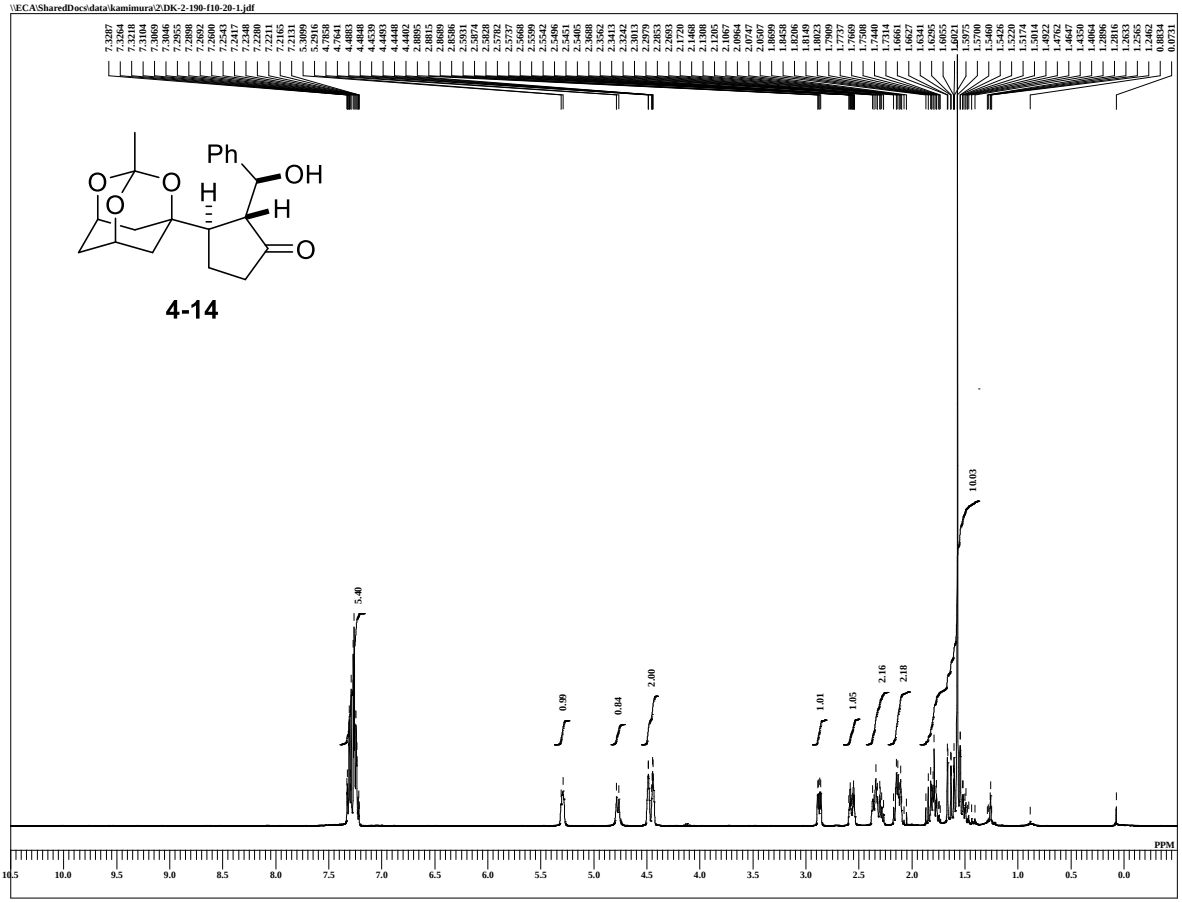


DFILE DK-6-107-f12-14 13C-1.jd
COMNT single pulse decoupled gat
DATIM 17-02-2014 14:59:56
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 151
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 151
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-107-f12-14 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

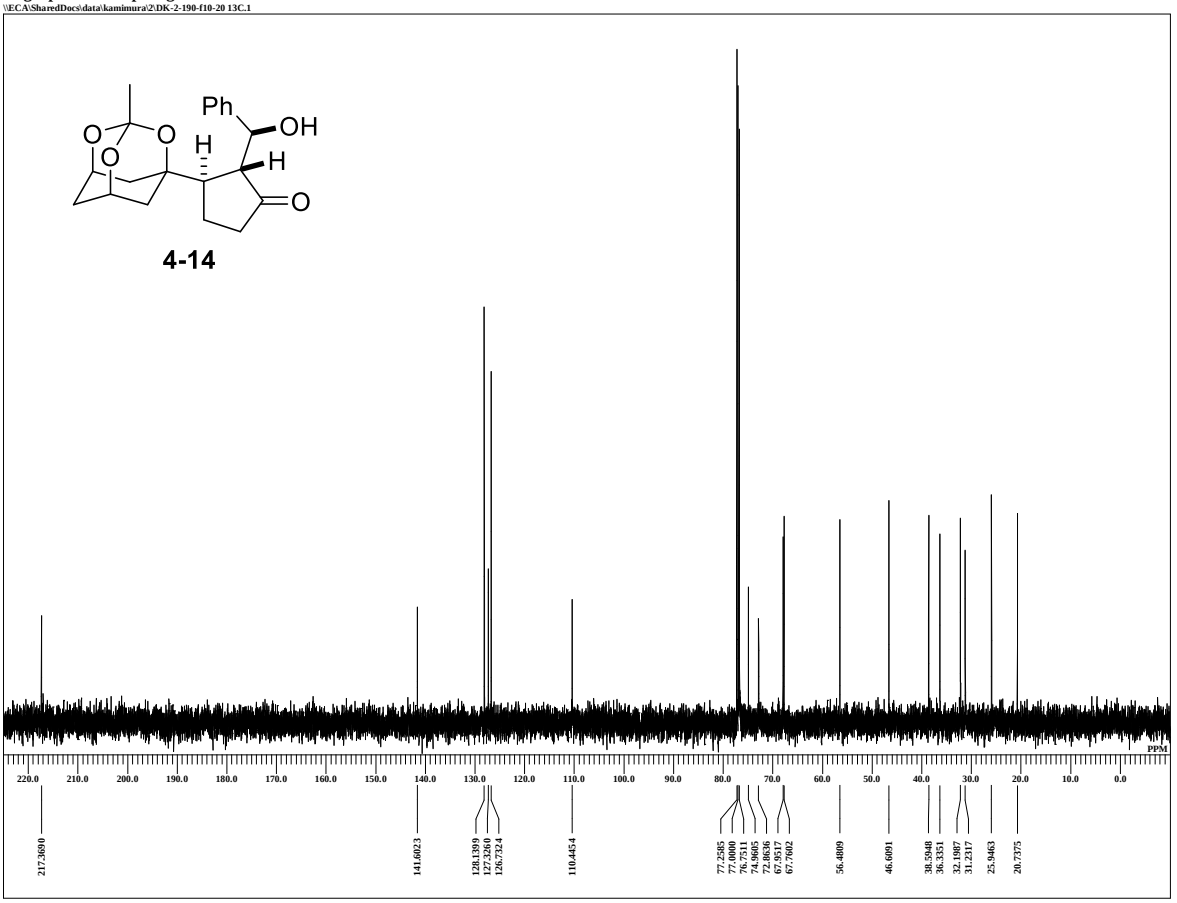
\\ECA\SharedDocs\data\kamimura\2\DK-2-190-f10-20-1.jdf



DFILE	DK-2-190-f10-20-1.jdf
COMNT	single_pulse
DATIM	15-12-2012 12:54:45
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	38
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-2-190-f10-20-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-190-f10-20 13C.1

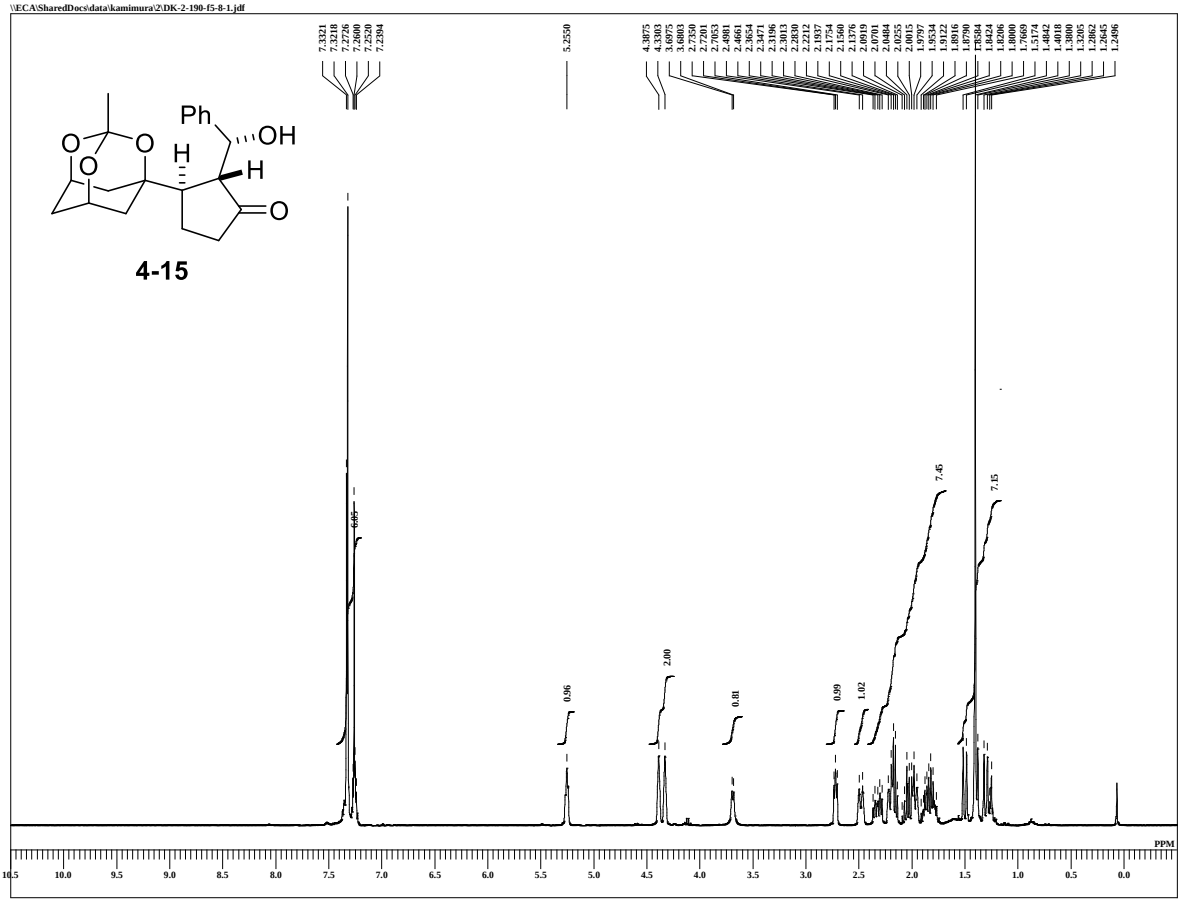


DFILE	DK-2-190-f10-20 13C.1
COMNT	single pulse decoupled gat
DATIM	13-11-2012 19:24:17
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	76
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	76
ADBIT	16
RGAIN	48
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-2-190-f10-20 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

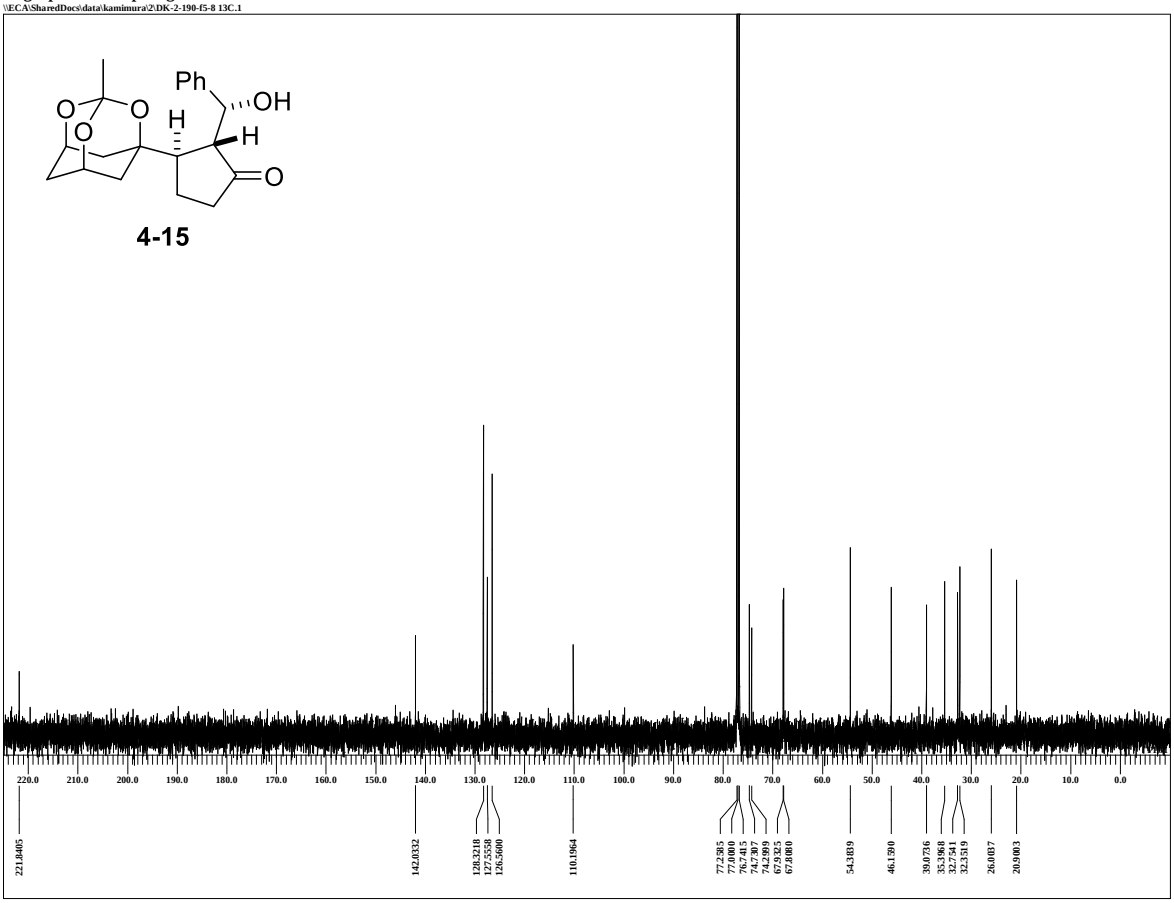
\\ECA\SharedDocs\data\kamimura\2\DK-2-190-f5-8-1.jdf




```
DFILE     DK-2-190-f5-8-1.jdf
COMNT     single_pulse
DATIM     19-12-2012 18:05:56
MENUF
OBNUC     1H
OFR         395.88 MHz
OBFRQ       395.88 MHz
OBSET         6.28 KHz
OBFIN         0.87 Hz
PW1           6.38 usec
DEADT         0.00 usec
PREDL      0.00000 msec
IWT           1.0000 sec
POINT        16384
SPO          16384
TIMES           16
DUMMY            1
FREQU      7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM         2.2073 sec
PD            2.0000 sec
SCANS           16
ADBIT           16
RGAIN           42
BF            0.01 Hz
T1            0.00
T2            0.00
T3          100.00
T4          100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR         395.88 MHz
IRSET         6.28 KHz
IRFIN         0.87 Hz
IRRPW          147 usec
IRATN           79
DFILE     DK-2-190-f5-8-1.jdf
SF
LKSET        13.20 KHz
LKFIN         75.7 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED            0 Hz
FILDC
FILDF
CTEMP         20.5 c
SLVNT     CDCL3
EXREF         7.26 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-190-f5-8 13C.1

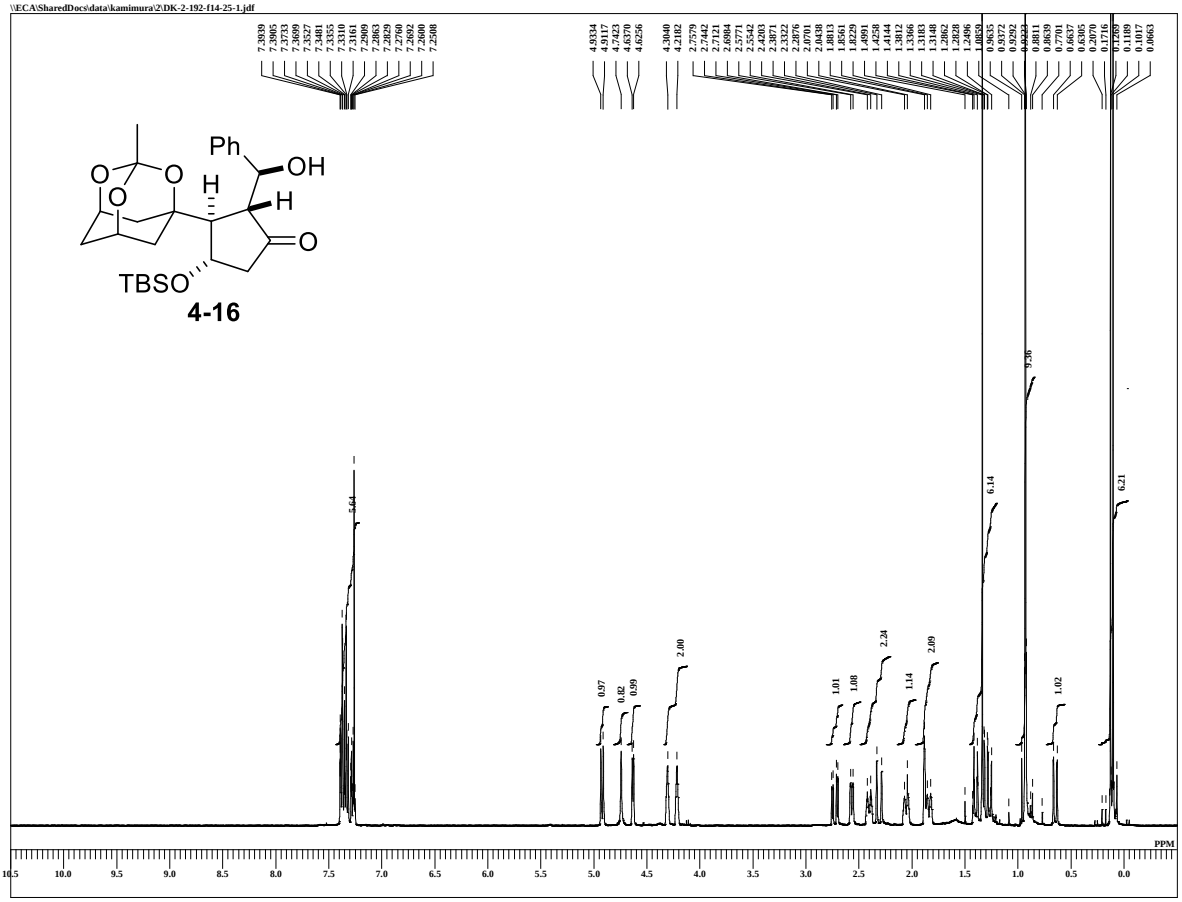



```
DFILE     DK-2-190-f5-8 13C.1
COMNT     single pulse decoupled gat
DATIM     13-11-2012 19:15:12
MENUF
OBNUC     13C
OFR         124.51 MHz
OBFRQ       124.51 MHz
OBSET         3.45 KHz
OBFIN         6.00 Hz
PW1           3.70 usec
DEADT         0.00 usec
PREDL      0.00000 msec
IWT           1.0000 sec
POINT        32768
SPO          32768
TIMES          167
DUMMY            4
FREQU      39062.50 Hz
FLT          157000 Hz
DELAY        20.80 usec
ACQTM         0.8389 sec
PD            2.0000 sec
SCANS          167
ADBIT           16
RGAIN           50
BF            1.00 Hz
T1            0.00
T2            0.00
T3          100.00
T4          100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR         495.13 MHz
IRSET         4.38 KHz
IRFIN         9.64 Hz
IRRPW          92 usec
IRATN           79
DFILE     DK-2-190-f5-8 13C.1
SF
LKSET        748.40 KHz
LKFIN         98.2 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED            0 Hz
FILDC
FILDF
CTEMP         23.5 c
SLVNT     CDCL3
EXREF         77.00 ppm
```

スペクトルデータ

single_pulse

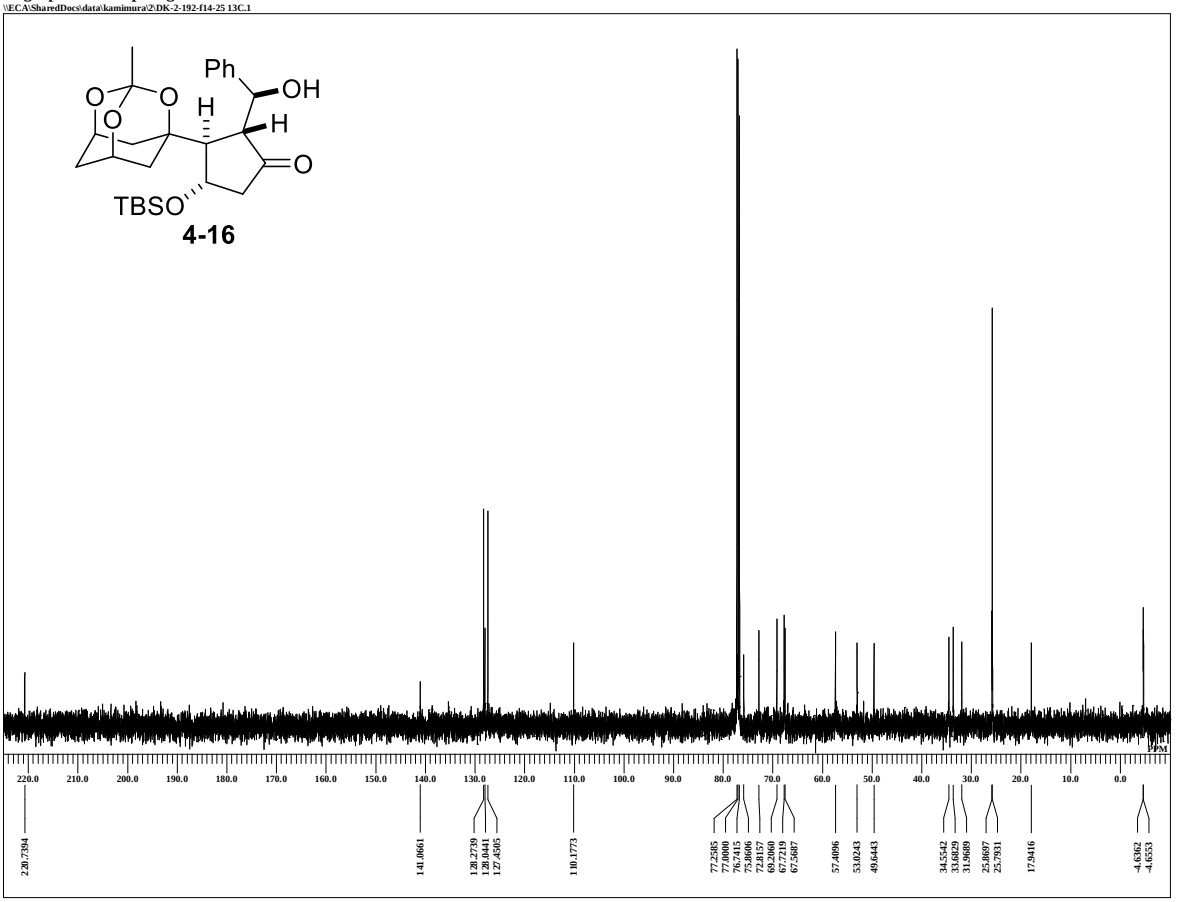
\\ECA\SharedDocs\data\kamimura\2\DK-2-192-f14-25-1.jdf



DFILE DK-2-192-f14-25-1.jdf
COMNT single_pulse
DATIM 20-12-2012 18:44:11
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.38 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 16
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 16
ADBIT 16
RGAIN 38
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-2-192-f14-25-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.8 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-192-f14-25 13C.1

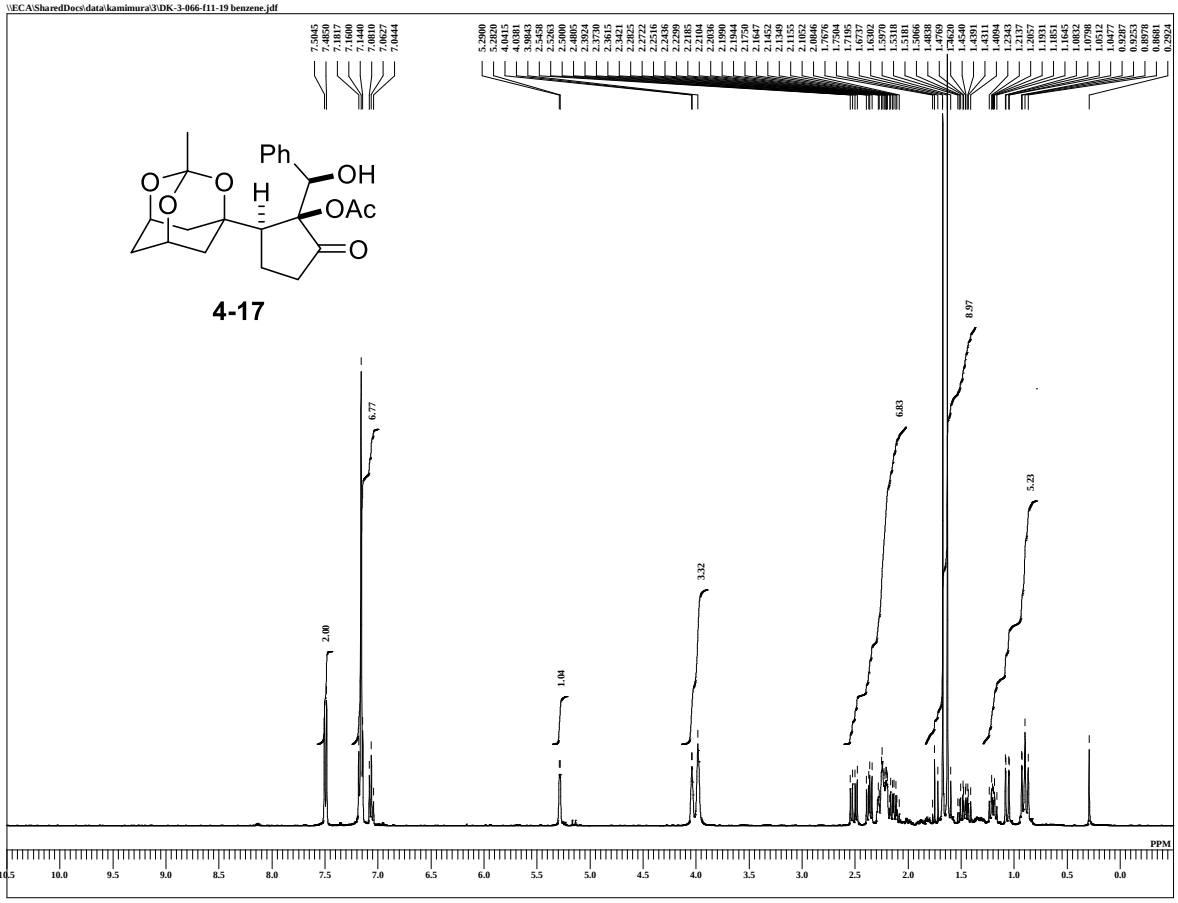


DFILE DK-2-192-f14-25 13C.1
COMNT single pulse decoupled gat
DATIM 14-11-2012 11:38:48
MENUF
OBNUC 13C
OFR 124.51 MHz
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
PW1 3.70 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 112
DUMMY 4
FREQU 39062.50 Hz
FLT 157000 Hz
DELAY 20.80 usec
ACQTM 0.8389 sec
PD 2.0000 sec
SCANS 112
ADBIT 16
RGAIN 48
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE DK-2-192-f14-25 13C.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

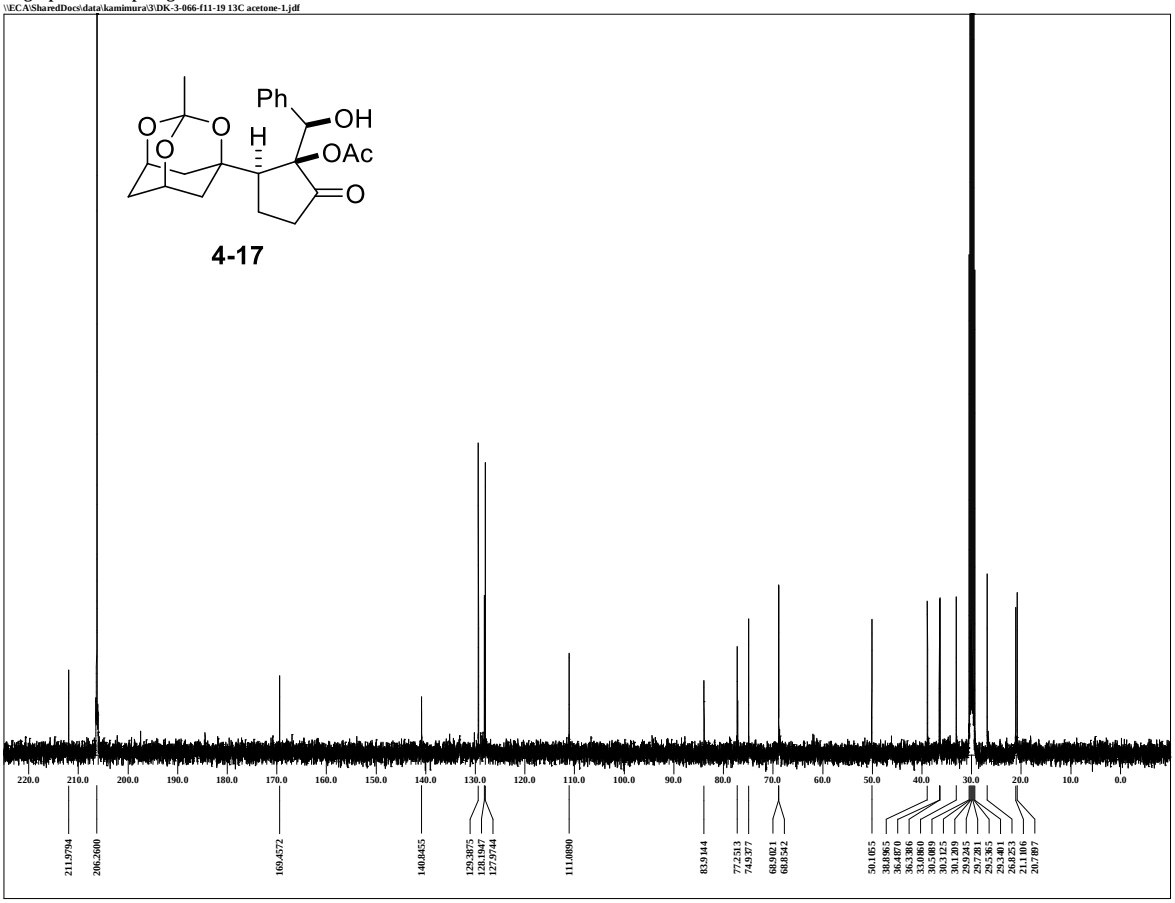
\\ECA\SharedDocs\data\kamimura\3\DK-3-066-f11-19 benzene.jdf




```
DFILE    DK-3-066-f11-19 benzene
COMNT    single_pulse
DATIM    11-03-2013 10:48:35
MENUF
OBNUC    1H
OFR        395.88 MHz
OBFRQ      395.88 MHz
OBSET        6.28 KHz
OBFIN        0.87 Hz
PW1          6.38 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        16384
SPO          16384
TIMES        8
DUMMY        1
FREQU      7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM        2.2073 sec
PD           2.0000 sec
SCANS        8
ADBIT        16
RGAIN        36
BF           0.01 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-3-066-f11-19 benzene
SF
LKSET        13.20 KHz
LKFIN        69.6 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        20.4 c
SLVNT    C6D6
EXREF        7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-066-f11-19 13C acetone-1.jdf

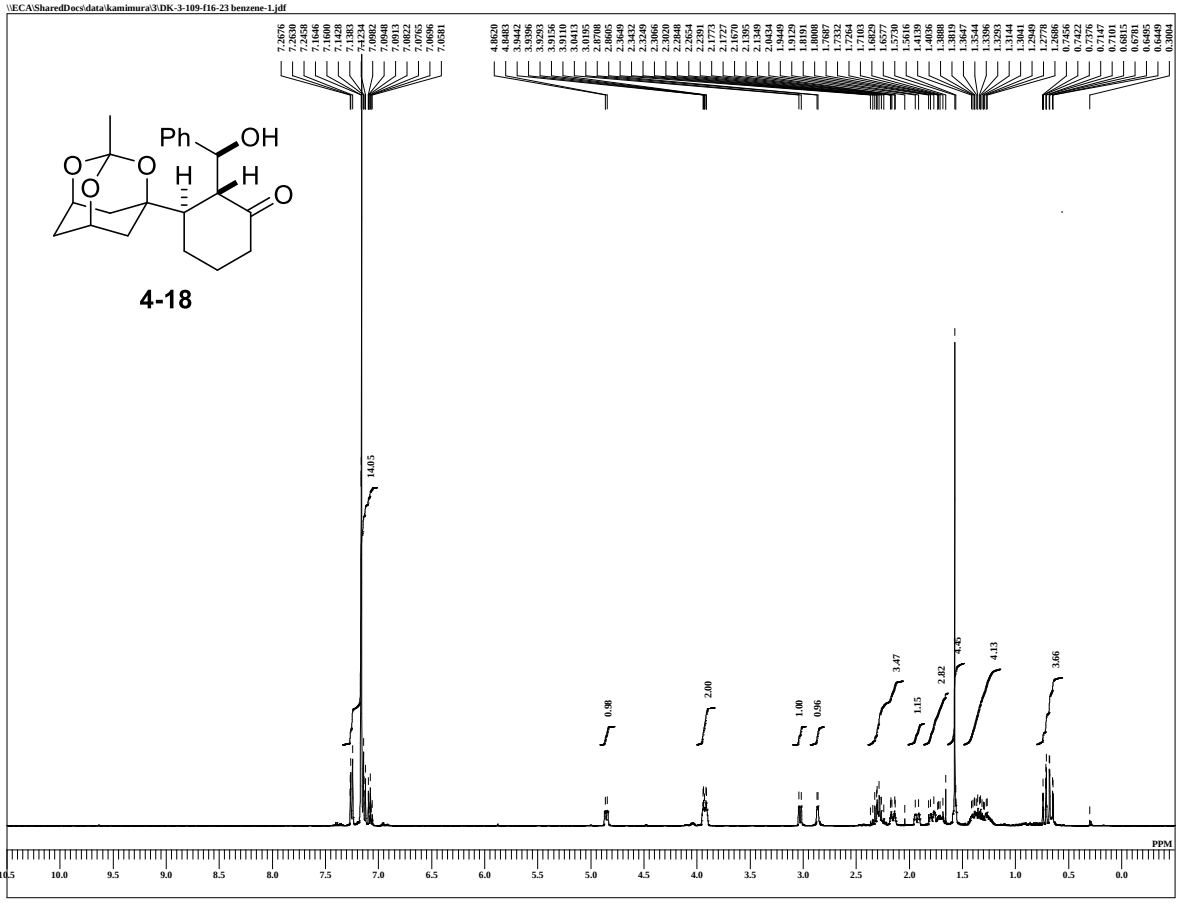



```
DFILE    DK-3-066-f11-19 13C ace
COMNT    single pulse decoupled gat
DATIM    11-03-2013 13:44:08
MENUF
OBNUC    13C
OFR        99.55 MHz
OBFRQ      99.55 MHz
OBSET        5.13 KHz
OBFIN        0.98 Hz
PW1          3.25 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        65536
SPO          65536
TIMES        212
DUMMY        4
FREQU      31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM        1.0486 sec
PD           2.0000 sec
SCANS        212
ADBIT        16
RGAIN        60
BF           1.00 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-3-066-f11-19 13C ace
SF
LKSET        12.90 KHz
LKFIN        59.1 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        20.7 c
SLVNT    ACETN
EXREF        206.26 ppm
```

スペクトルデータ

single_pulse

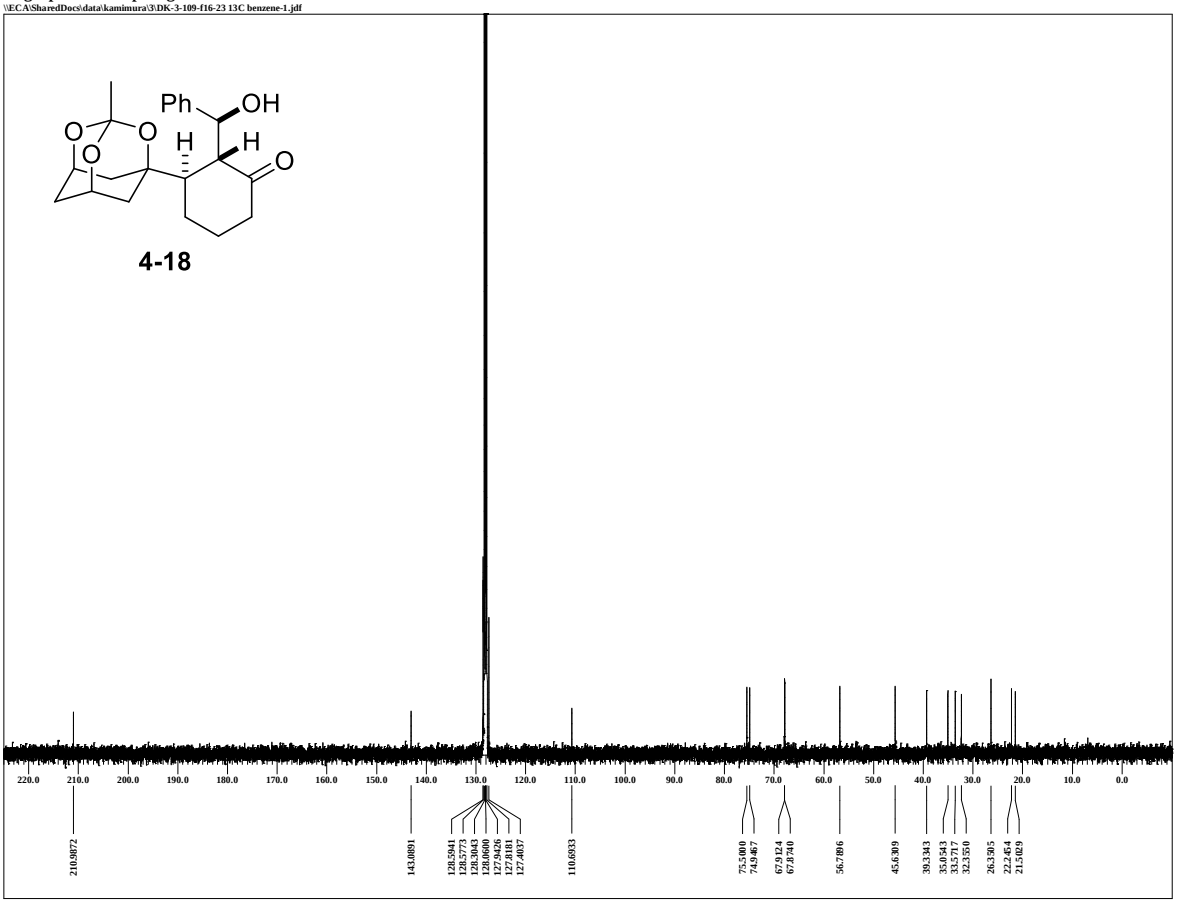
\\ECA\SharedDocs\data\kamimura\3\DK-3-109-f16-23 benzene-1.jdf




```
DFILE    DK-3-109-f16-23 benzene
COMNT    single_pulse
DATIM    07-02-2013 11:22:21
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.38 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    32
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    32
ADBIT    16
RGAIN    38
BF       0.01 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-3-109-f16-23 benzene
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.6 c
SLVNT    C6D6
EXREF    7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-109-f16-23 13C benzene-1.jdf

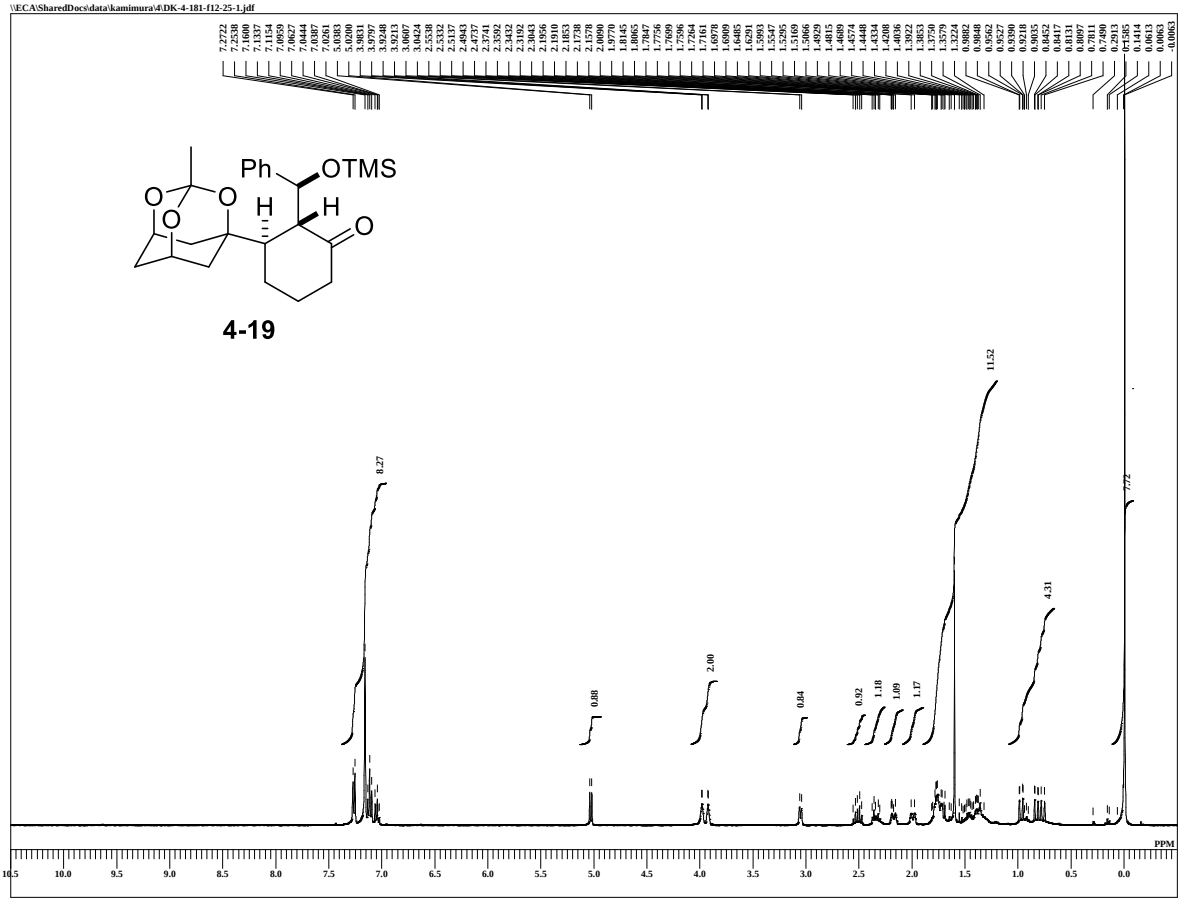



```
DFILE    DK-3-109-f16-23 13C ben
COMNT    single pulse decoupled gat
DATIM    07-02-2013 11:33:04
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.25 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    131072
SPO      131072
TIMES    196
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    196
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-3-109-f16-23 13C ben
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.8 c
SLVNT    C6D6
EXREF    128.06 ppm
```

スペクトルデータ

single_pulse

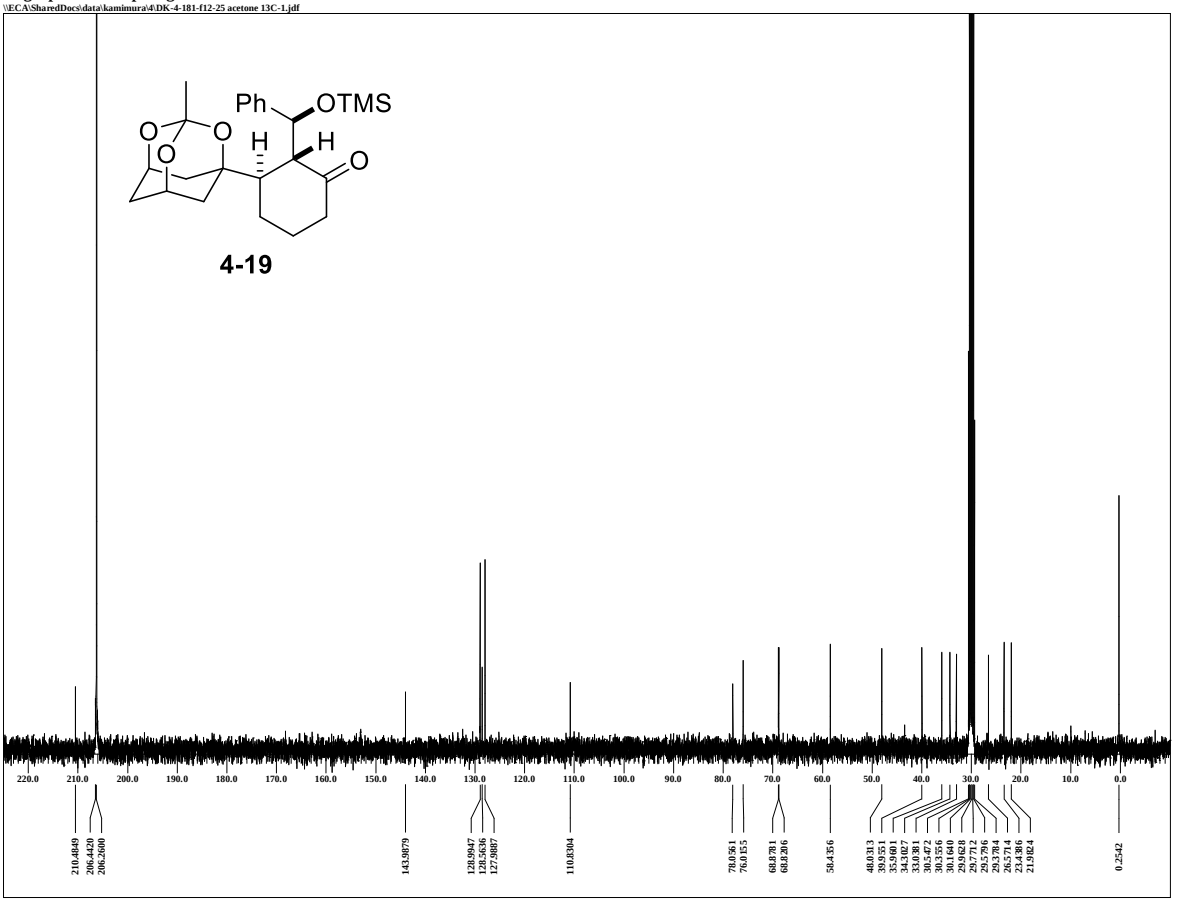
\\ECA\SharedDocs\data\kamimura\4\DK-4-181-f12-25-1.jdf




```
DFILE     DK-4-181-f12-25-1.jdf
COMNT     single_pulse
DATIM     22-07-2013 09:45:41
MENUF
OBNUC     1H
OFR         395.88 MHz
OBFRQ       395.88 MHz
OBSET         6.28 KHz
OBFIN         0.87 Hz
PW1           6.50 usec
DEADT         0.00 usec
PREDL         0.00000 msec
IWT           1.0000 sec
POINT        16384
SPO          16384
TIMES            8
DUMMY            1
FREQU      7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM         2.2073 sec
PD            2.0000 sec
SCANS            8
ADBIT           16
RGAIN           34
BF            0.01 Hz
T1            0.00
T2            0.00
T3          100.00
T4          100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR         395.88 MHz
IRSET         6.28 KHz
IRFIN         0.87 Hz
IRRPW         115 usec
IRATN           79
DFILE     DK-4-181-f12-25-1.jdf
SF
LKSET        13.20 KHz
LKFIN        69.6 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED            0 Hz
FILDC
FILDF
CTEMP        22.1 c
SLVNT     C6D6
EXREF        7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-181-f12-25 acetone 13C-1.jdf

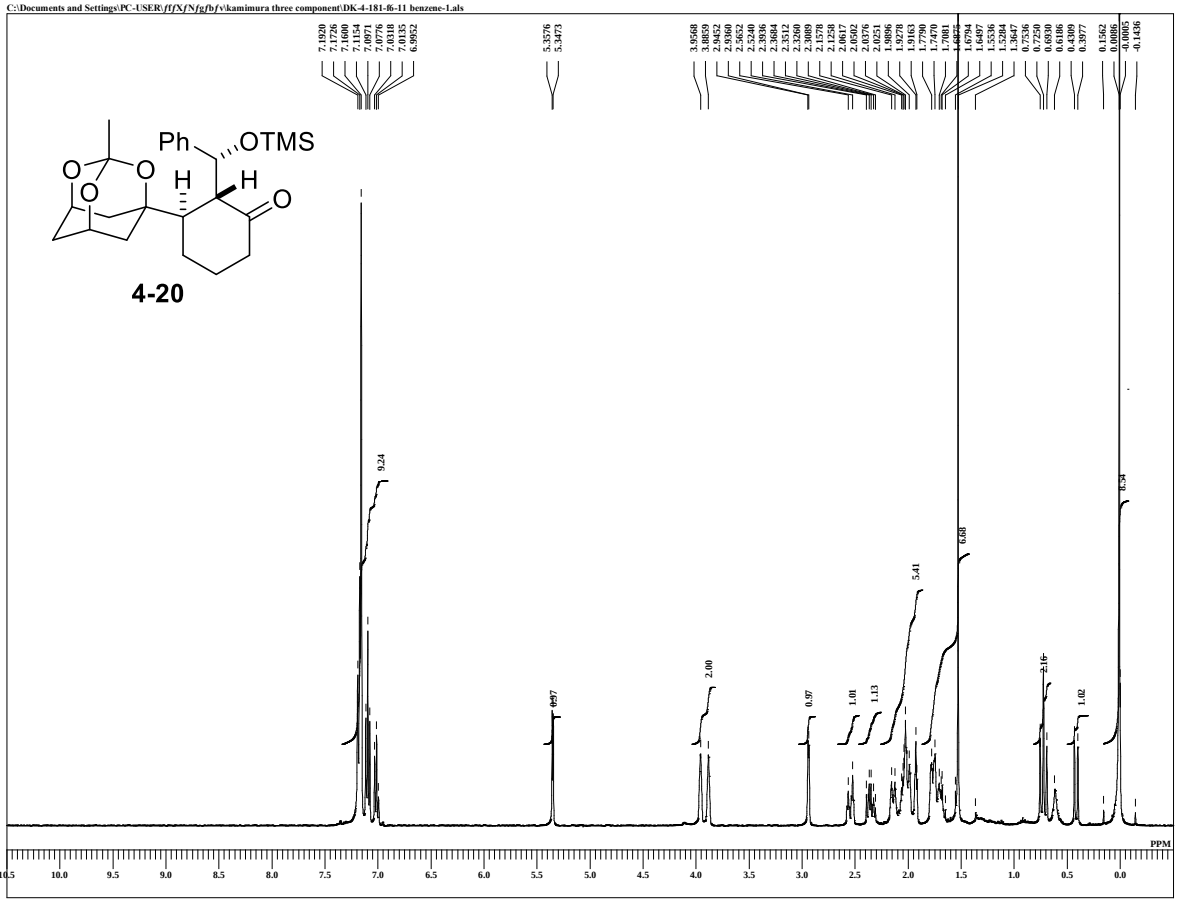



```
DFILE     DK-4-181-f12-25 acetone
COMNT     single pulse decoupled gat
DATIM     25-07-2013 08:58:30
MENUF
OBNUC     13C
OFR         99.55 MHz
OBFRQ       99.55 MHz
OBSET         5.13 KHz
OBFIN         0.98 Hz
PW1           2.90 usec
DEADT         0.00 usec
PREDL         0.00000 msec
IWT           1.0000 sec
POINT        32768
SPO          32768
TIMES          113
DUMMY            4
FREQU      31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM         1.0486 sec
PD            2.0000 sec
SCANS          113
ADBIT           16
RGAIN           60
BF            1.00 Hz
T1            0.00
T2            0.00
T3          100.00
T4          100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR         395.88 MHz
IRSET         6.28 KHz
IRFIN         0.87 Hz
IRRPW         115 usec
IRATN           79
DFILE     DK-4-181-f12-25 acetone
SF
LKSET        12.90 KHz
LKFIN        59.1 Hz
LKLEV            0
LGAIN            0
LKPHS            0
LKSIG            0
CSPED            0 Hz
FILDC
FILDF
CTEMP        23.2 c
SLVNT     ACETN
EXREF        206.26 ppm
```

スペクトルデータ

single_pulse

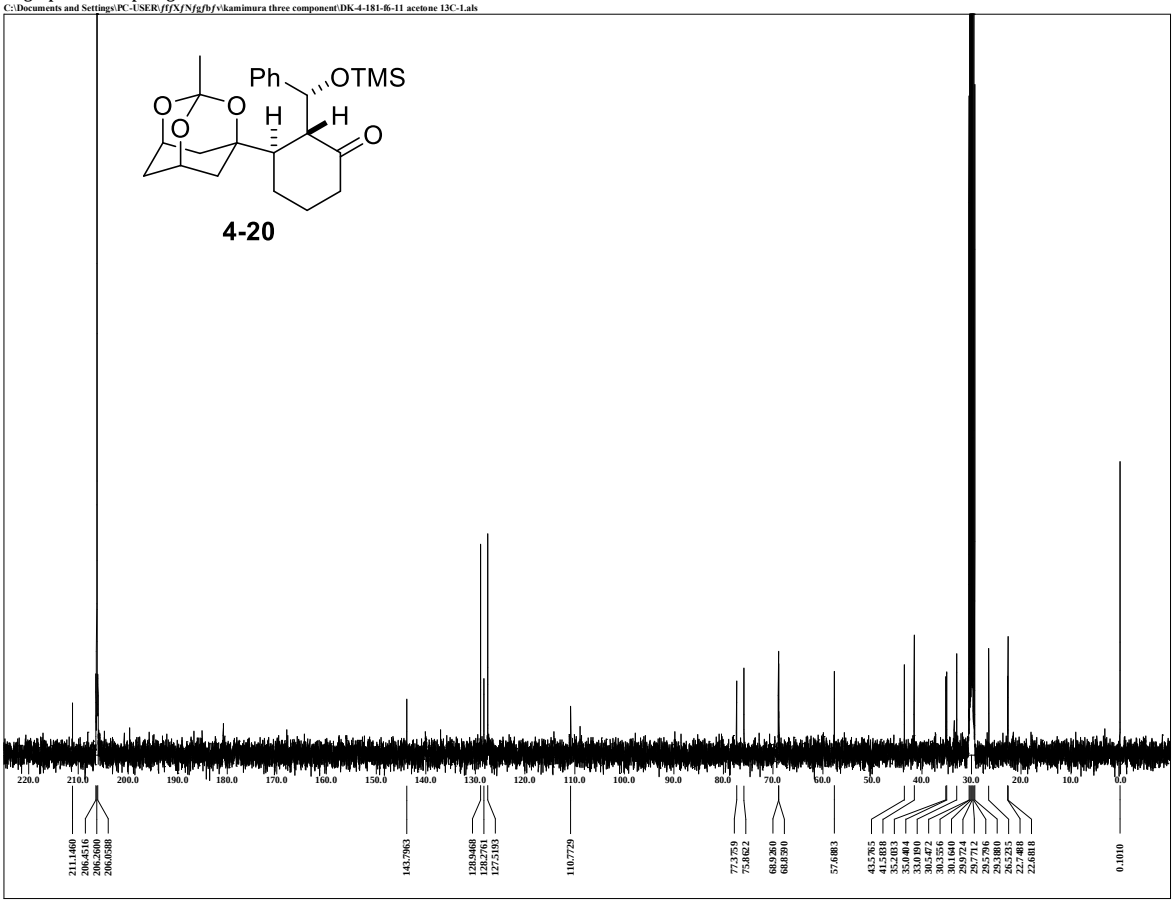
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\kamimura three component\DK-4-181-f6-11 benzene-1.als



DFILE DK-4-181-f6-11 benzene-1
COMNT single_pulse
DATIM 24-07-2013 11:33:07
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.50 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-4-181-f6-11 benzene-1
SF
LKSET 13.20 KHz
LKFIN 69.6 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.4 c
SLVNT C6D6
EXREF 7.16 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\kamimura three component\DK-4-181-f6-11 acetone 13C-1.als

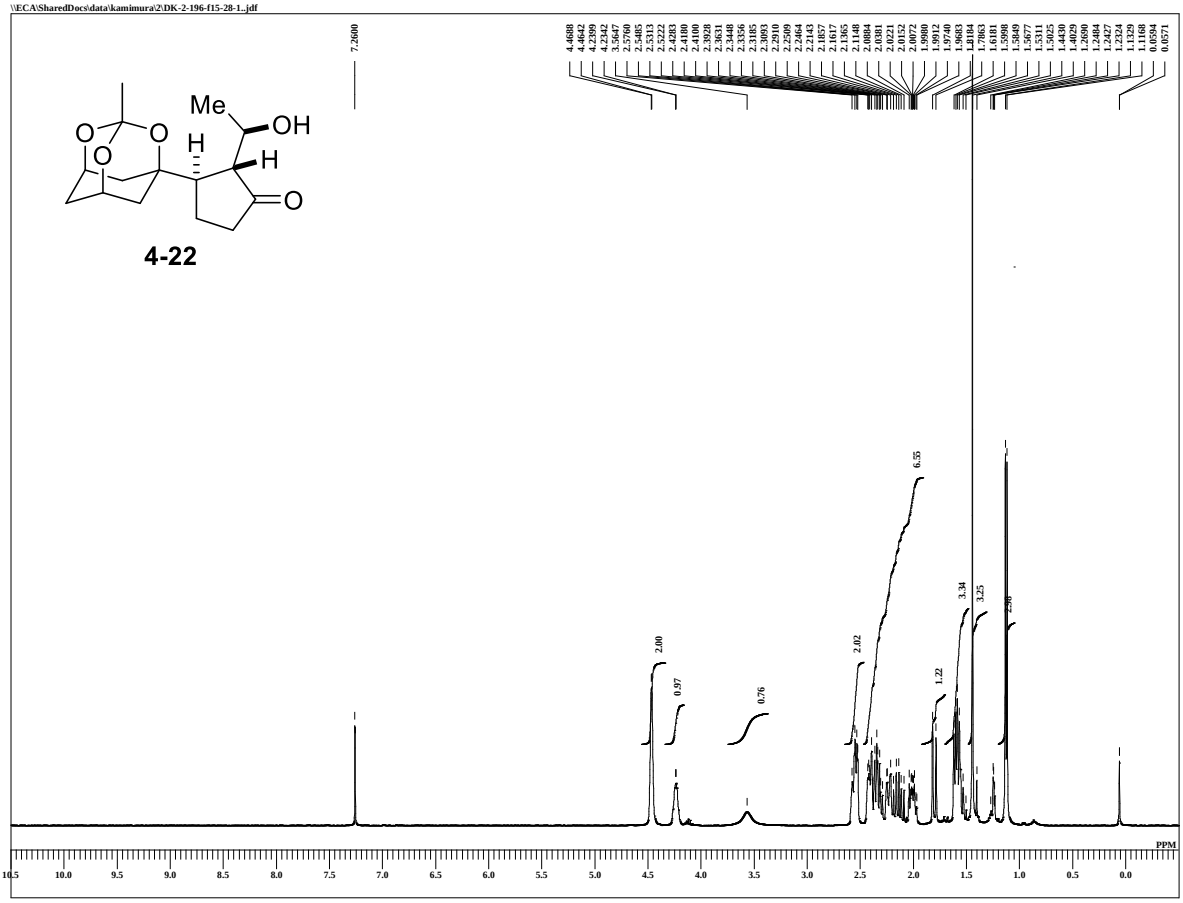


DFILE DK-4-181-f6-11 acetone 1
COMNT single pulse decoupled gat
DATIM 25-07-2013 08:46:20
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.90 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 302
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 302
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-4-181-f6-11 acetone 1
SF
LKSET 12.90 KHz
LKFIN 59.1 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.1 c
SLVNT ACETN
EXREF 206.26 ppm

スペクトルデータ

single_pulse

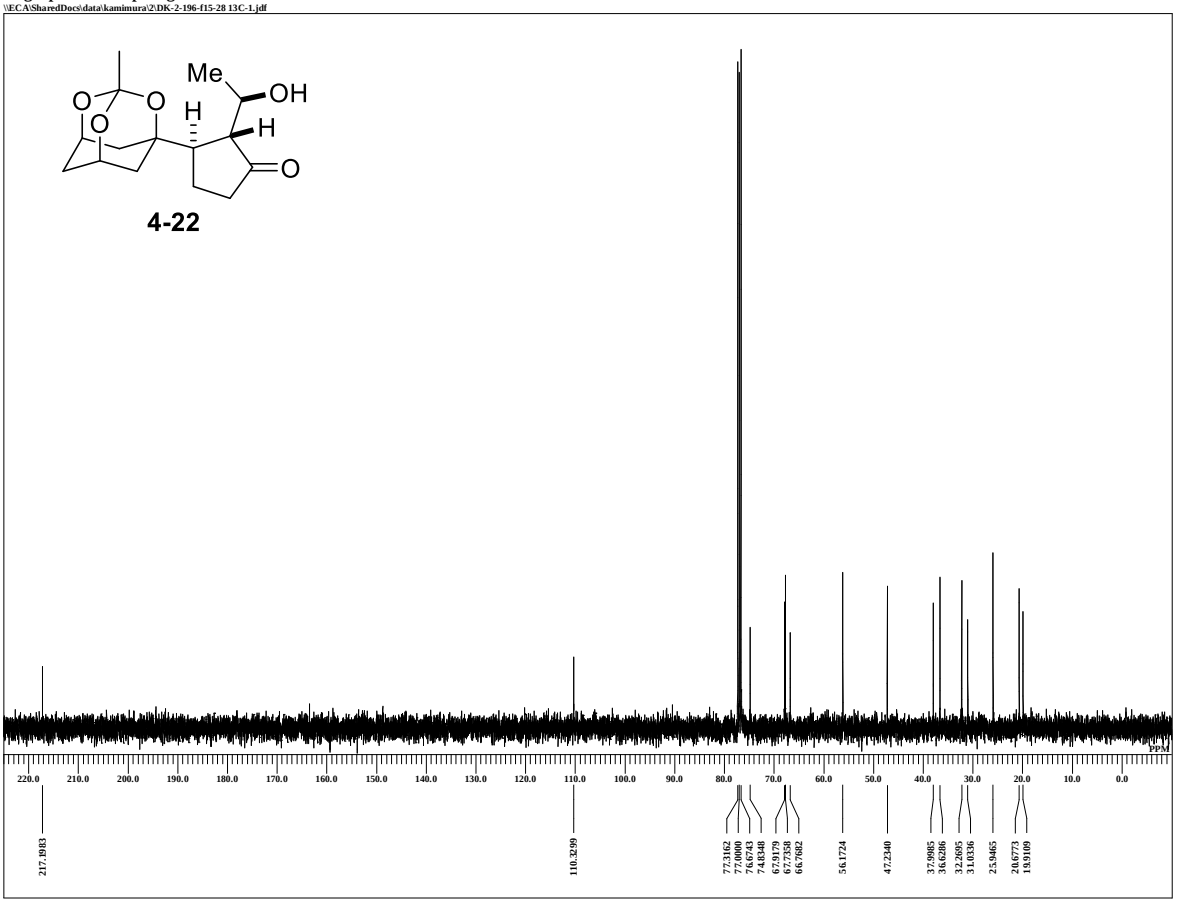
\\ECA\SharedDocs\data\kamimura\2\DK-2-196-f15-28-1..jdf




```
DFILE    DK-2-196-f15-28-1..jdf
COMNT    single_pulse
DATIM    08-01-2013 18:00:44
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.38 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    16
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    16
ADBIT    16
RGAIN    38
BF       0.01 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-2-196-f15-28-1..jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.2 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-196-f15-28 13C-1.jdf

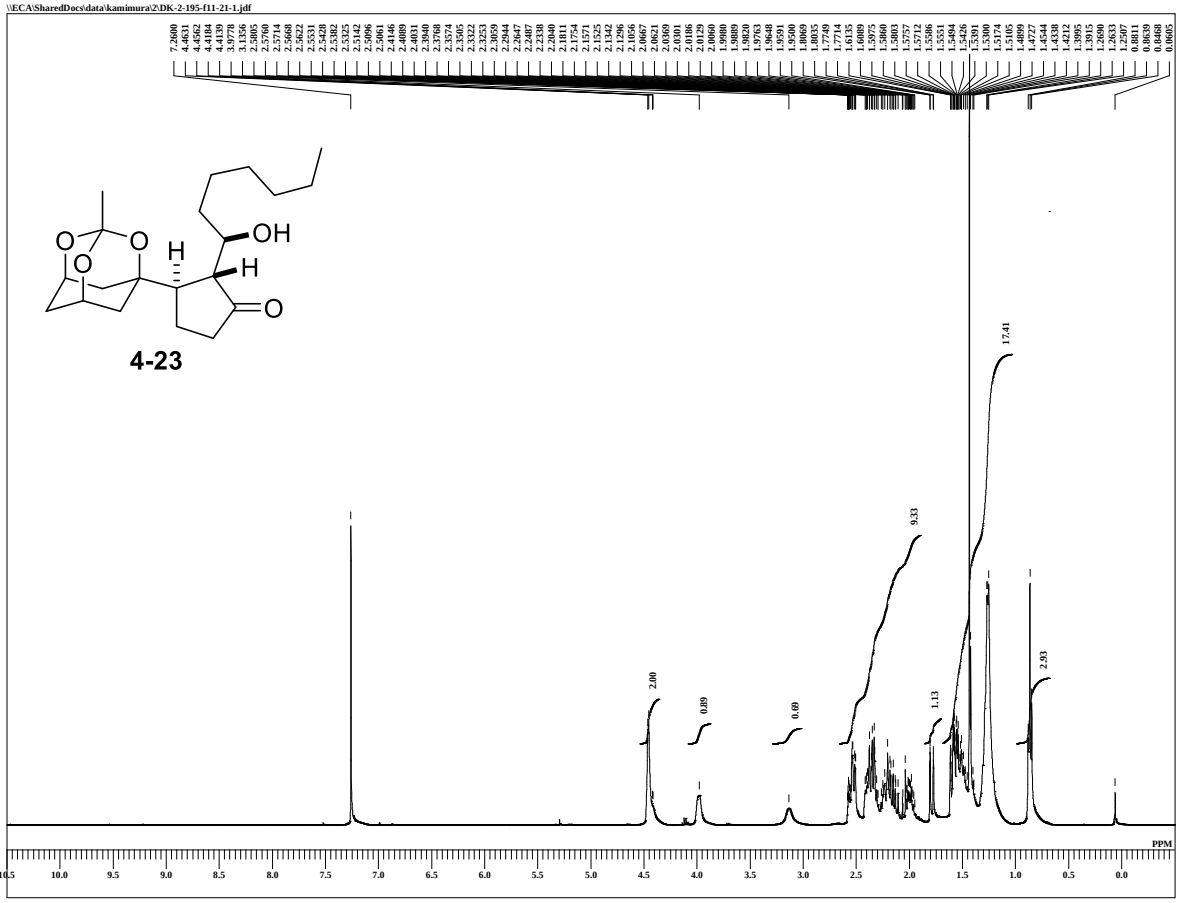



```
DFILE    DK-2-196-f15-28 13C-1.jd
COMNT    single pulse decoupled gat
DATIM    16-11-2012 08:41:12
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.25 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    73
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    73
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-2-196-f15-28 13C-1.jd
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    21.9 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

スペクトルデータ

single_pulse

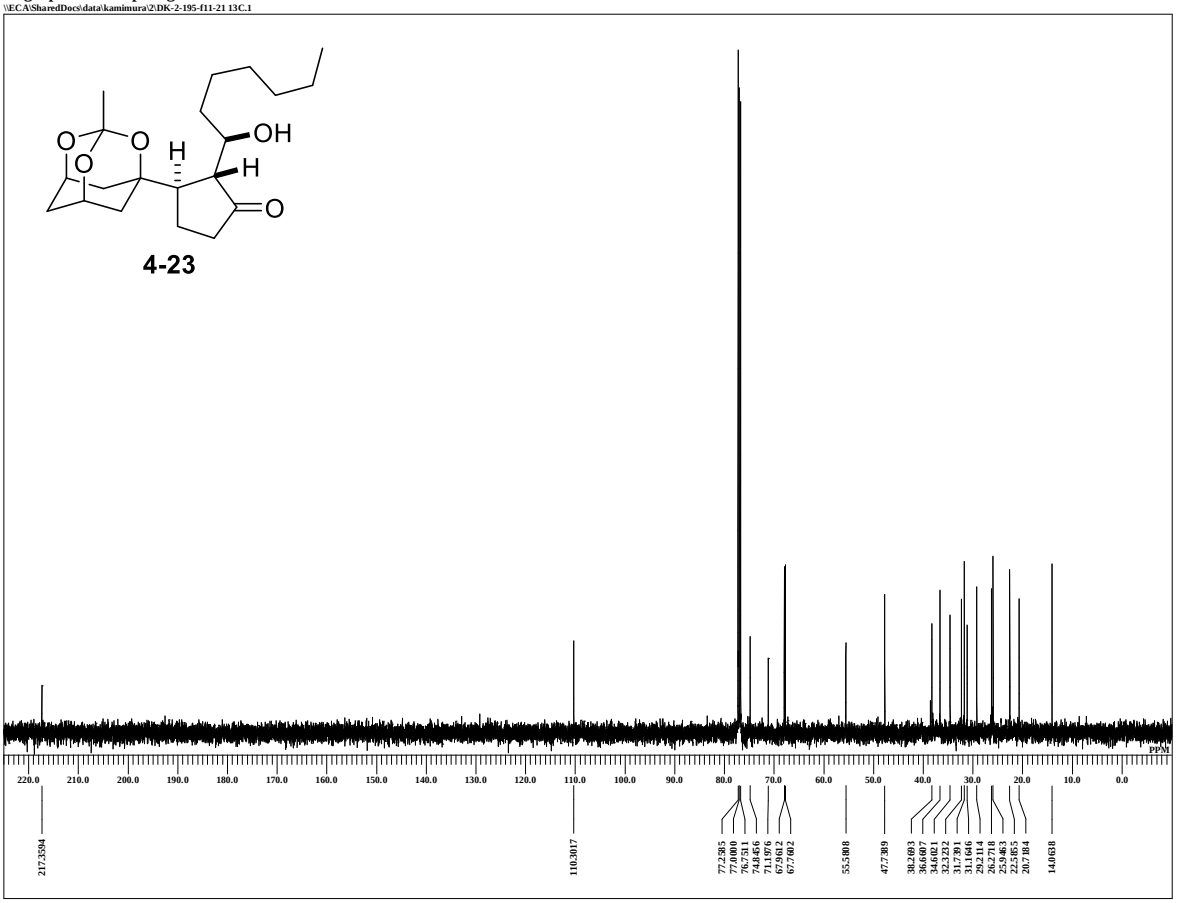
\\ECA\SharedDocs\data\kamimura\2\DK-2-195-f11-21-1.jdf



DFILE	DK-2-195-f11-21-1.jdf
COMNT	single_pulse
DATIM	15-11-2012 12:47:05
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-2-195-f11-21-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-195-f11-21 13C.1

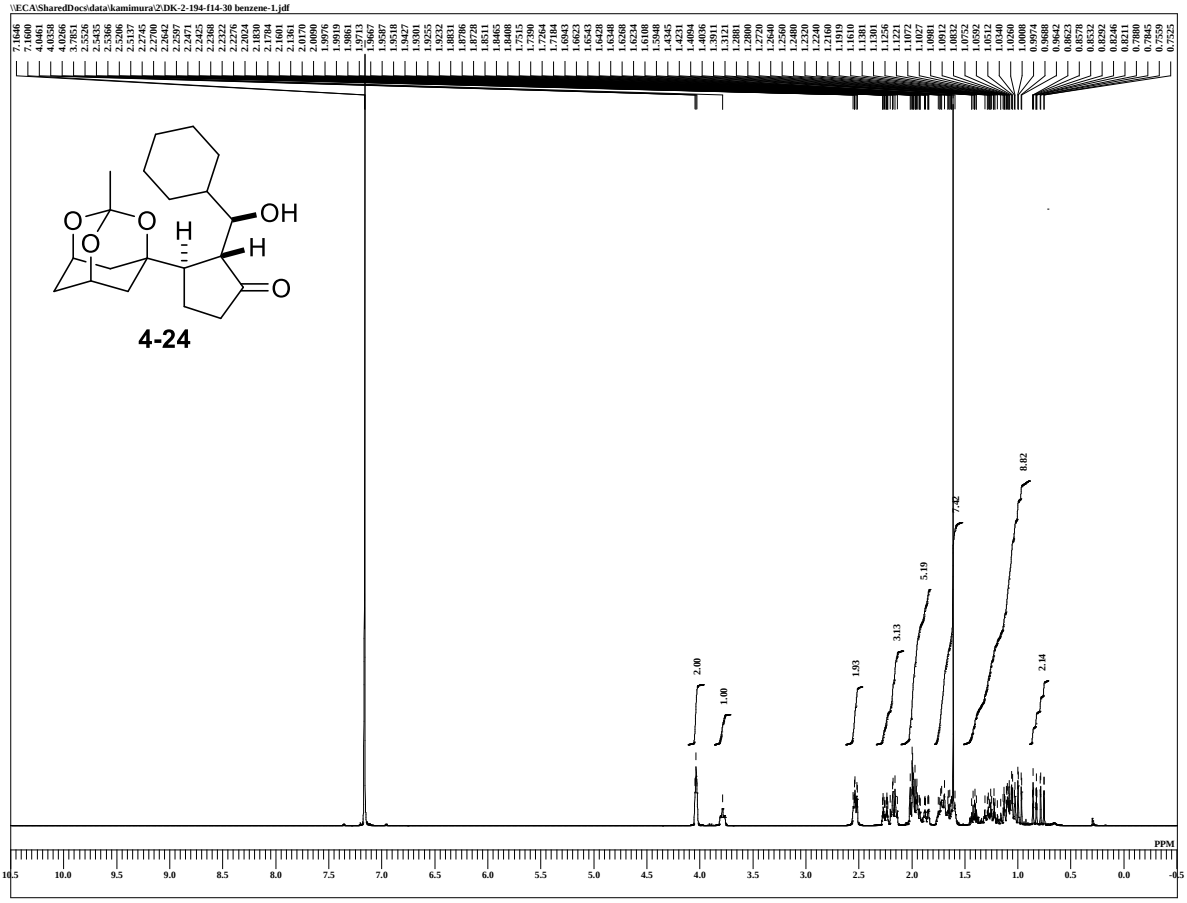


DFILE	DK-2-195-f11-21 13C.1
COMNT	single pulse decoupled gat
DATIM	15-11-2012 13:48:35
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	142
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	142
ADBIT	16
RGAIN	50
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-2-195-f11-21 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

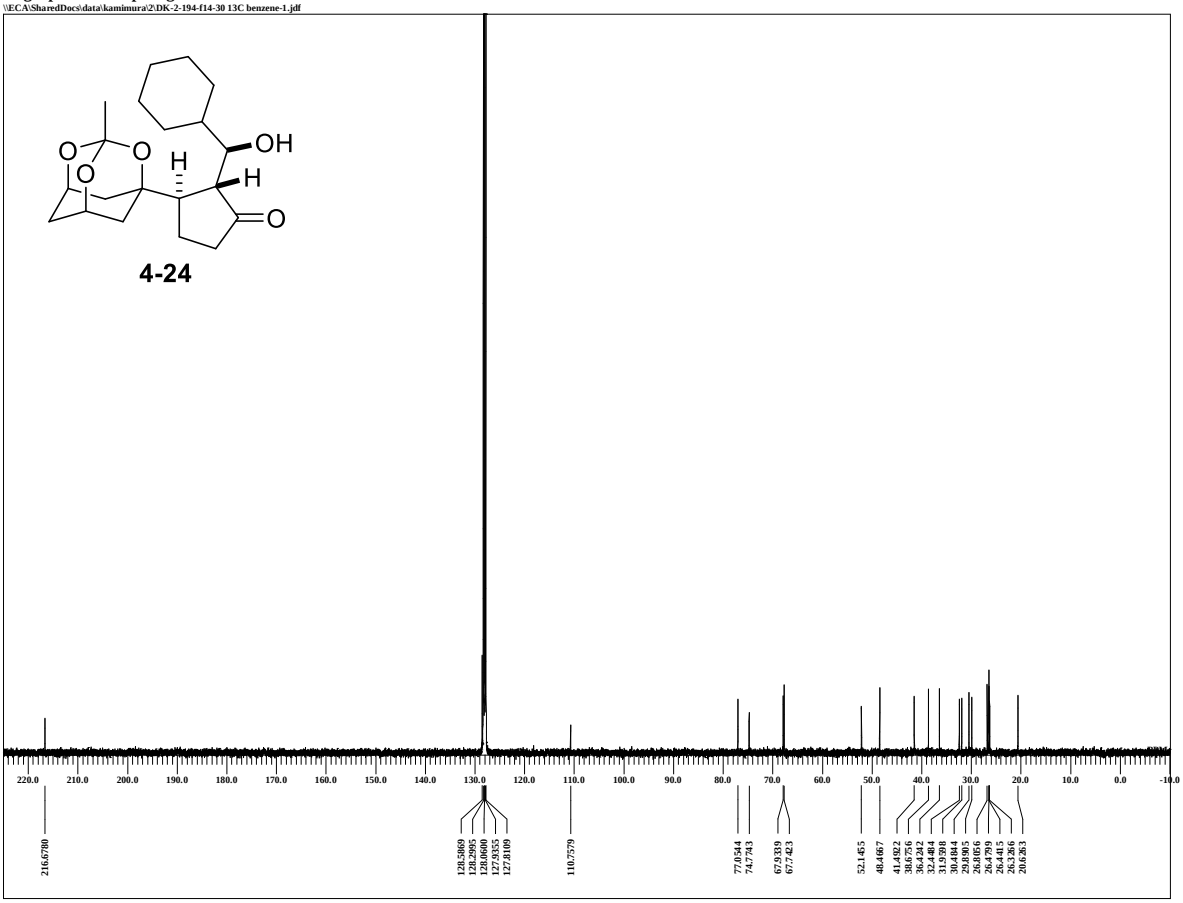
\\ECA\SharedDocs\data\kamimura\2\DK-2-194-f14-30 benzene-1.jdf




```
DFILE   DK-2-194-f14-30 benzene
COMNT   single_pulse
DATIM   04-03-2013 18:13:33
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.38 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT      16384
SPO        16384
TIMES          8
DUMMY          1
FREQU    7422.80 Hz
FLT     30000 Hz
DELAY      16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS          8
ADBIT         16
RGAIN         34
BF          0.01 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       115 usec
IRATN          79
DFILE   DK-2-194-f14-30 benzene
SF
LKSET      13.20 KHz
LKFIN      69.6 Hz
LKLEV          0
LGAIN          0
LKPHS          0
LKSIG          0
CSPED          0 Hz
FILDC
FILDF
CTEMP      20.5 c
SLVNT   C6D6
EXREF      7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-194-f14-30 13C benzene-1.jdf

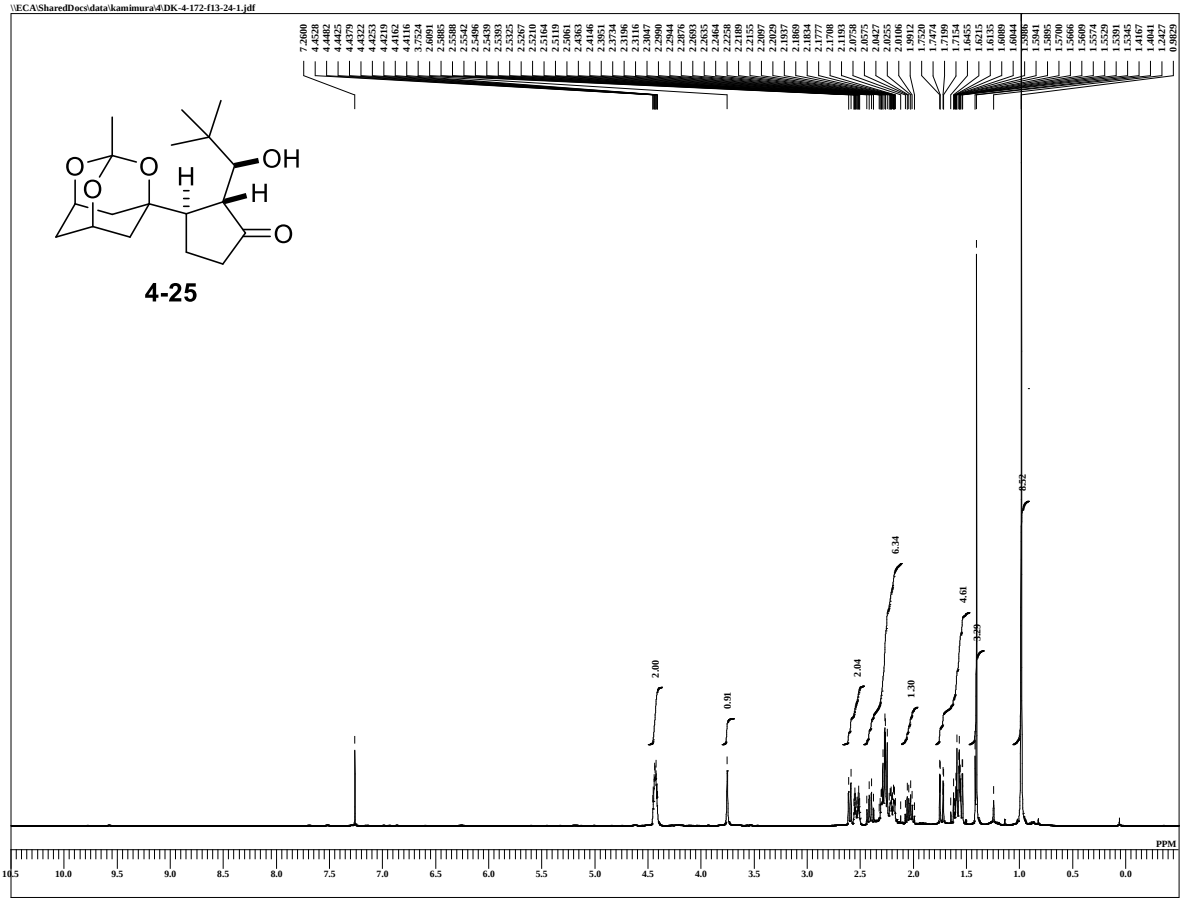



```
DFILE   DK-2-194-f14-30 13C ben
COMNT   single pulse decoupled gat
DATIM   04-03-2013 18:21:41
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
PW1        3.25 usec
DEADT      0.00 usec
PREDL      0.00000 msec
IWT        1.0000 sec
POINT     32768
SPO       32768
TIMES       145
DUMMY         4
FREQU   31250.00 Hz
FLT     125000 Hz
DELAY     20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS       145
ADBIT        16
RGAIN        58
BF         1.00 Hz
T1         0.00
T2         0.00
T3       100.00
T4       100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET      6.28 KHz
IRFIN      0.87 Hz
IRRPW      115 usec
IRATN         79
DFILE   DK-2-194-f14-30 13C ben
SF
LKSET     13.20 KHz
LKFIN     69.6 Hz
LKLEV         0
LGAIN         0
LKPHS         0
LKSIG         0
CSPED         0 Hz
FILDC
FILDF
CTEMP     20.7 c
SLVNT   C6D6
EXREF     128.06 ppm
```

スペクトルデータ

single_pulse

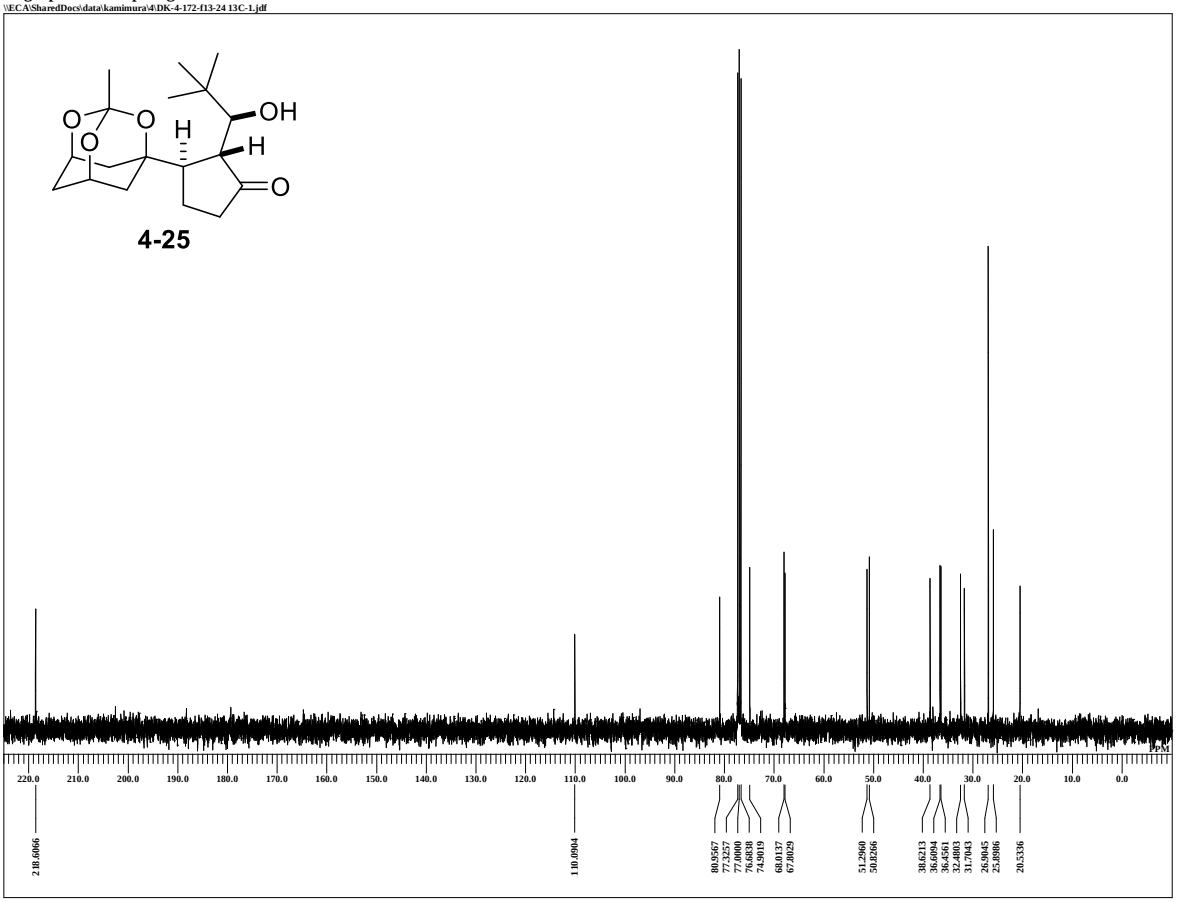
\\ECA\SharedDocs\data\kamimura\4\DK-4-172-f13-24-1.jdf




```
DFILE    DK-4-172-f13-24-1.jdf
COMNT    single_pulse
DATIM    12-07-2013 15:25:09
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.50 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    34
BF       0.01 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-4-172-f13-24-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    25.2 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-172-f13-24 13C-1.jdf




```
DFILE    DK-4-172-f13-24 13C-1.jd
COMNT    single pulse decoupled gat
DATIM    12-07-2013 15:30:43
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      2.90 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    60
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    60
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-4-172-f13-24 13C-1.jd
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    25.0 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

スペクトルデータ

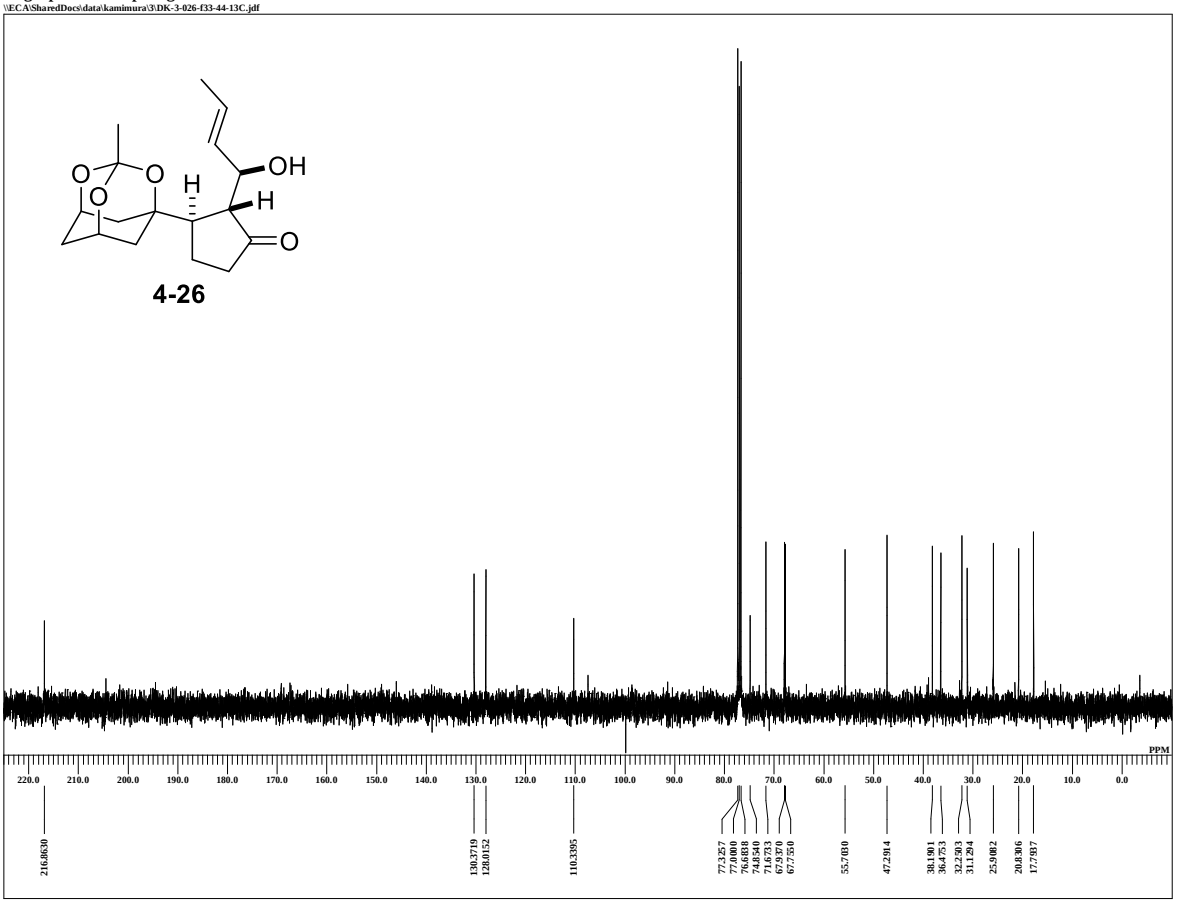
single_pulse

\\ECA\SharedDocs\data\kamimura\3\DK-3-026-f33-44-1.jdf


```
DFILE    DK-3-026-f33-44-1.jdf
COMNT    single_pulse
DATIM    12-12-2012 14:19:23
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.38 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    34
BF       0.01 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-3-026-f33-44-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.3 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-026-f33-44-13C.jdf

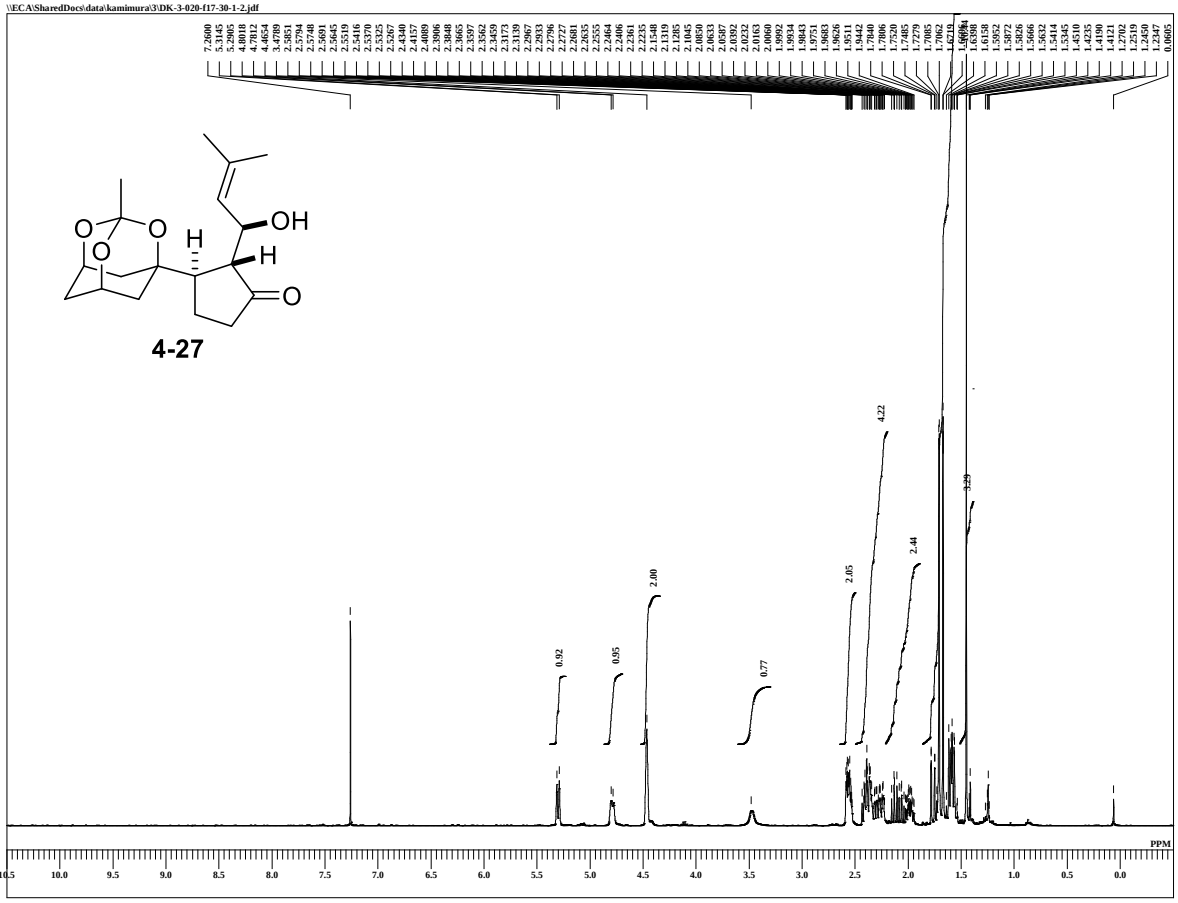



```
DFILE    DK-3-026-f33-44-13C.jdf
COMNT    single pulse decoupled gat
DATIM    12-12-2012 14:23:14
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.25 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    58
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    58
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-3-026-f33-44-13C.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.4 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

スペクトルデータ

single_pulse

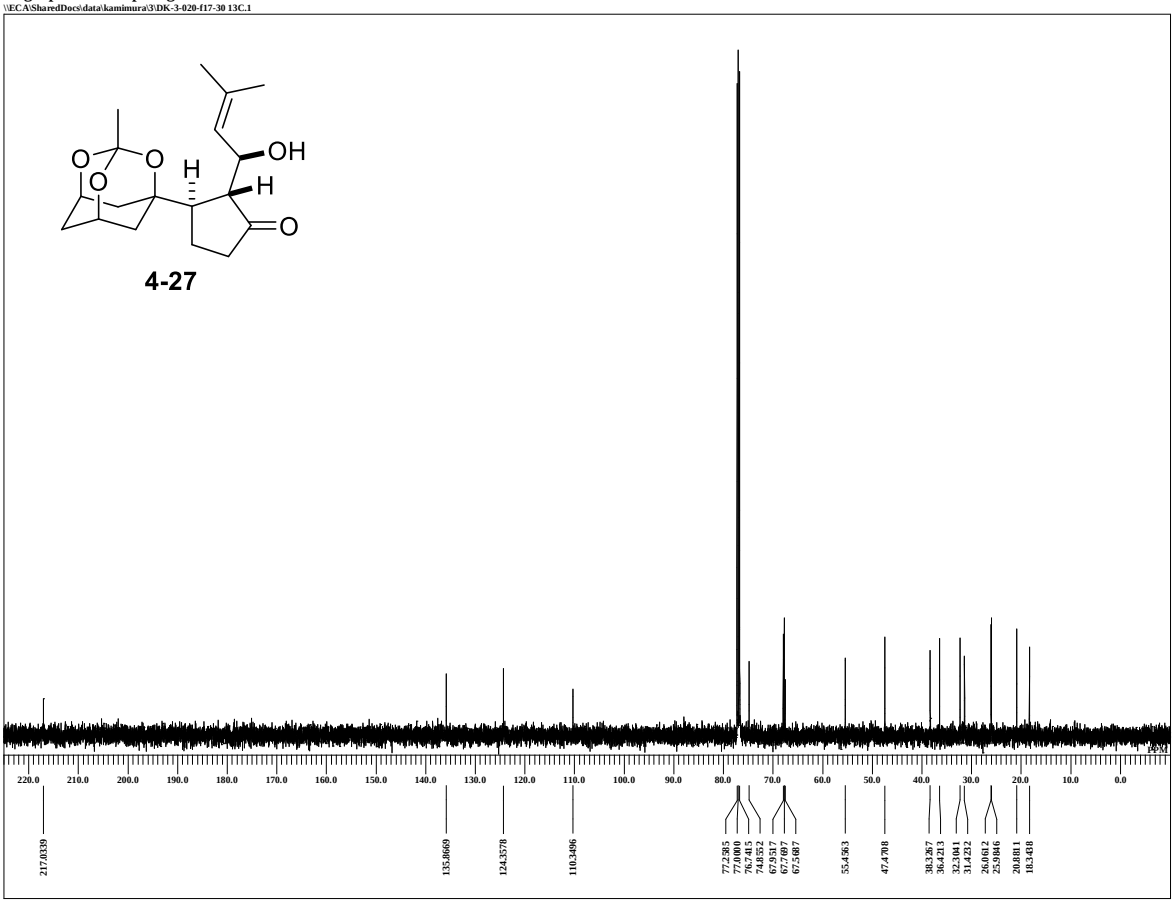
\\ECA\SharedDocs\data\kamimura\3\DK-3-020-f17-30-1-2.jdf



DFILE	DK-3-020-f17-30-1-2.jdf
COMNT	single_pulse
DATIM	26-01-2013 17:24:29
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	38
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-020-f17-30-1-2.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-020-f17-30 13C.1

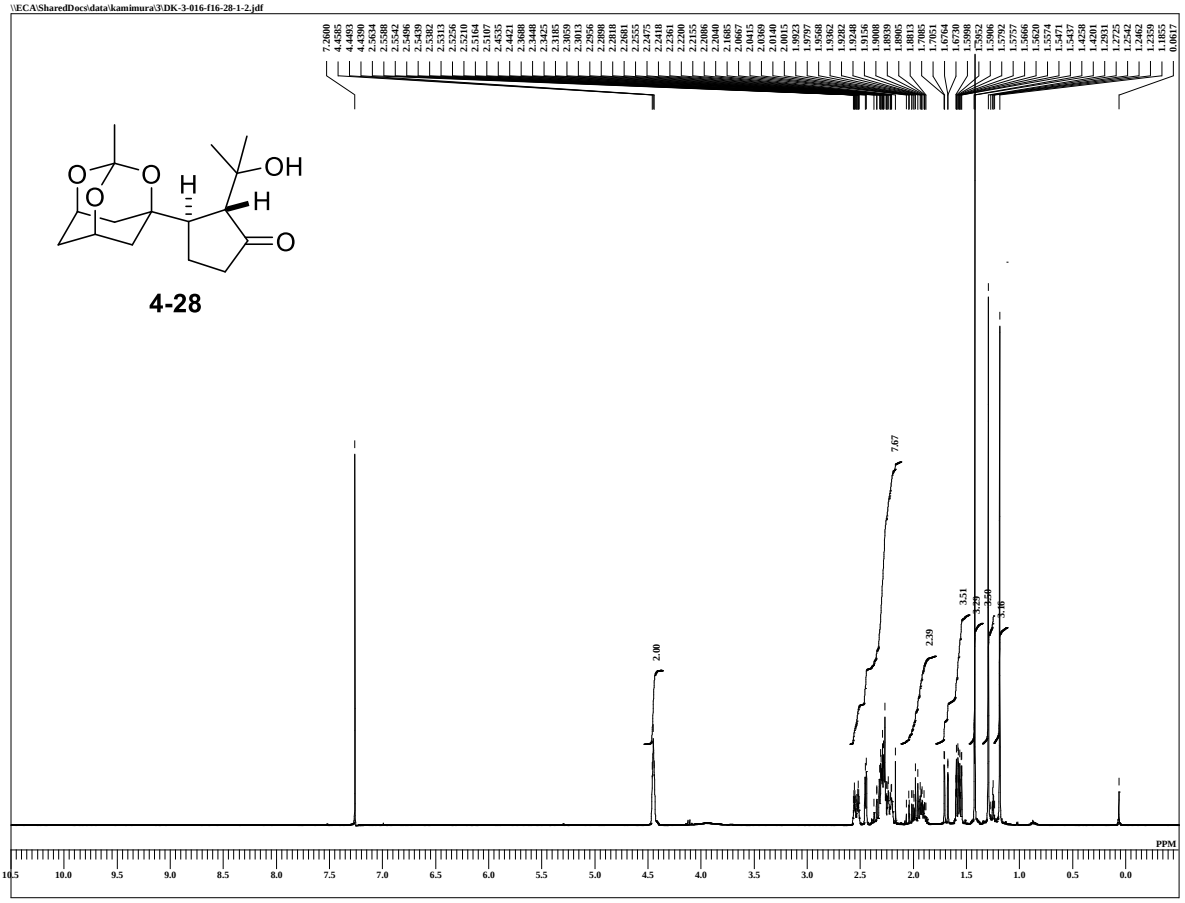


DFILE	DK-3-020-f17-30 13C.1
COMNT	single pulse decoupled gat
DATIM	10-12-2012 15:50:19
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	149
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	149
ADBIT	16
RGAIN	50
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-3-020-f17-30 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

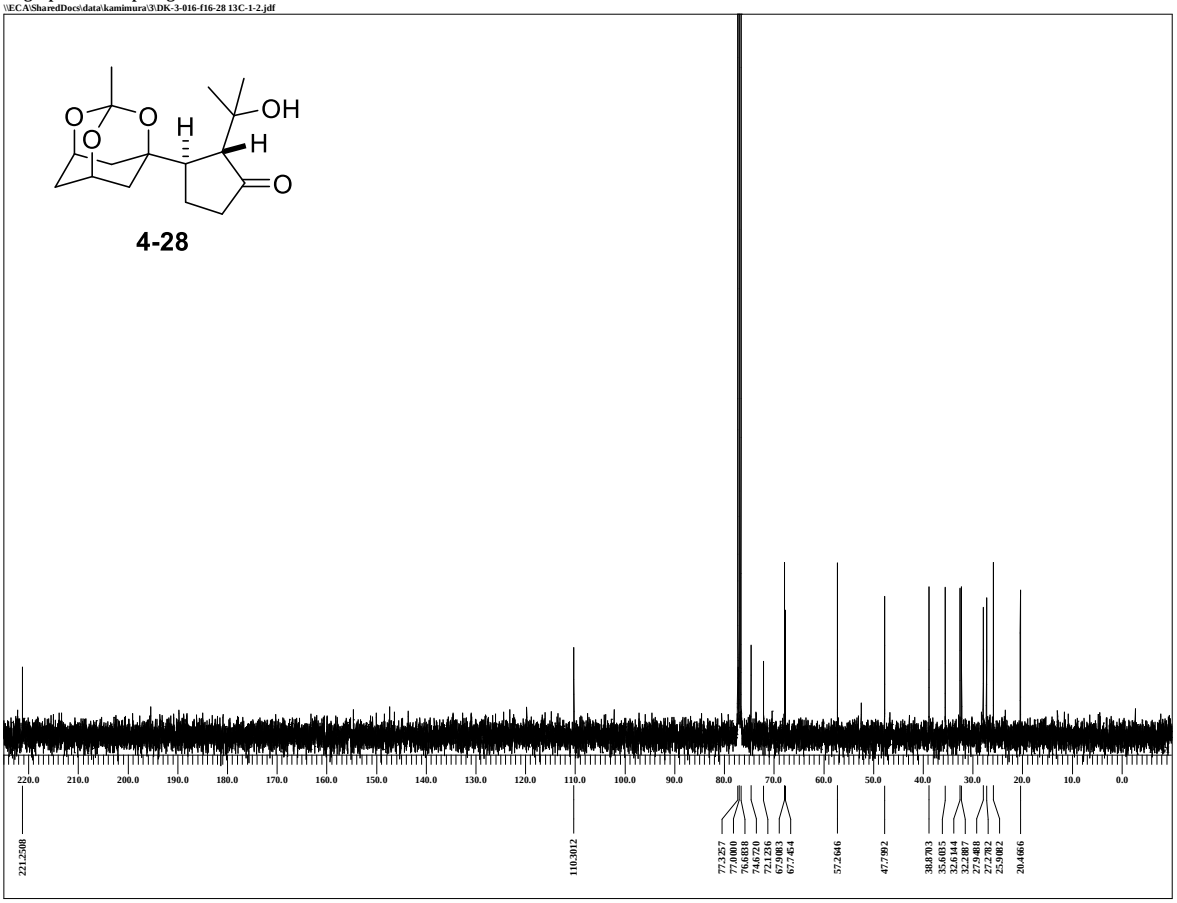
\\ECA\SharedDocs\data\kamimura\3\DK-3-016-f16-28-1-2.jdf



DFILE	DK-3-016-f16-28-1-2.jdf
COMNT	single_pulse
DATIM	24-01-2013 18:11:49
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	40
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-016-f16-28-1-2.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-016-f16-28 13C-1-2.jdf

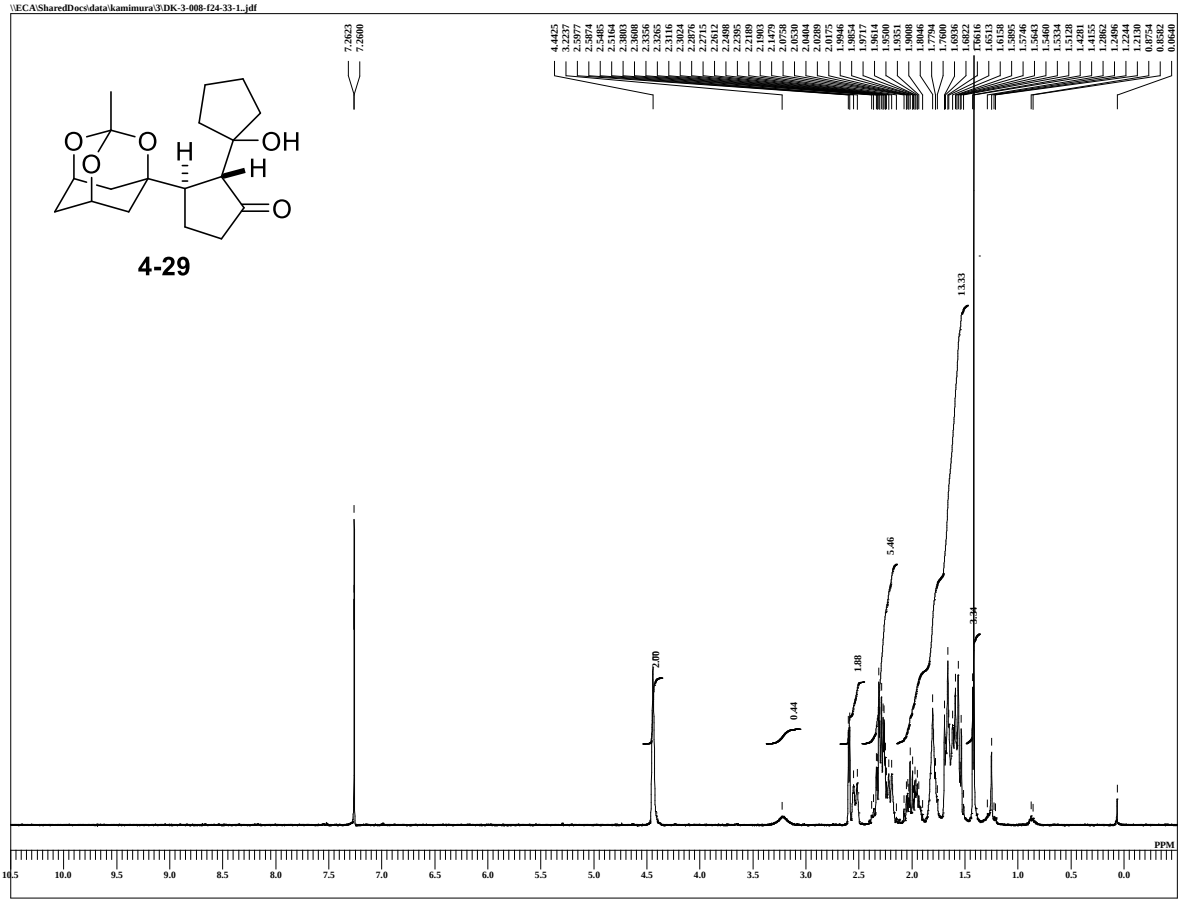


DFILE	DK-3-016-f16-28 13C-1-2
COMNT	single pulse decoupled gat
DATIM	24-01-2013 18:17:48
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	104
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	104
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-016-f16-28 13C-1-2
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

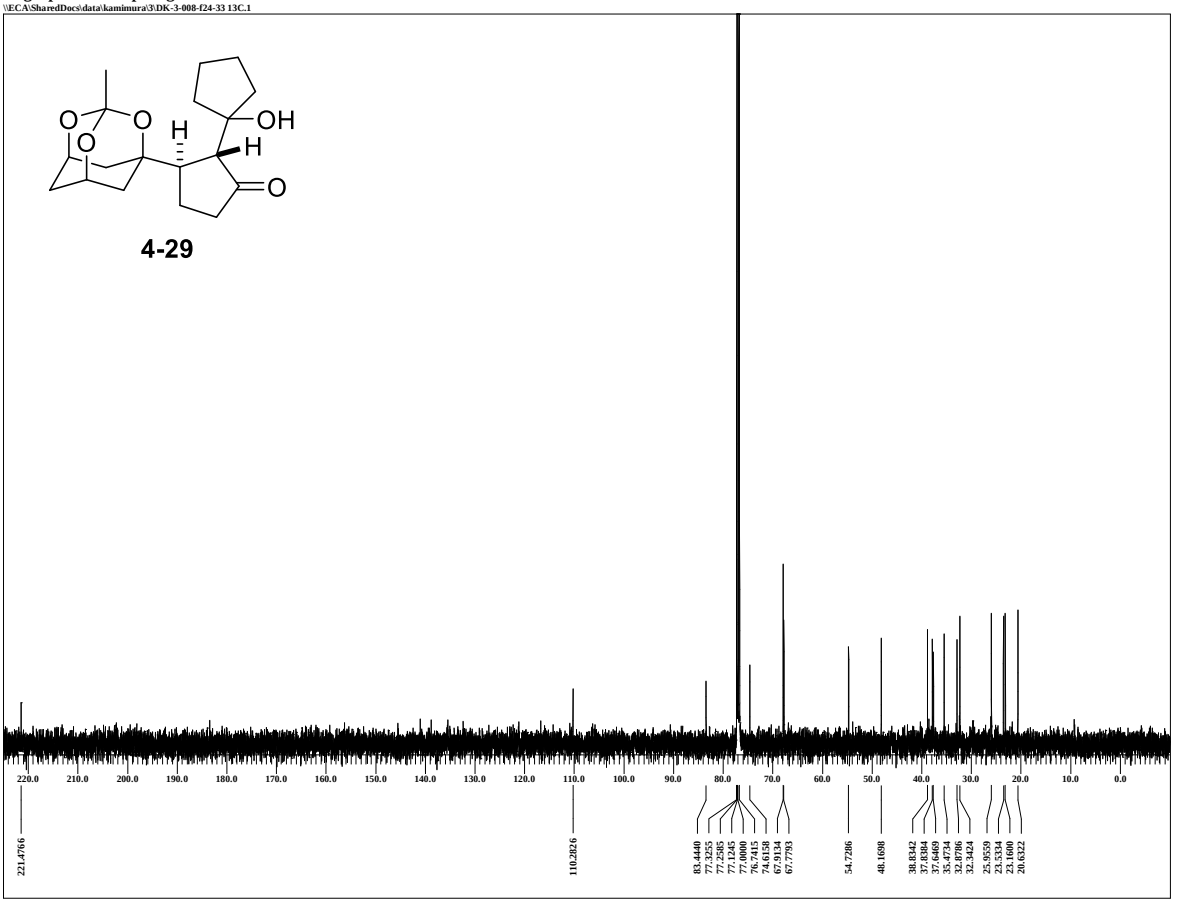
\\ECA\SharedDocs\data\kamimura\3\DK-3-008-f24-33-1_.jdf



DFILE	DK-3-008-f24-33-1_.jdf
COMNT	single_pulse
DATIM	23-01-2013 10:05:15
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	44
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-008-f24-33-1_.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-008-f24-33 13C.1

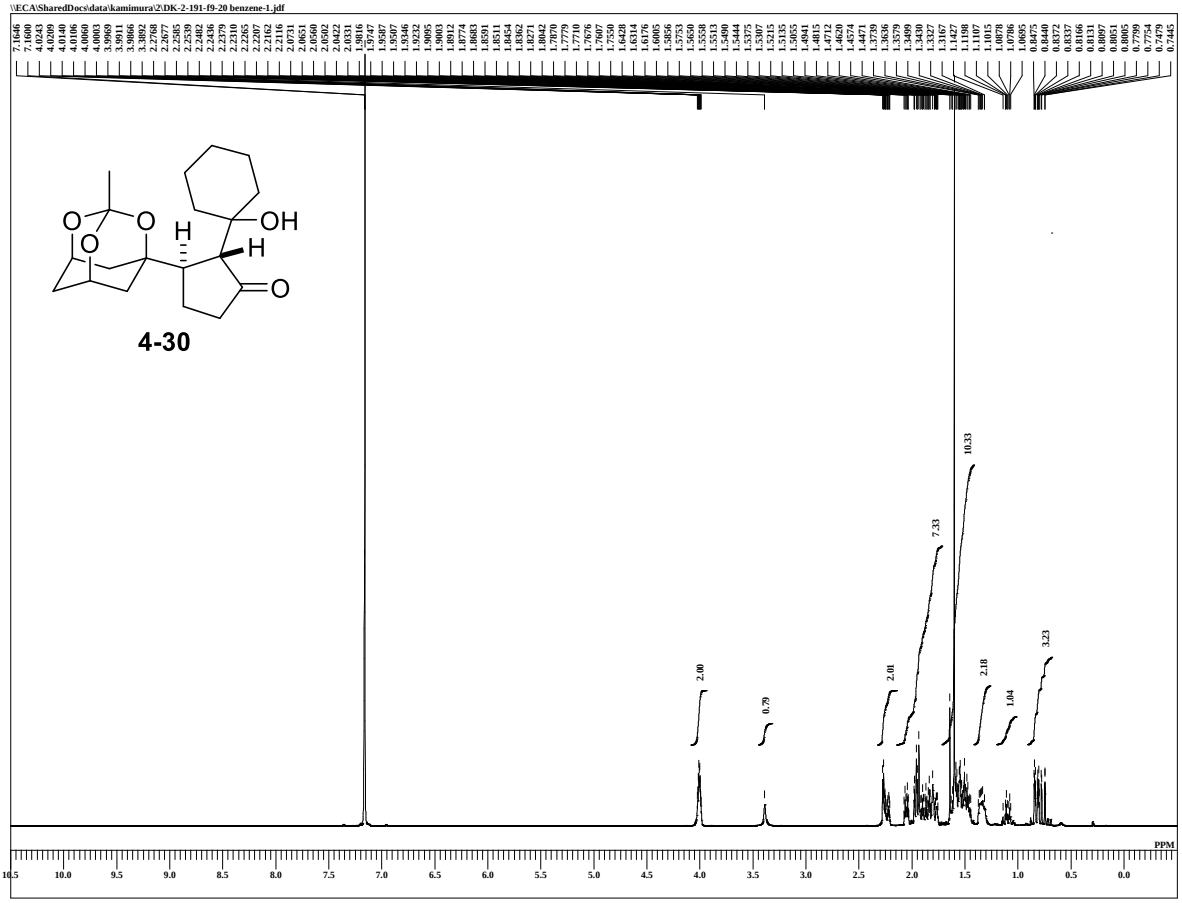


DFILE	DK-3-008-f24-33 13C.1
COMNT	single pulse decoupled gat
DATIM	03-12-2012 20:07:22
MENUF	
OBNUC	13C
OFR	124.51 MHz
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
PW1	3.70 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	569
DUMMY	4
FREQU	39062.50 Hz
FLT	157000 Hz
DELAY	20.80 usec
ACQTM	0.8389 sec
PD	2.0000 sec
SCANS	569
ADBIT	16
RGAIN	48
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	495.13 MHz
IRSET	4.38 KHz
IRFIN	9.64 Hz
IRRPW	92 usec
IRATN	79
DFILE	DK-3-008-f24-33 13C.1
SF	
LKSET	748.40 KHz
LKFIN	98.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

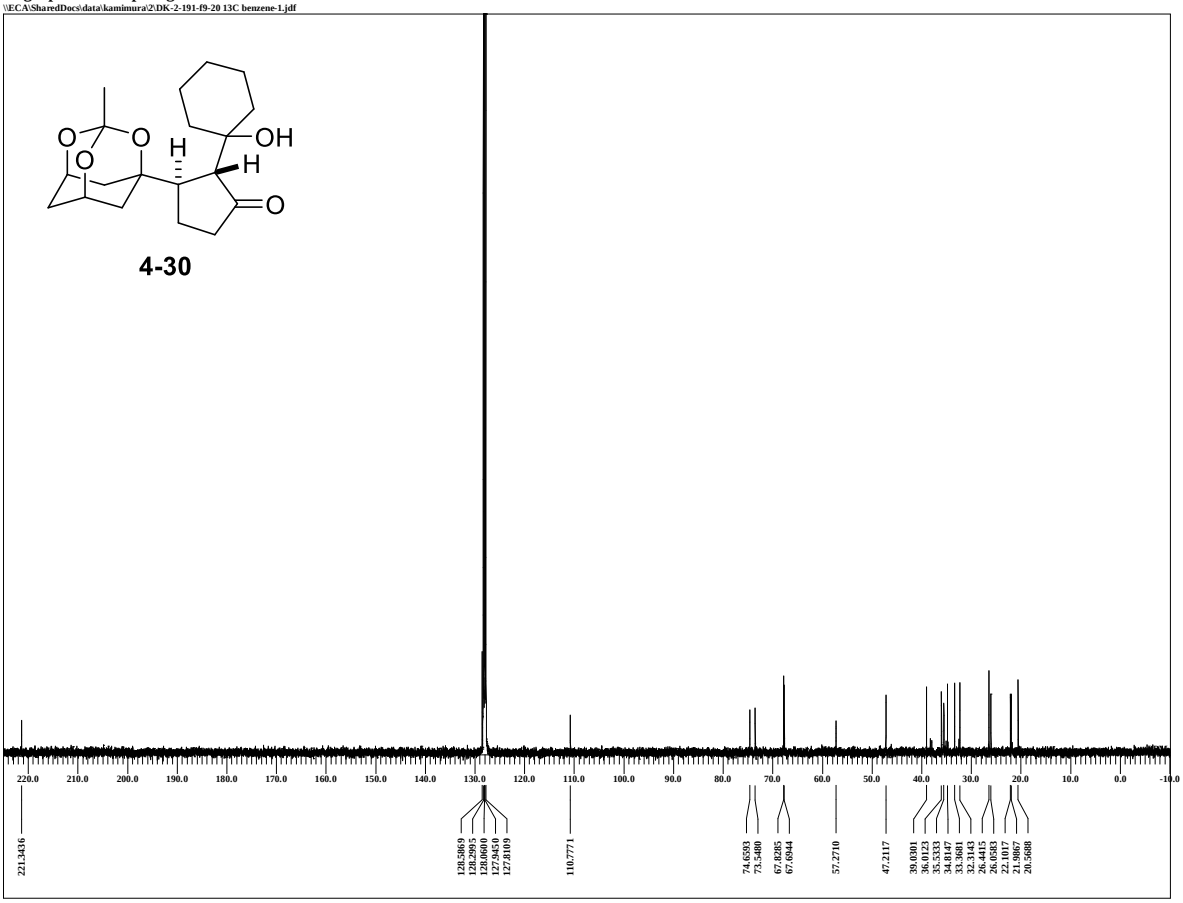
\\ECA\SharedDocs\data\kamimura\2\DK-2-191-f9-20 benzene-1.jdf



DFILE	DK-2-191-f9-20 benzene-1
COMNT	single_pulse
DATIM	02-03-2013 17:19:46
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-2-191-f9-20 benzene-1
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.2 c
SLVNT	C6D6
EXREF	7.16 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-191-f9-20 13C benzene-1.jdf

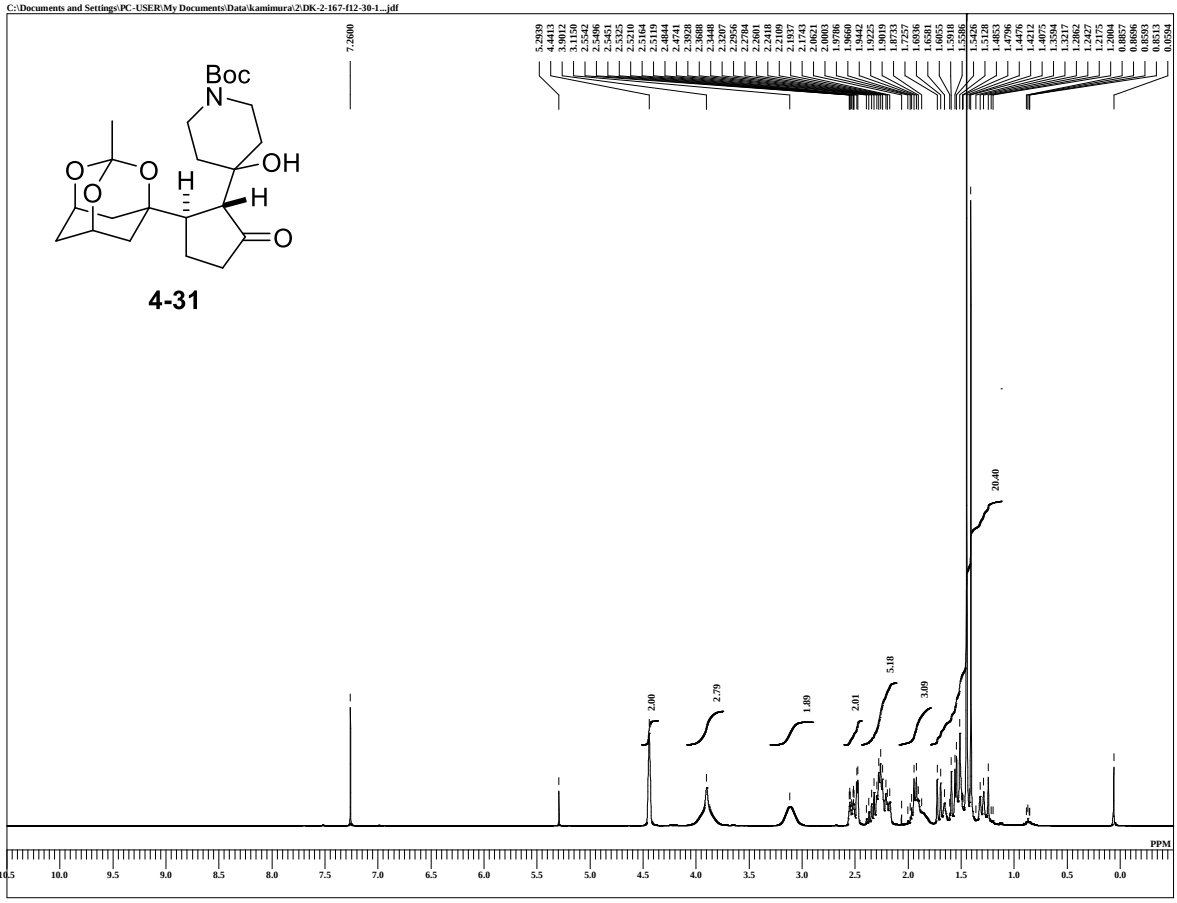


DFILE	DK-2-191-f9-20 13C benz
COMNT	single pulse decoupled gat
DATIM	02-03-2013 17:27:24
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	136
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	136
ADBIT	16
RGAIN	58
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-2-191-f9-20 13C benz
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.5 c
SLVNT	C6D6
EXREF	128.06 ppm

スペクトルデータ

single_pulse

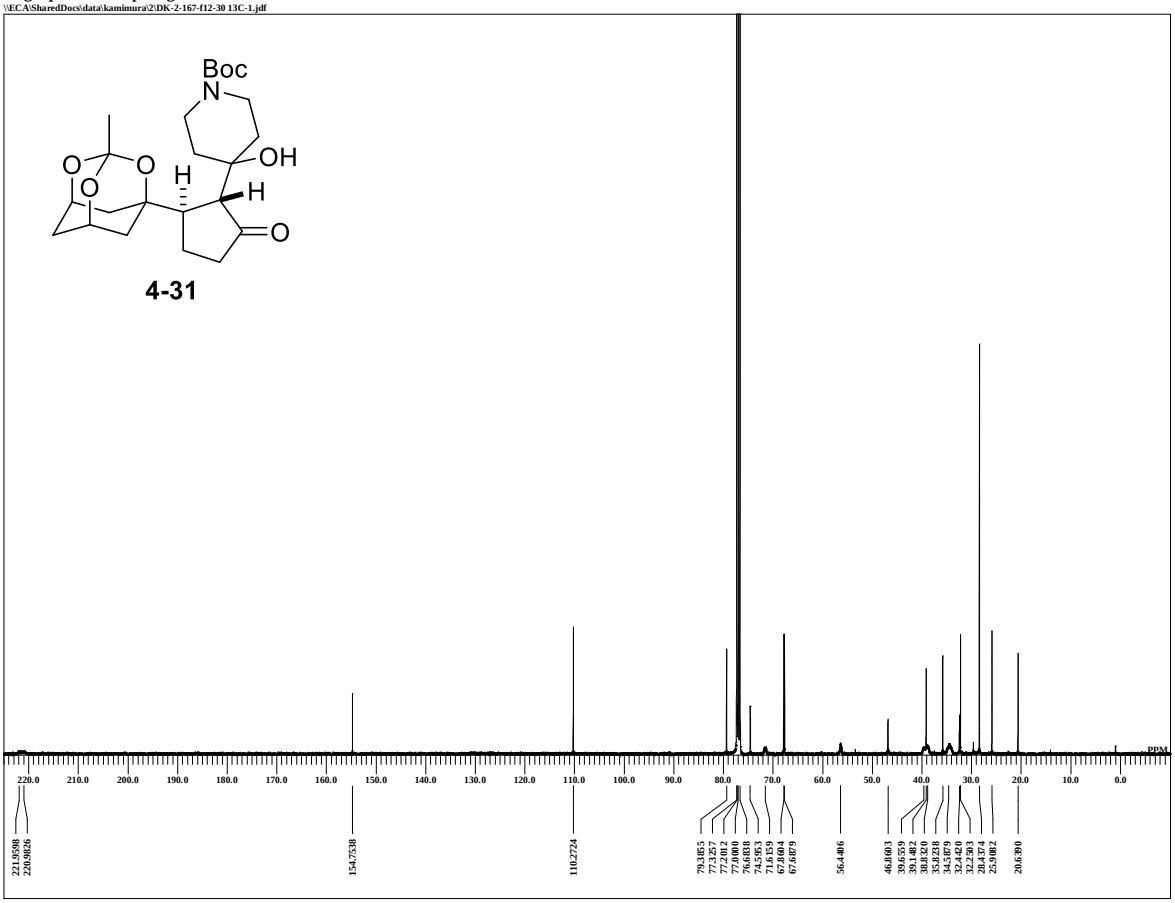
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\DK-2-167-f12-30-1...jdf



DFILE DK-2-167-f12-30-1...jdf
COMNT single_pulse
DATIM 20-03-2013 18:31:33
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.38 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 32
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 32
ADBIT 16
RGAIN 36
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-2-167-f12-30-1...jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\2\DK-2-167-f12-30 13C-1.jdf

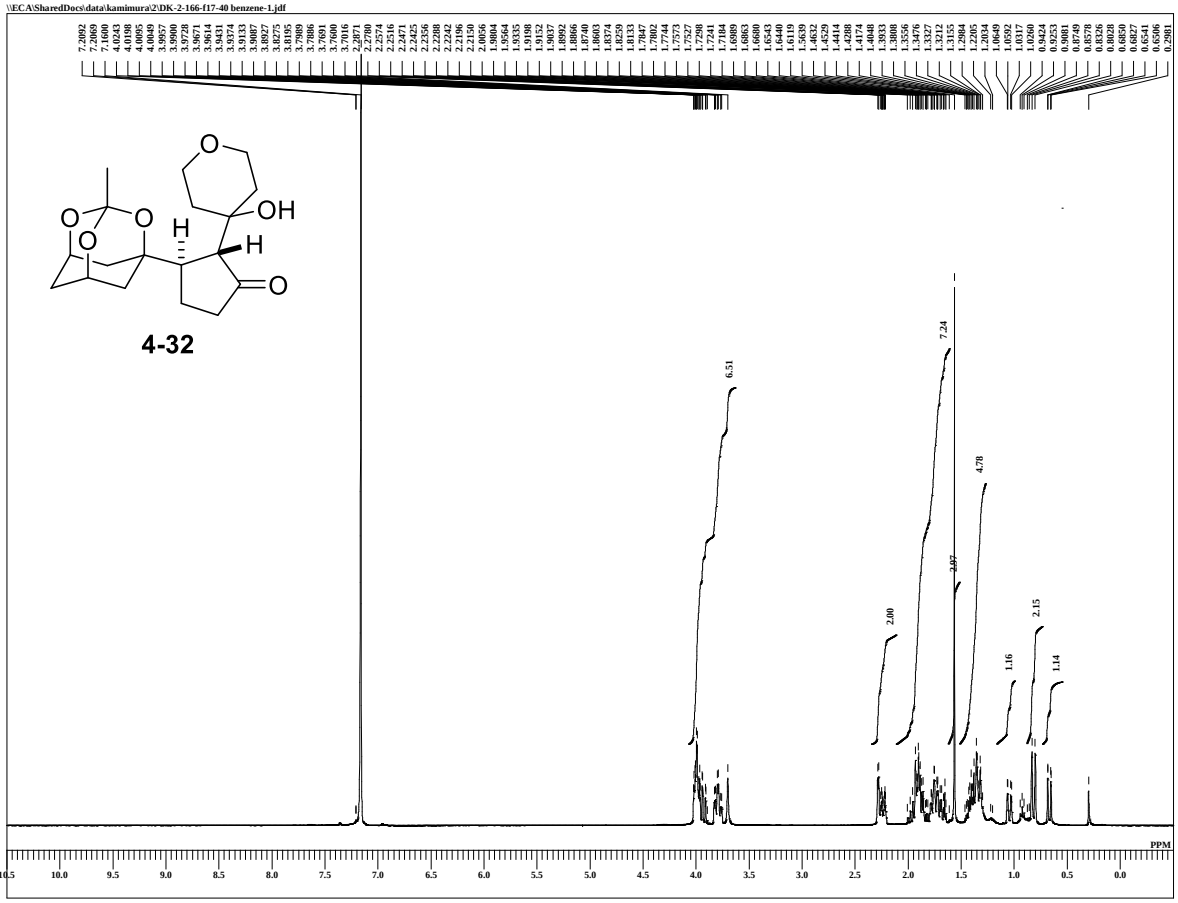


DFILE DK-2-167-f12-30 13C-1.jd
COMNT single pulse decoupled gat
DATIM 21-03-2013 07:37:44
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.25 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 7500
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 5.0000 sec
SCANS 7500
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-2-167-f12-30 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

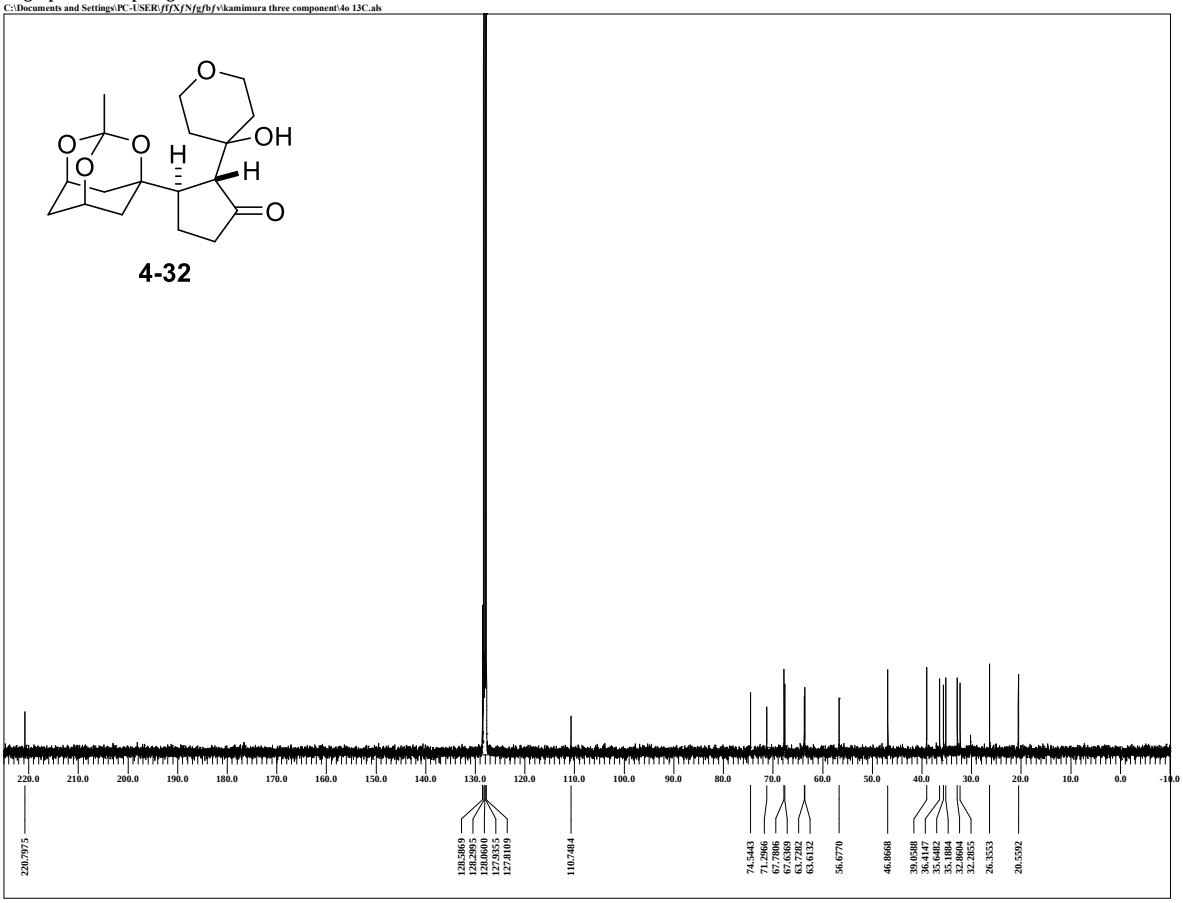
\\ECA\SharedDocs\data\kamimura\2\DK-2-166-f17-40 benzene-1.jdf



DFILE	DK-2-166-f17-40 benzene
COMNT	single_pulse
DATIM	28-02-2013 16:08:28
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-2-166-f17-40 benzene
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.2 c
SLVNT	C6D6
EXREF	7.16 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER*ffXfNfgfbf*v\kamimura three component\4o 13C.als

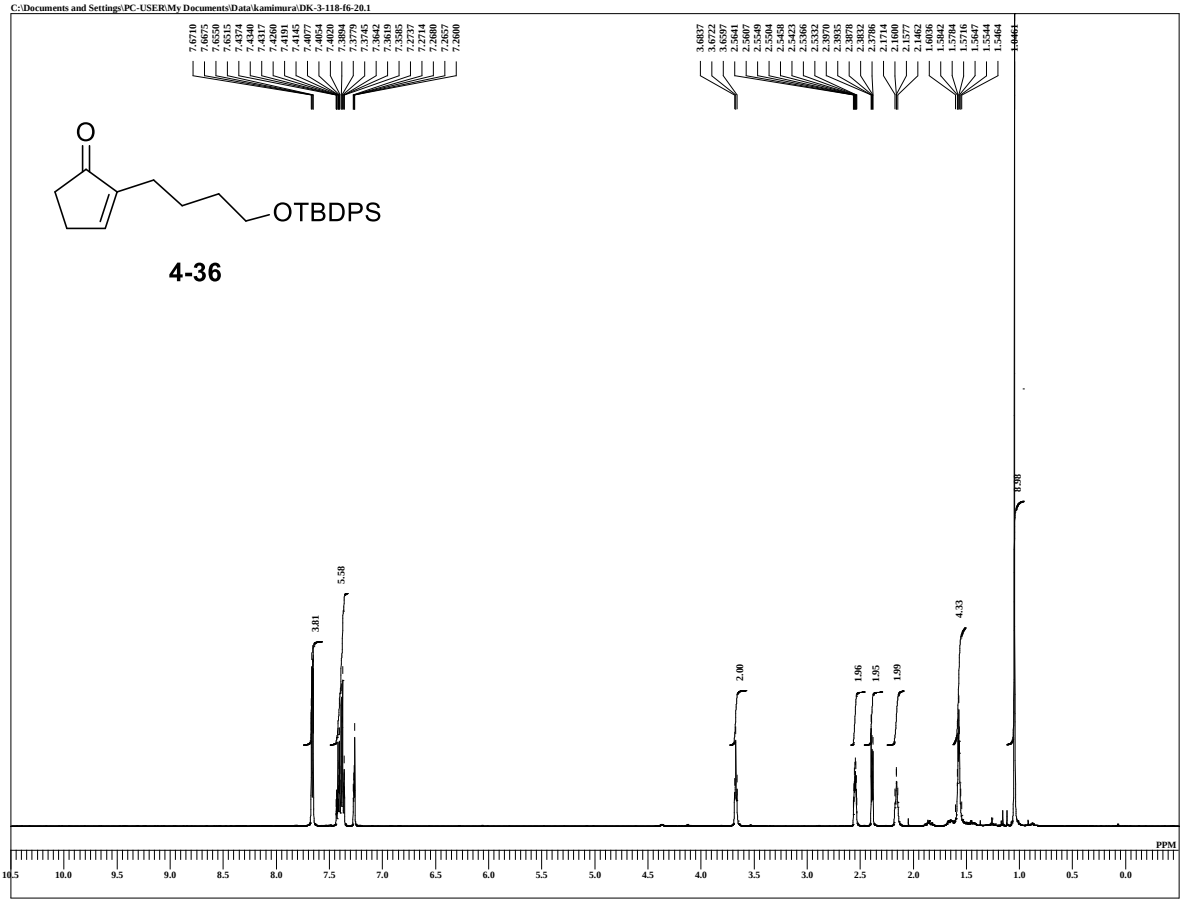


DFILE	4o 13C.als
COMNT	single pulse decoupled gat
DATIM	28-02-2013 16:15:56
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	113
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	113
ADBIT	16
RGAIN	58
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	4o 13C.als
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.4 c
SLVNT	C6D6
EXREF	128.06 ppm

スペクトルデータ

single_pulse

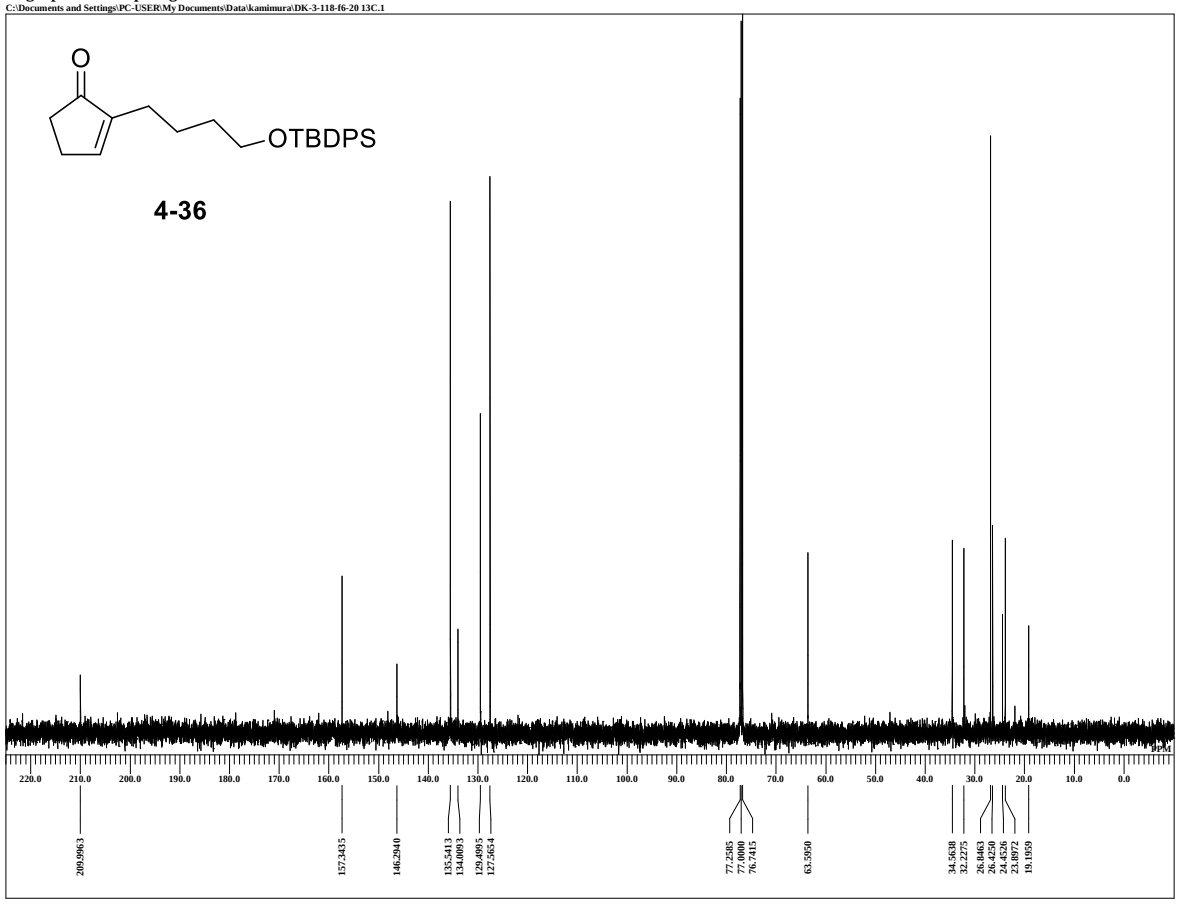
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-3-118-f6-20.1




```
DFILE    DK-3-118-f6-20.1
COMNT    single_pulse
DATIM    09-10-2013 15:15:04
MENUF
OBNUC    1H
OFR        495.13 MHz
OBFRQ      495.13 MHz
OBSET        4.38 KHz
OBFIN        9.64 Hz
PW1          6.00 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        16384
SPO          16384
TIMES        8
DUMMY        1
FREQU        9286.78 Hz
FLT          38000 Hz
DELAY        13.16 usec
ACQTM        1.7642 sec
PD           2.0000 sec
SCANS        8
ADBIT        16
RGAIN        40
BF           0.10 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR        495.13 MHz
IRSET        4.38 KHz
IRFIN        9.64 Hz
IRRPW        92 usec
IRATN        79
DFILE    DK-3-118-f6-20.1
SF
LKSET      748.40 KHz
LKFIN        98.2 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        22.9 c
SLVNT    CDCL3
EXREF        7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-3-118-f6-20 13C.1

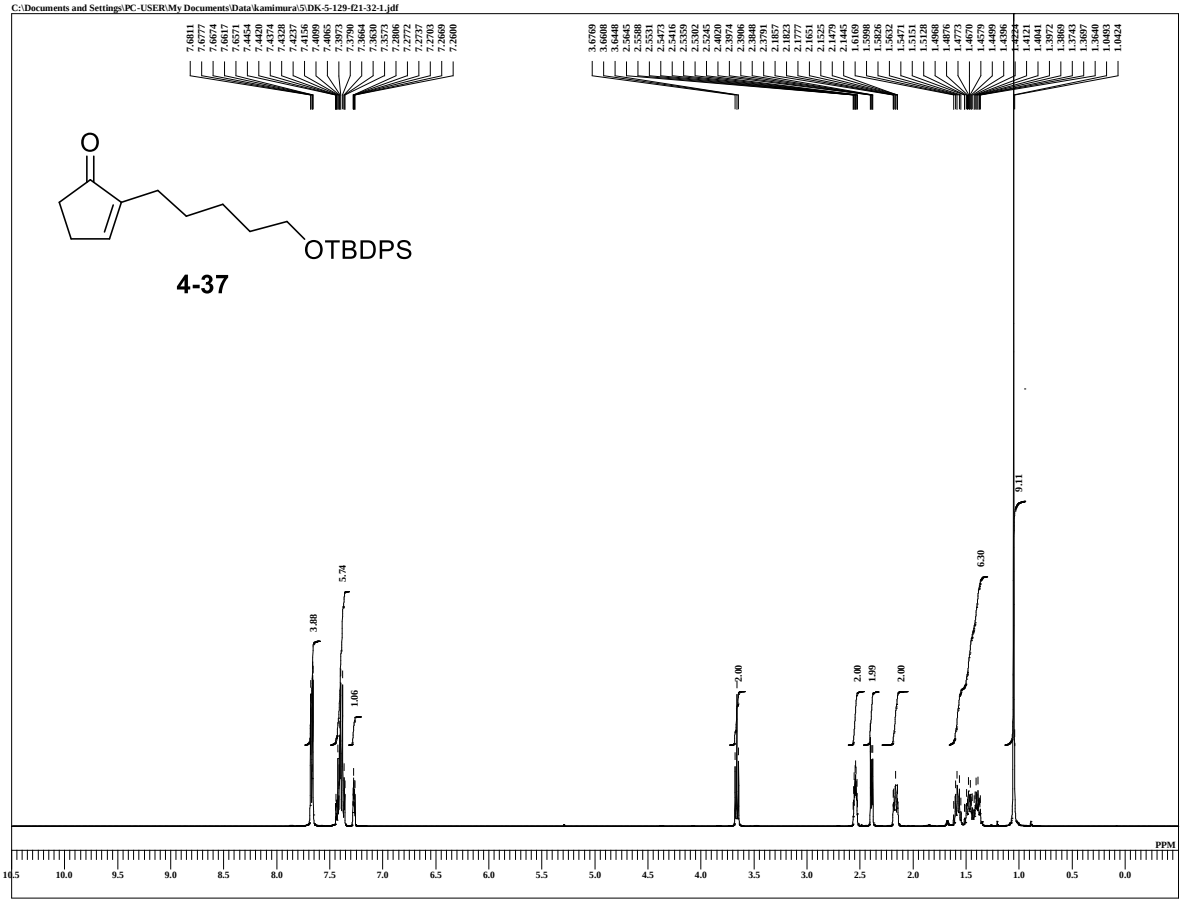



```
DFILE    DK-3-118-f6-20 13C.1
COMNT    single pulse decoupled gat
DATIM    09-10-2013 15:21:42
MENUF
OBNUC    13C
OFR        124.51 MHz
OBFRQ      124.51 MHz
OBSET        3.45 KHz
OBFIN        6.00 Hz
PW1          3.70 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        32768
SPO          32768
TIMES        118
DUMMY        4
FREQU        39062.50 Hz
FLT          157000 Hz
DELAY        20.80 usec
ACQTM        0.8389 sec
PD           2.0000 sec
SCANS        118
ADBIT        16
RGAIN        48
BF           1.00 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        495.13 MHz
IRSET        4.38 KHz
IRFIN        9.64 Hz
IRRPW        92 usec
IRATN        79
DFILE    DK-3-118-f6-20 13C.1
SF
LKSET      748.40 KHz
LKFIN        98.2 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        23.4 c
SLVNT    CDCL3
EXREF        77.00 ppm
```

スペクトルデータ

single_pulse

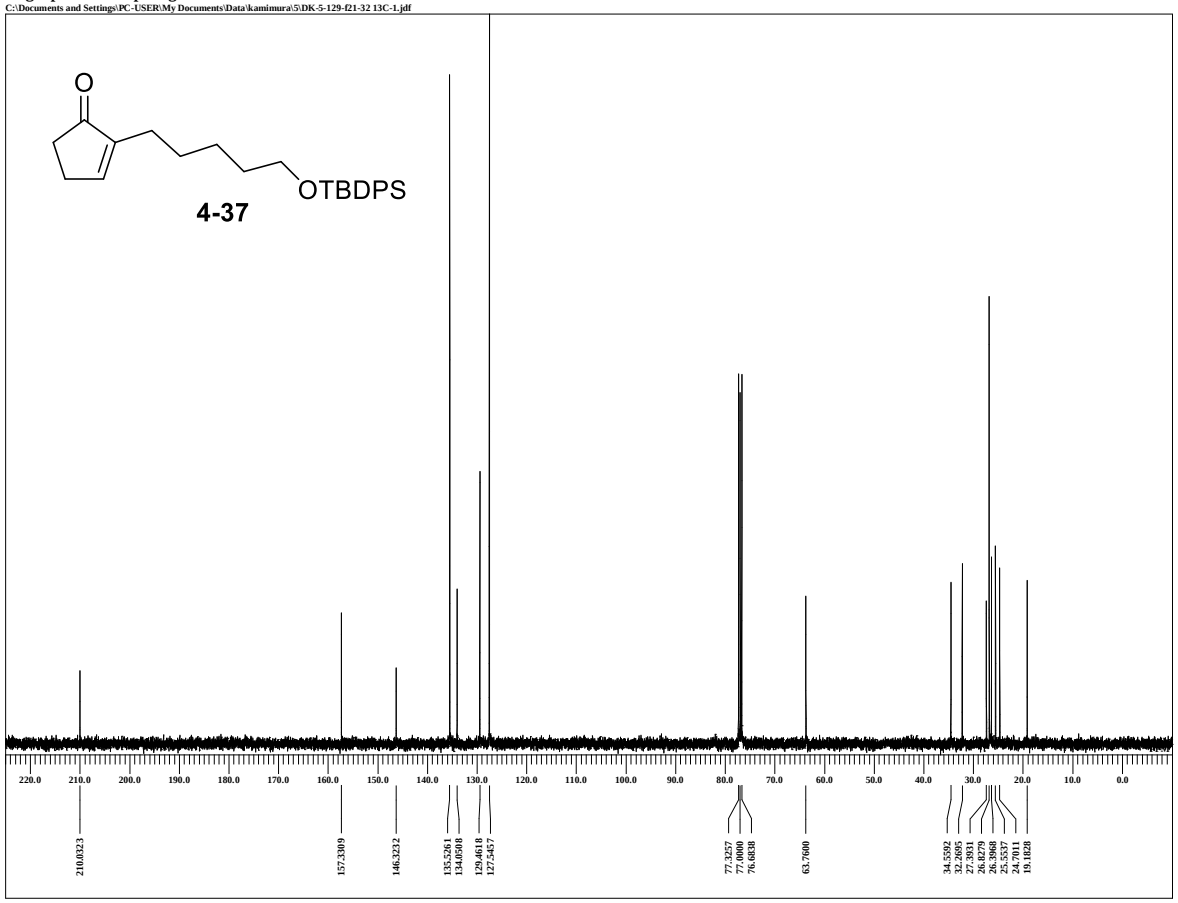
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-129-f21-32-1.jdf



DFILE DK-5-129-f21-32-1.jdf
COMNT single_pulse
DATIM 19-10-2013 09:11:25
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 26
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-129-f21-32-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.5 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-129-f21-32 13C-1.jdf

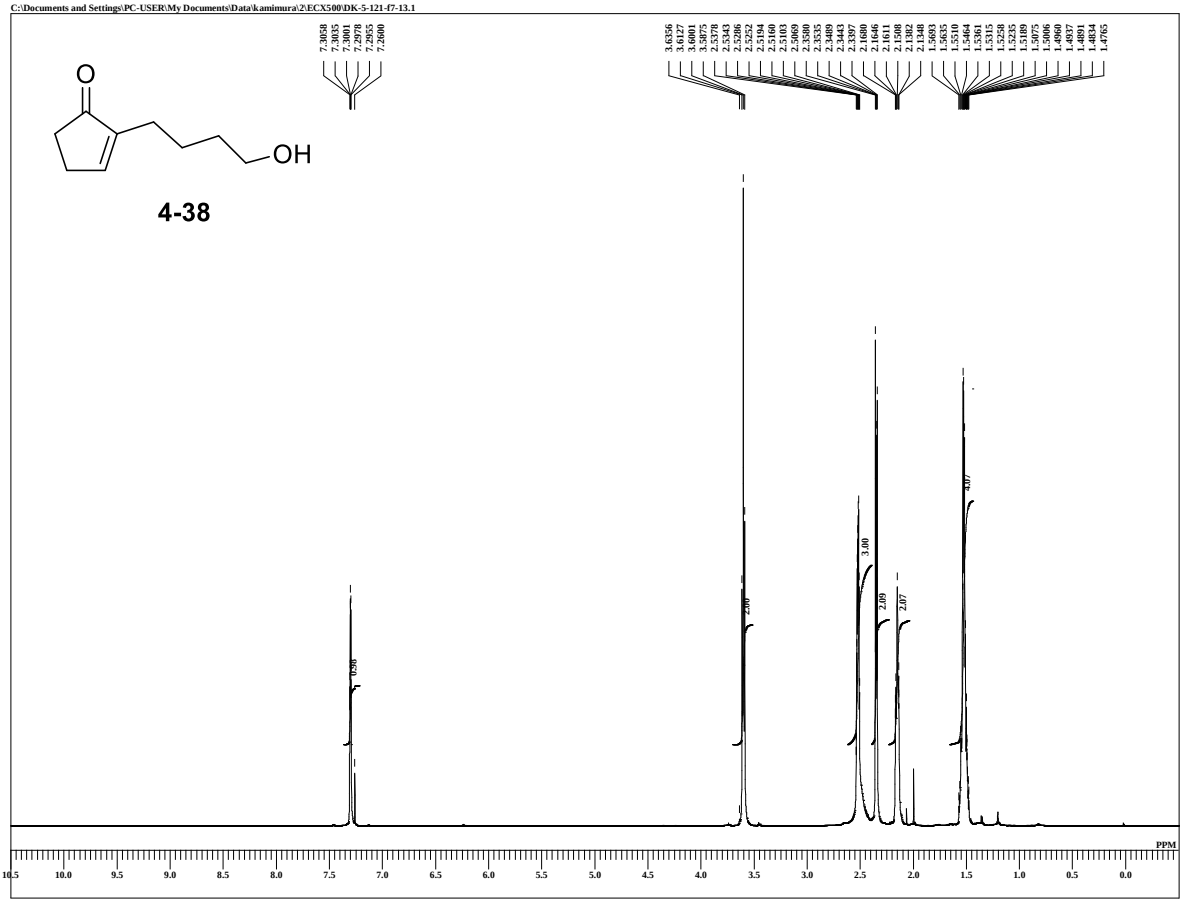


DFILE DK-5-129-f21-32 13C-1.jd
COMNT single pulse decoupled gat
DATIM 19-10-2013 09:16:02
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 79
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 79
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-129-f21-32 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.6 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

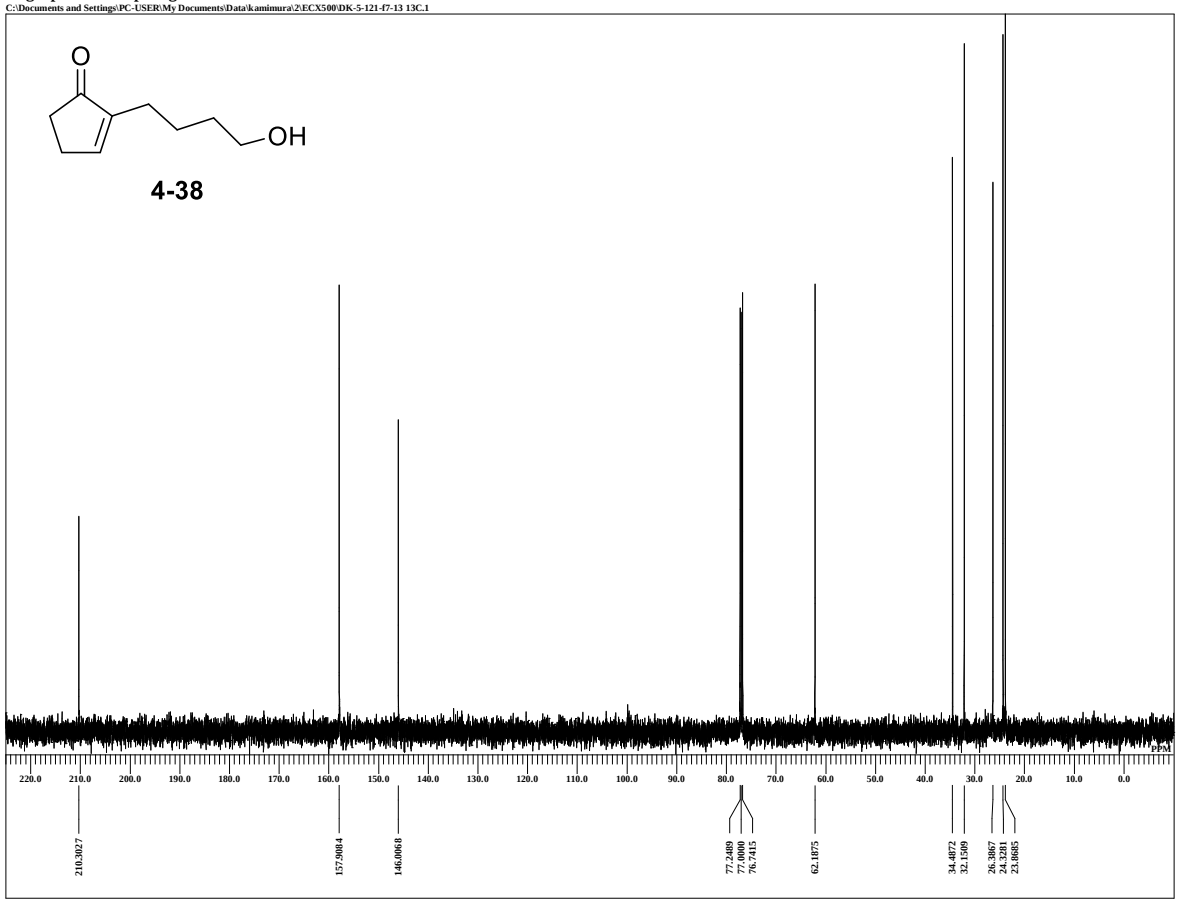
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-121-f7-13.1



DFILE DK-5-121-f7-13.1
COMNT single_pulse
DATIM 10-10-2013 15:46:36
MENUF
OBNUC 1H
OFR 495.13 MHz
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
PW1 6.00 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 9286.78 Hz
FLT 38000 Hz
DELAY 13.16 usec
ACQTM 1.7642 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE DK-5-121-f7-13.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.4 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\2\ECX500\DK-5-121-f7-13 13C.1

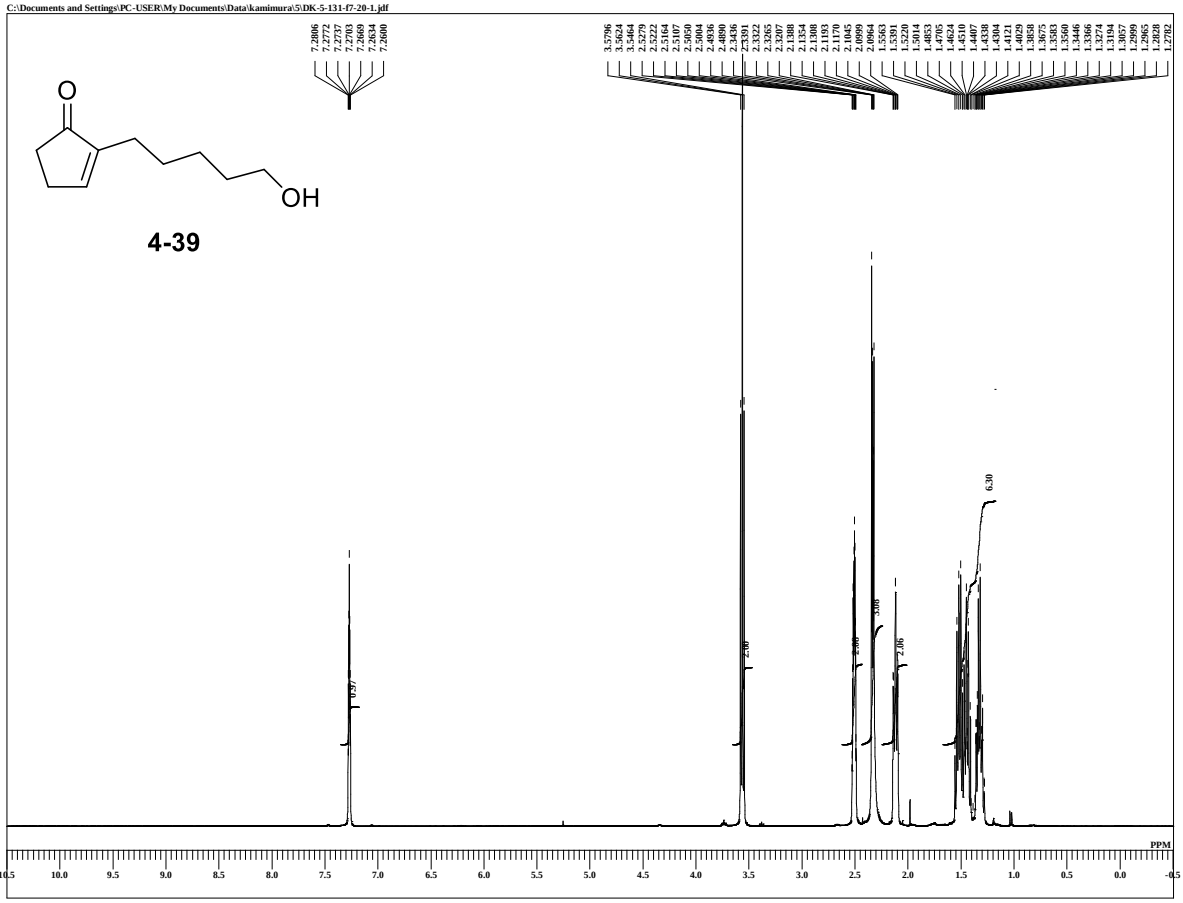


DFILE DK-5-121-f7-13 13C.1
COMNT single pulse decoupled gat
DATIM 10-10-2013 15:50:01
MENUF
OBNUC 13C
OFR 124.51 MHz
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
PW1 3.70 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 37
DUMMY 4
FREQU 39062.50 Hz
FLT 157000 Hz
DELAY 20.80 usec
ACQTM 0.8389 sec
PD 2.0000 sec
SCANS 37
ADBIT 16
RGAIN 48
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE DK-5-121-f7-13 13C.1
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

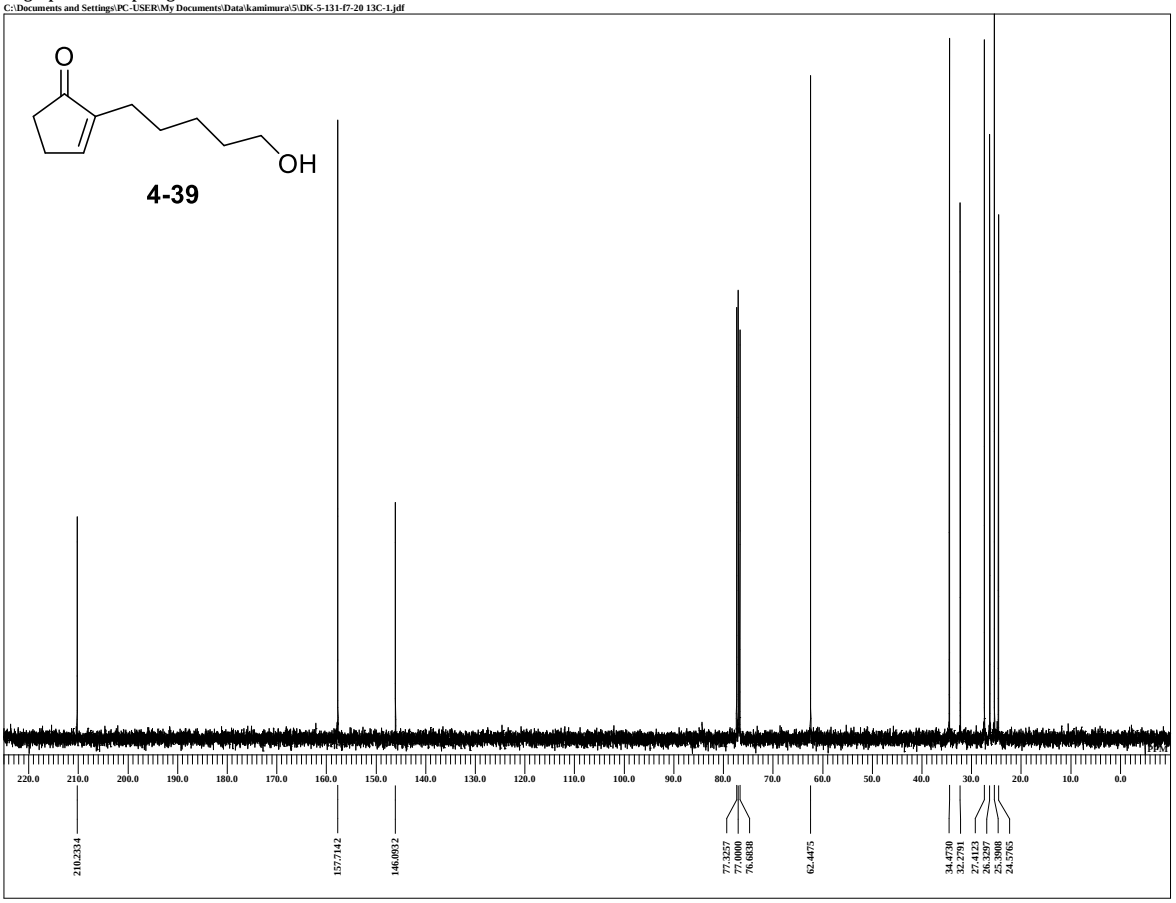
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-131-f7-20-1.jdf



DFILE DK-5-131-f7-20-1.jdf
COMNT single_pulse
DATIM 21-10-2013 17:20:40
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 24
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 1 usec
IRATN 10
DFILE DK-5-131-f7-20-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-131-f7-20 13C-1.jdf

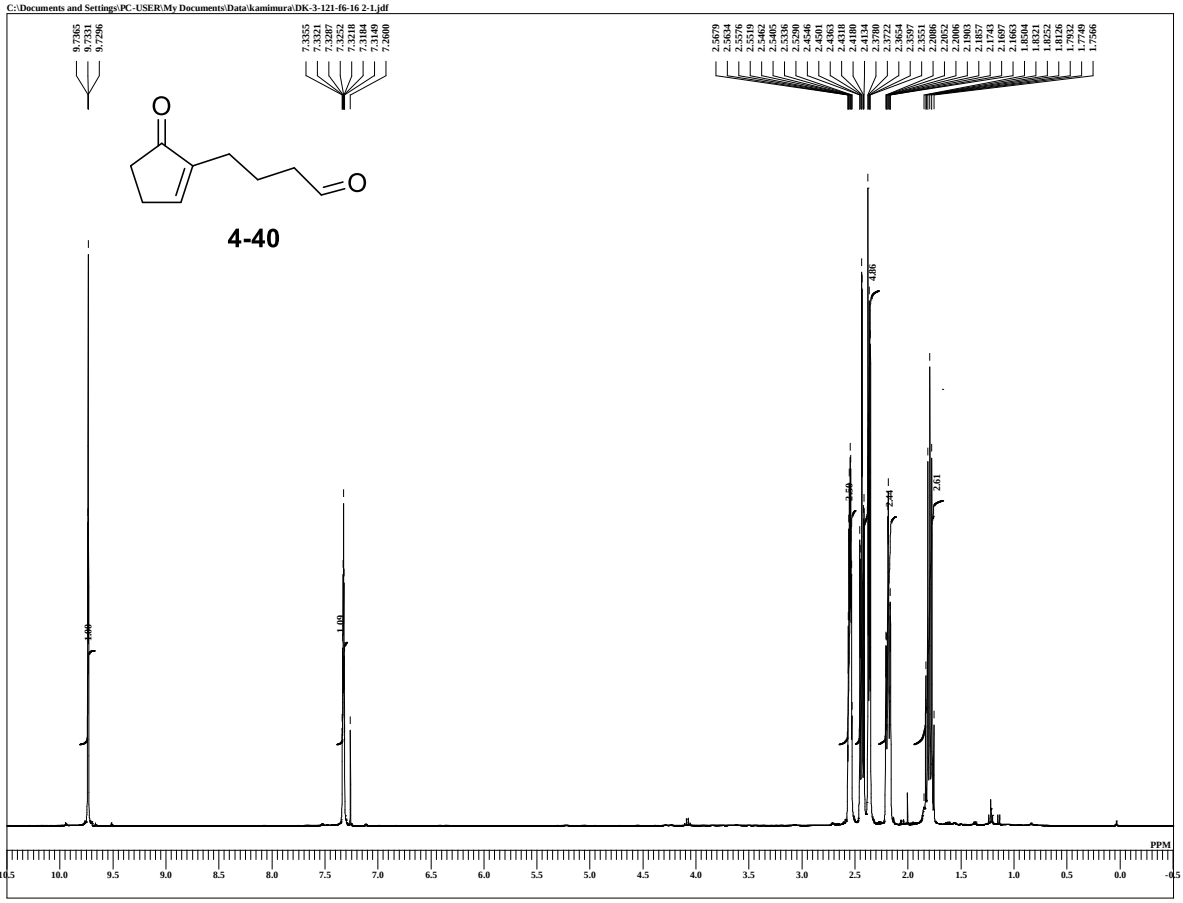


DFILE DK-5-131-f7-20 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 21-10-2013 17:23:44
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 49
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 49
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-131-f7-20 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.1 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

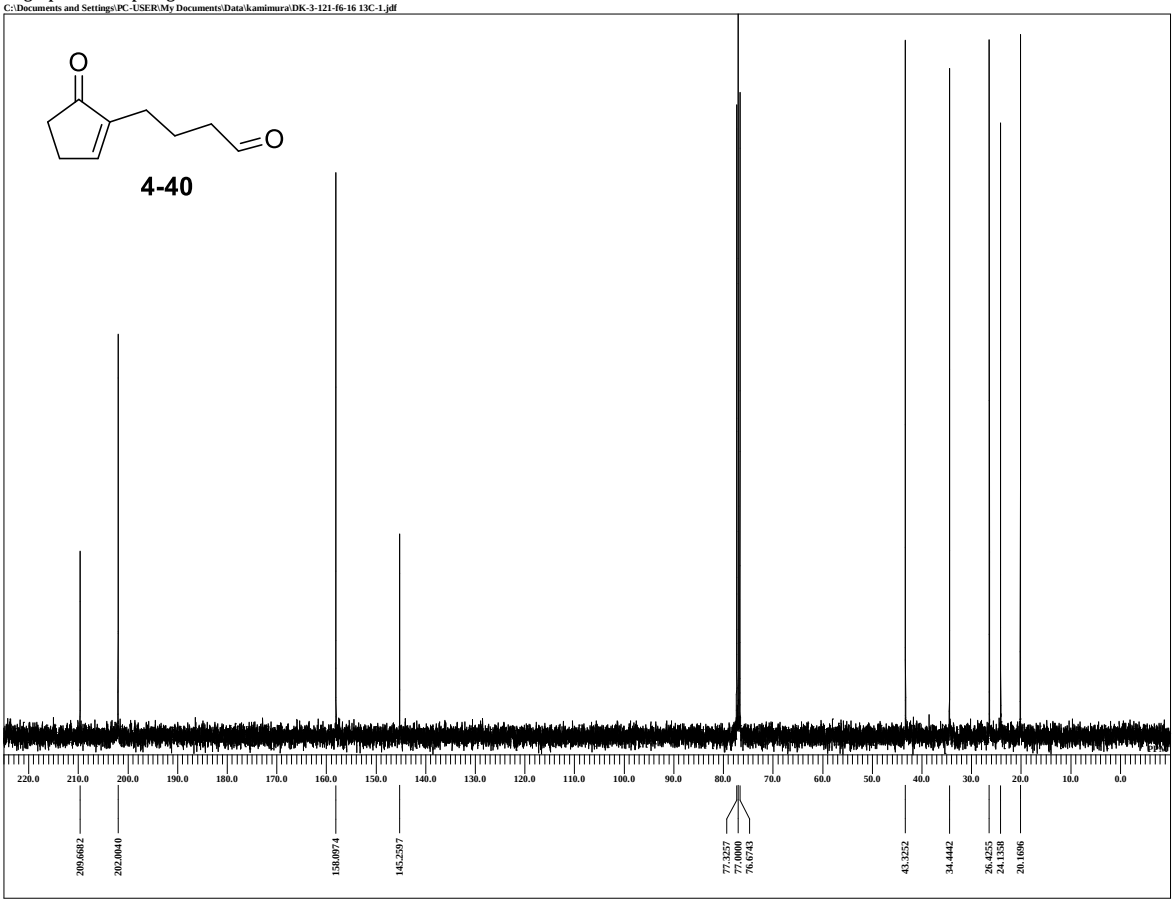
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-3-121-f6-16 2-1.jdf



DFILE DK-3-121-f6-16 2-1.jdf
COMNT single_pulse
DATIM 09-10-2013 18:16:11
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-121-f6-16 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.4 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-3-121-f6-16 13C-1.jdf

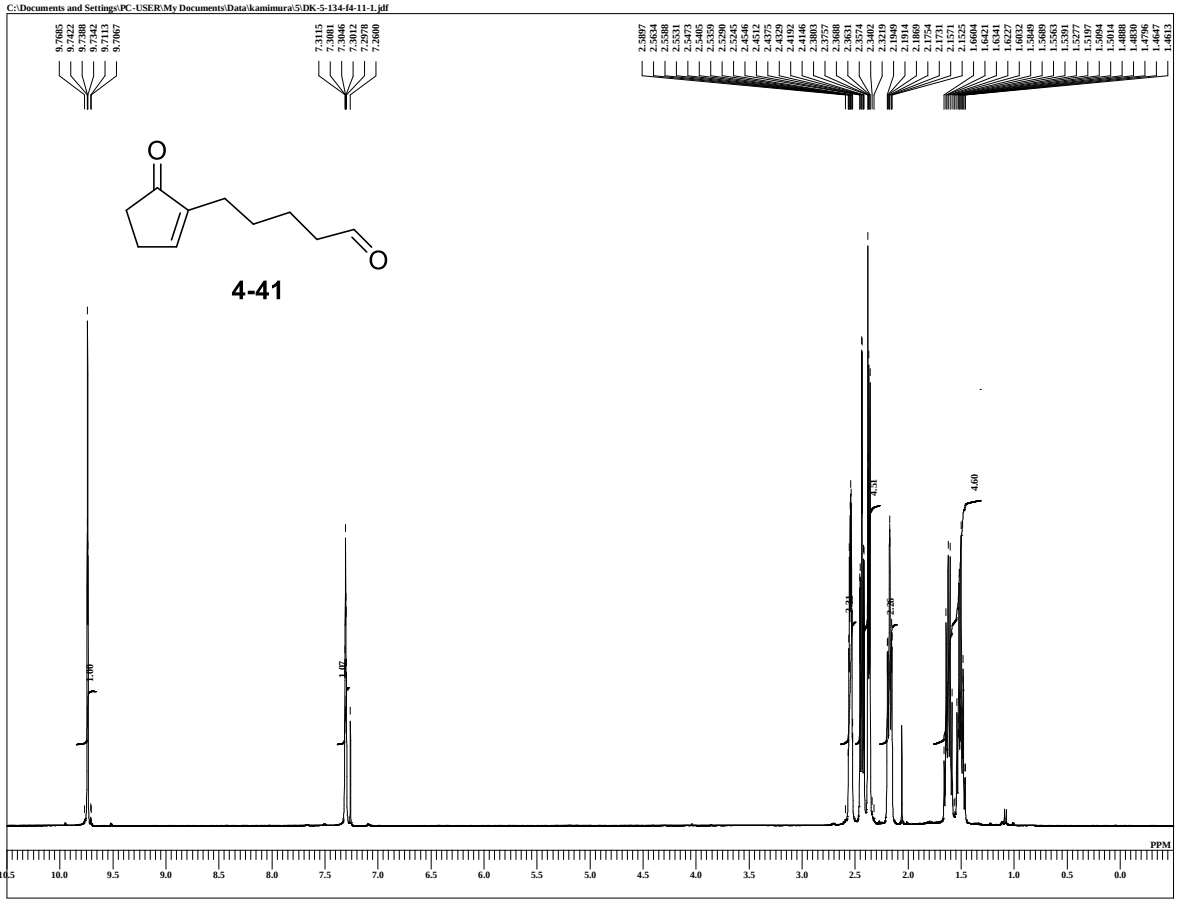


DFILE DK-3-121-f6-16 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 09-10-2013 18:20:28
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 73
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 73
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-3-121-f6-16 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

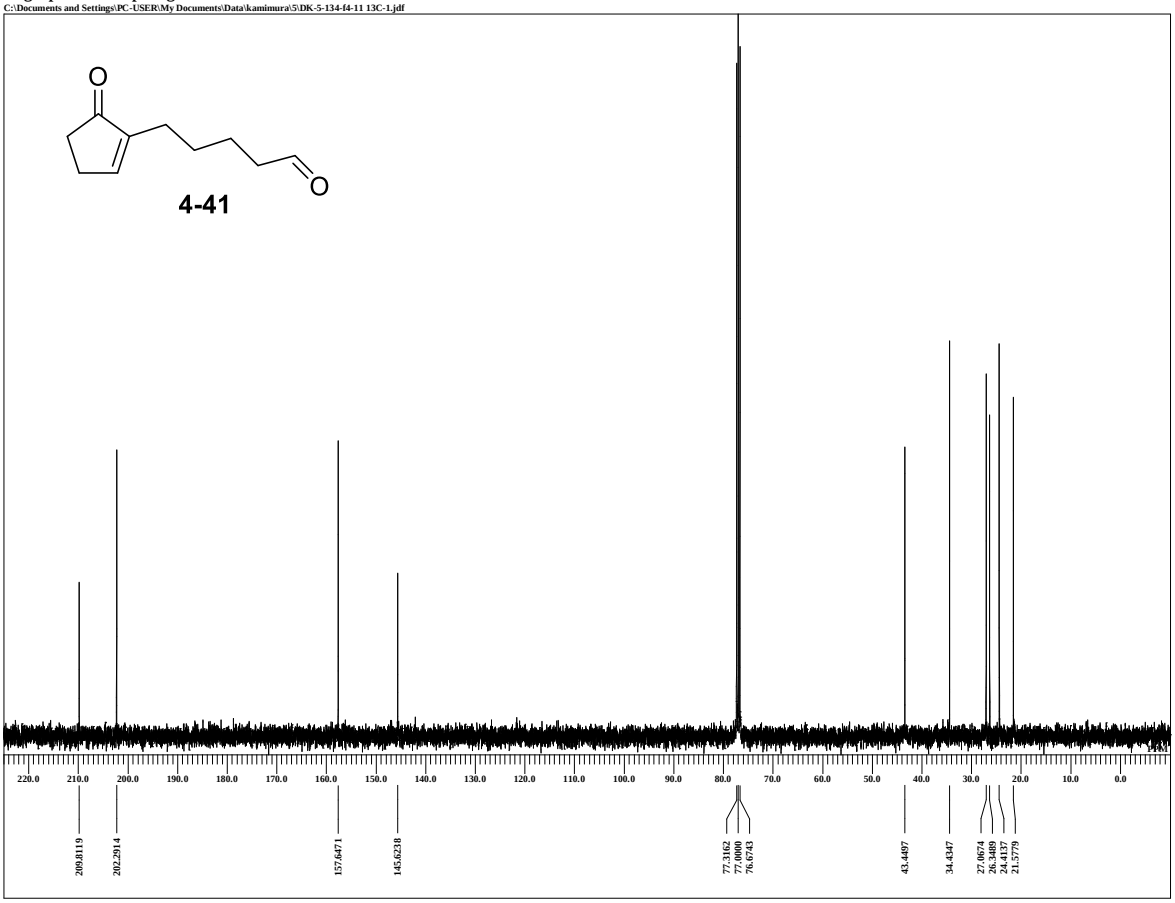
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-134-f4-11-1.jdf



DFILE DK-5-134-f4-11-1.jdf
COMNT single_pulse
DATIM 22-10-2013 16:34:21
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-134-f4-11-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.3 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-134-f4-11 13C-1.jdf

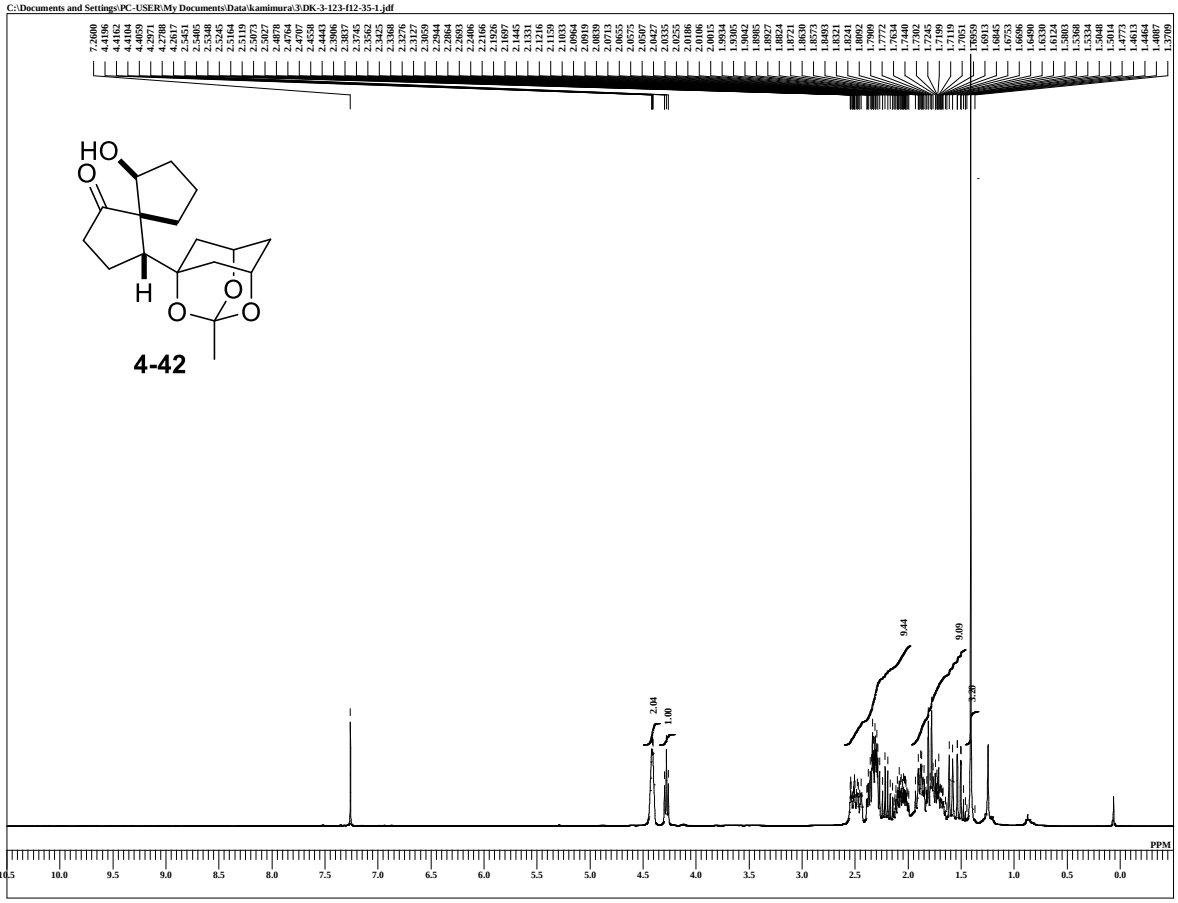


DFILE DK-5-134-f4-11 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 22-10-2013 16:37:13
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 43
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 43
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-134-f4-11 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.5 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

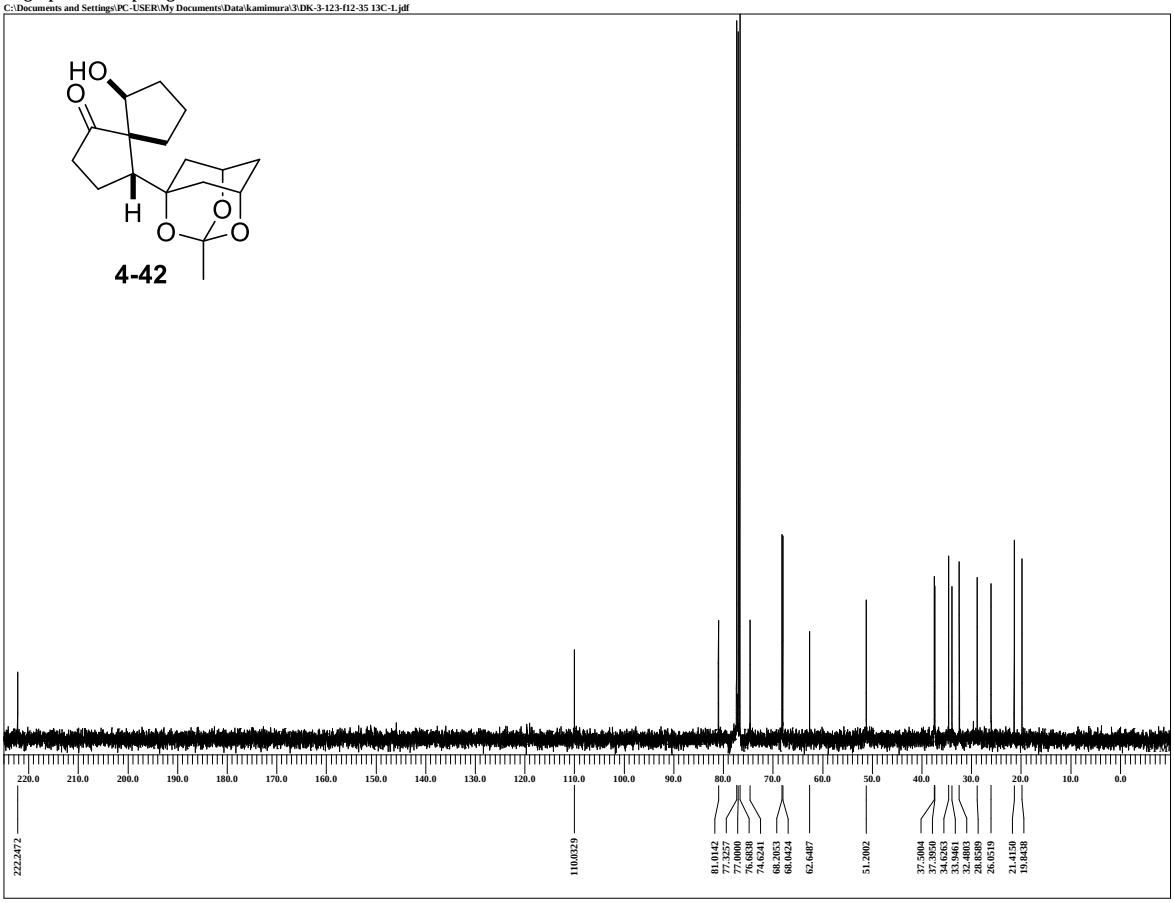
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-123-f12-35-1.jdf



DFILE	DK-3-123-f12-35-1.jdf
COMNT	single_pulse
DATIM	14-03-2013 14:29:41
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	32
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	32
ADBIT	16
RGAIN	36
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-123-f12-35-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\3\DK-3-123-f12-35 13C-1.jdf

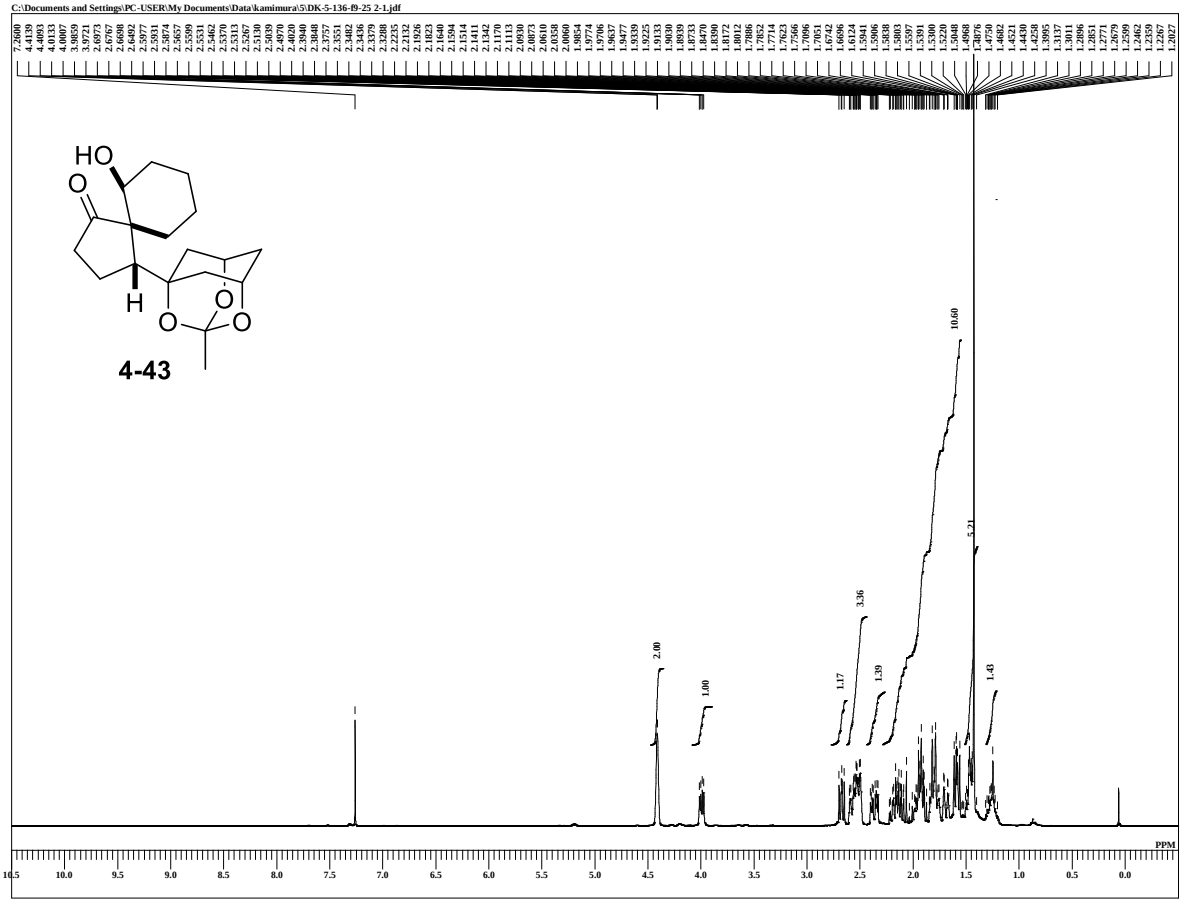


DFILE	DK-3-123-f12-35 13C-1.jd
COMNT	single pulse decoupled gat
DATIM	15-02-2013 15:01:46
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	152
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	152
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-3-123-f12-35 13C-1.jd
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

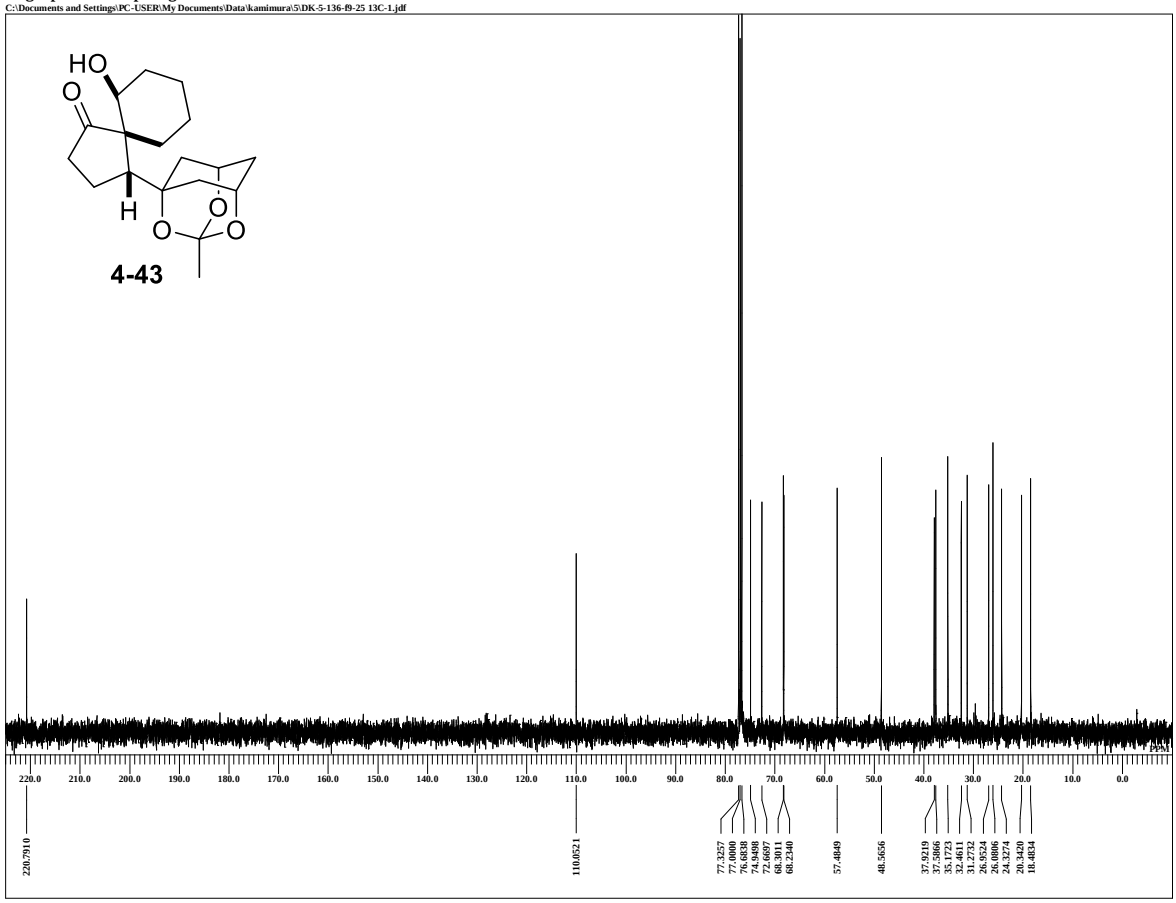
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-136-f9-25 2-1.jdf



DFILE	DK-5-136-f9-25 2-1.jdf
COMNT	single_pulse
DATIM	24-10-2013 11:15:15
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.44 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.10 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-136-f9-25 2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	25.7 c
SLVNT	CDCL3
EXREF	7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-136-f9-25 13C-1.jdf

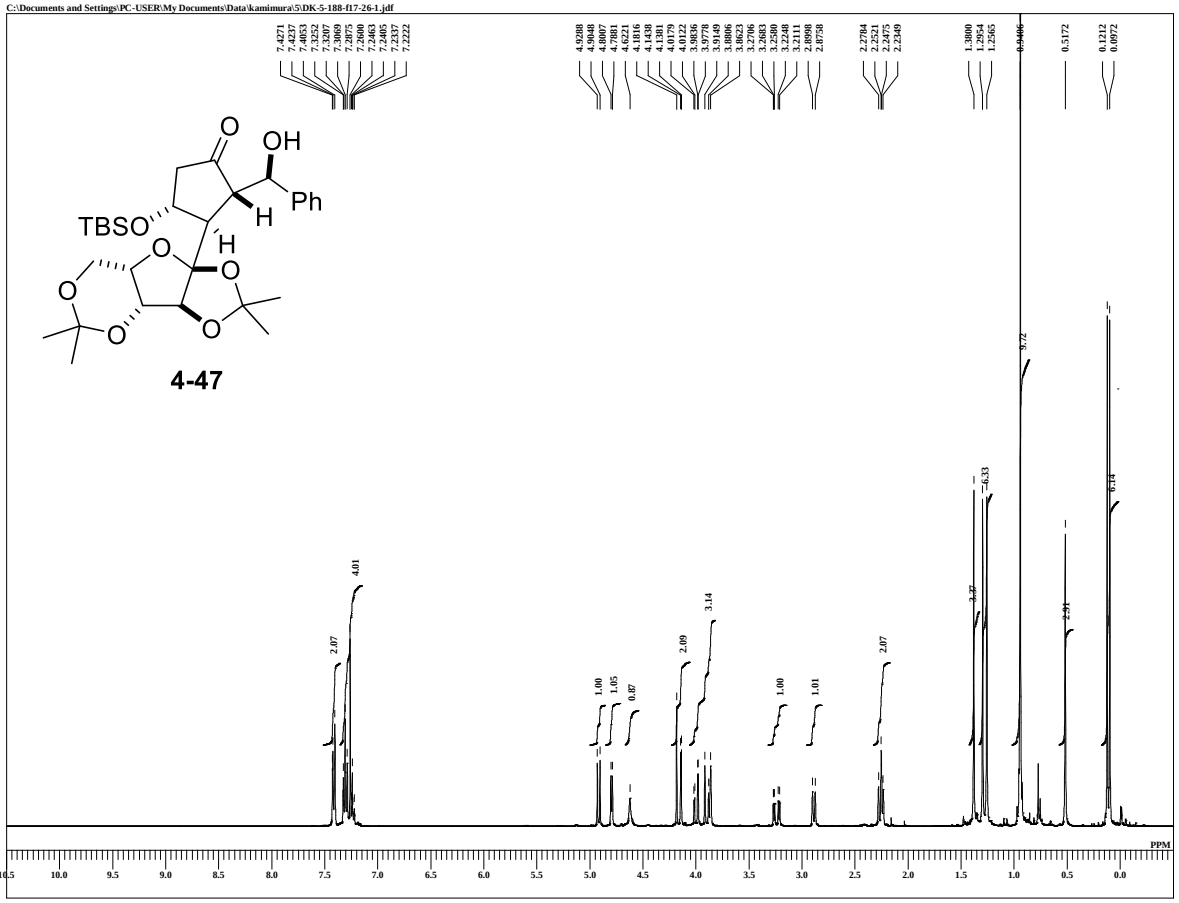


DFILE	DK-5-136-f9-25 13C-1.jdf
COMNT	single pulse decoupled gat
DATIM	23-10-2013 09:14:25
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.03 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	70
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	70
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-5-136-f9-25 13C-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	24.9 c
SLVNT	CDCL3
EXREF	77.00 ppm

スペクトルデータ

single_pulse

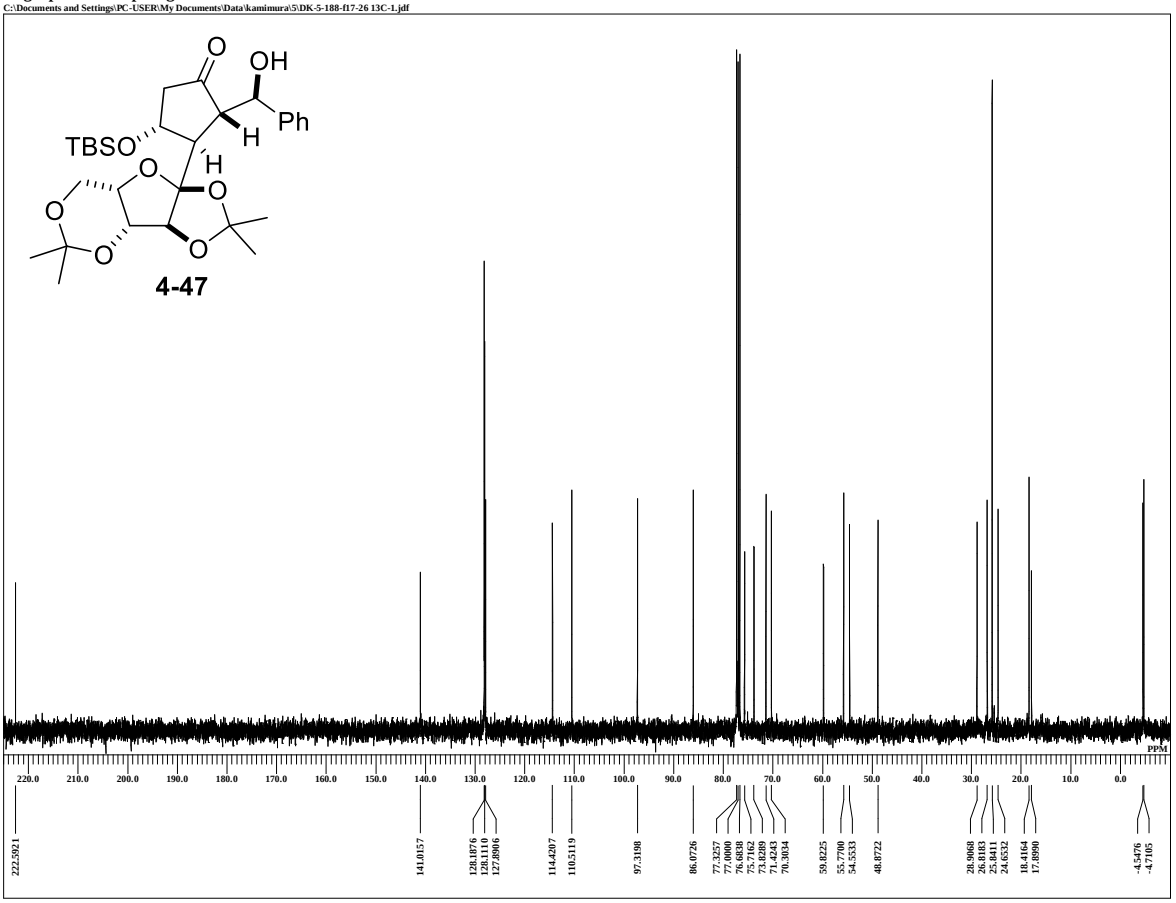
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-188-f17-26-1.jdf



DFILE DK-5-188-f17-26-1.jdf
COMNT single_pulse
DATIM 16-11-2013 16:16:25
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 26
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-188-f17-26-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.1 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-188-f17-26 13C-1.jdf

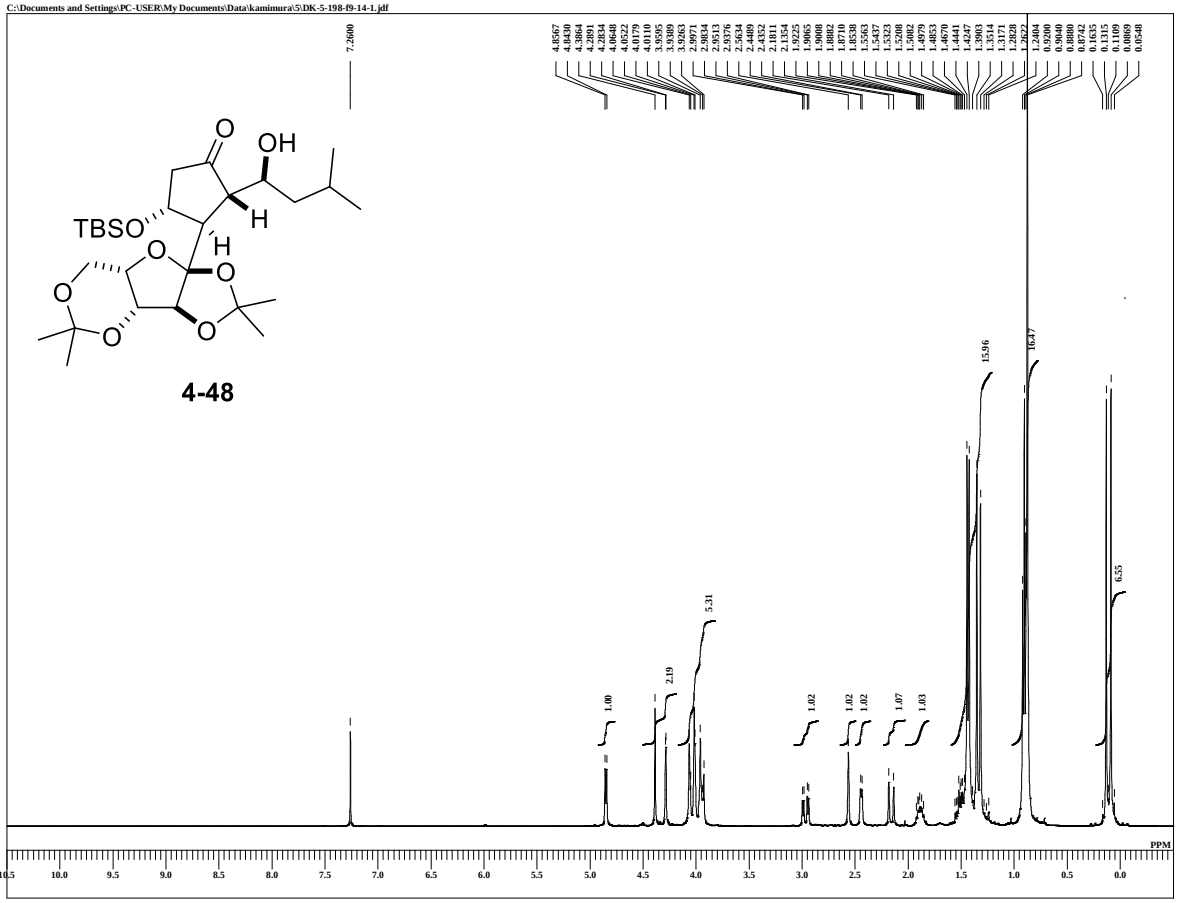


DFILE DK-5-188-f17-26 13C-1.jd
COMNT single pulse decoupled gat
DATIM 16-11-2013 16:20:52
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 76
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 76
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-188-f17-26 13C-1.jd
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

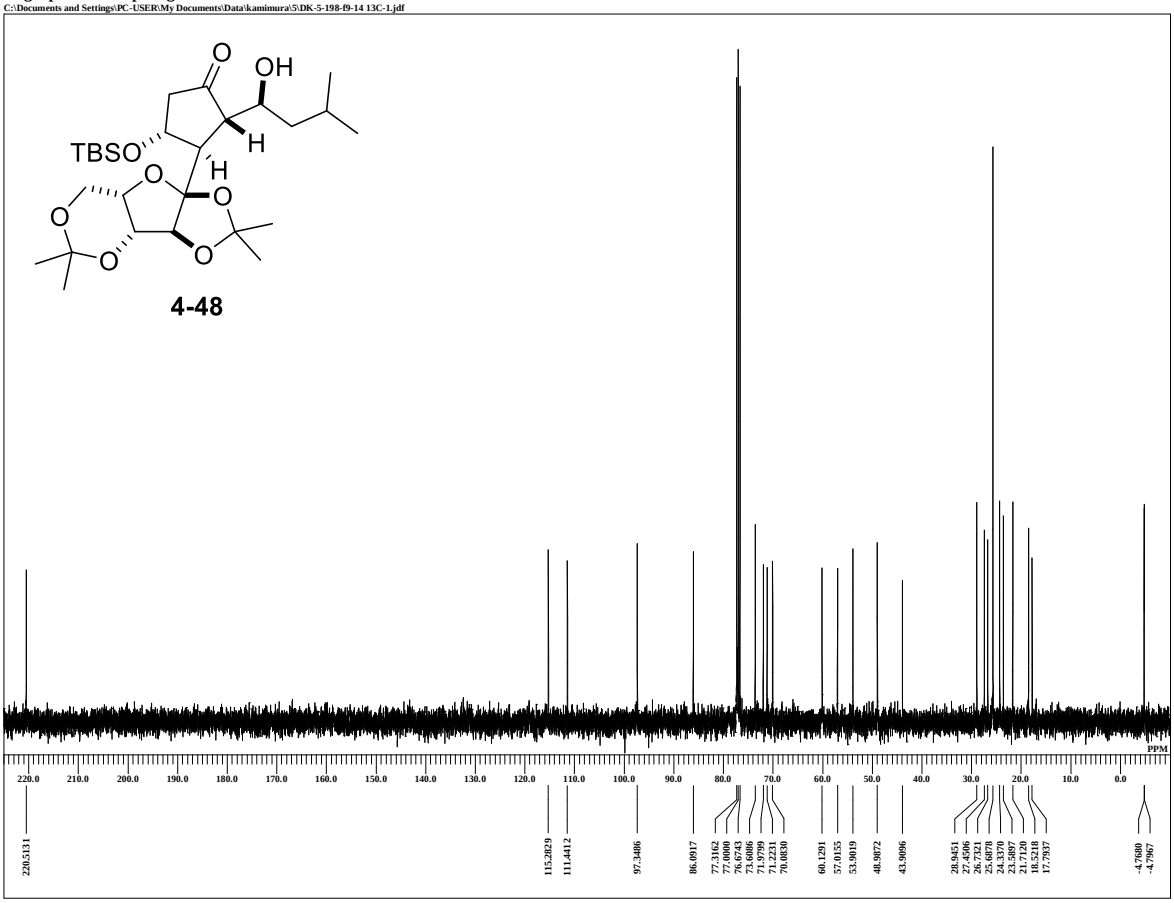
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-198-f9-14-1.jdf



DFILE DK-5-198-f9-14-1.jdf
COMNT single_pulse
DATIM 20-11-2013 11:00:19
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 26
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-198-f9-14-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 460.0 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-198-f9-14 13C-1.jdf

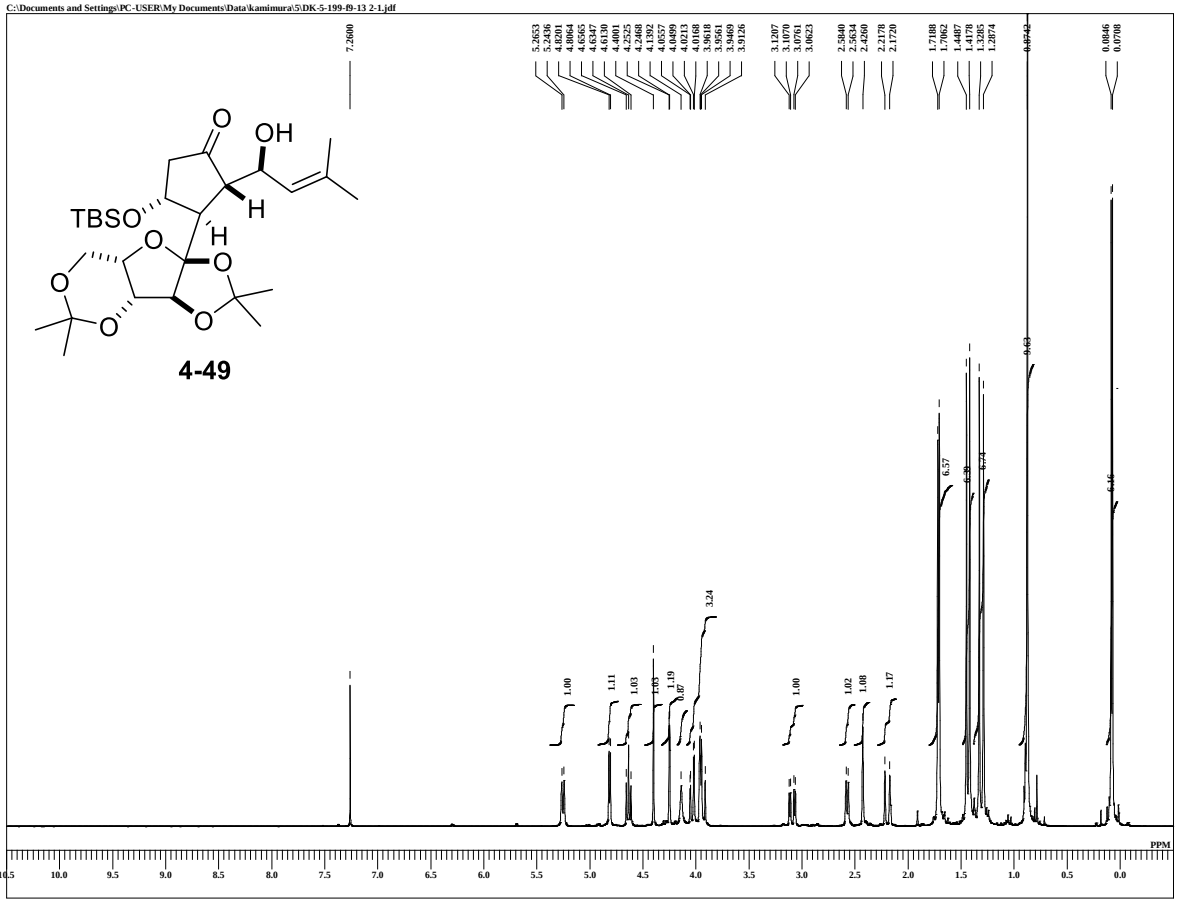


DFILE DK-5-198-f9-14 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 20-11-2013 11:03:35
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 51
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 51
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-198-f9-14 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 460.0 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-199-f9-13 2-1.jdf

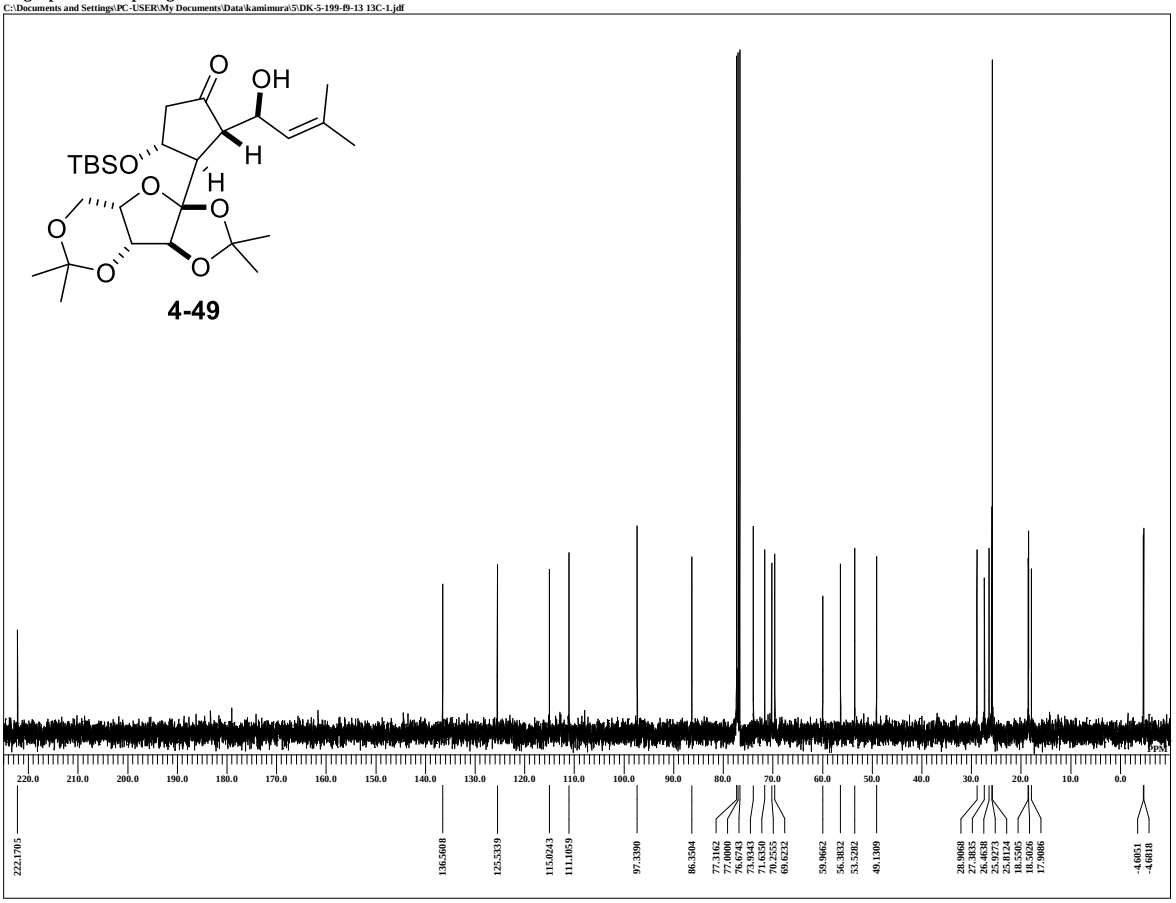


4-49

DFILE DK-5-199-f9-13 2-1.jdf
COMNT single_pulse
DATIM 20-11-2013 15:32:32
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 28
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-199-f9-13 2-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\5\DK-5-199-f9-13 13C-1.jdf



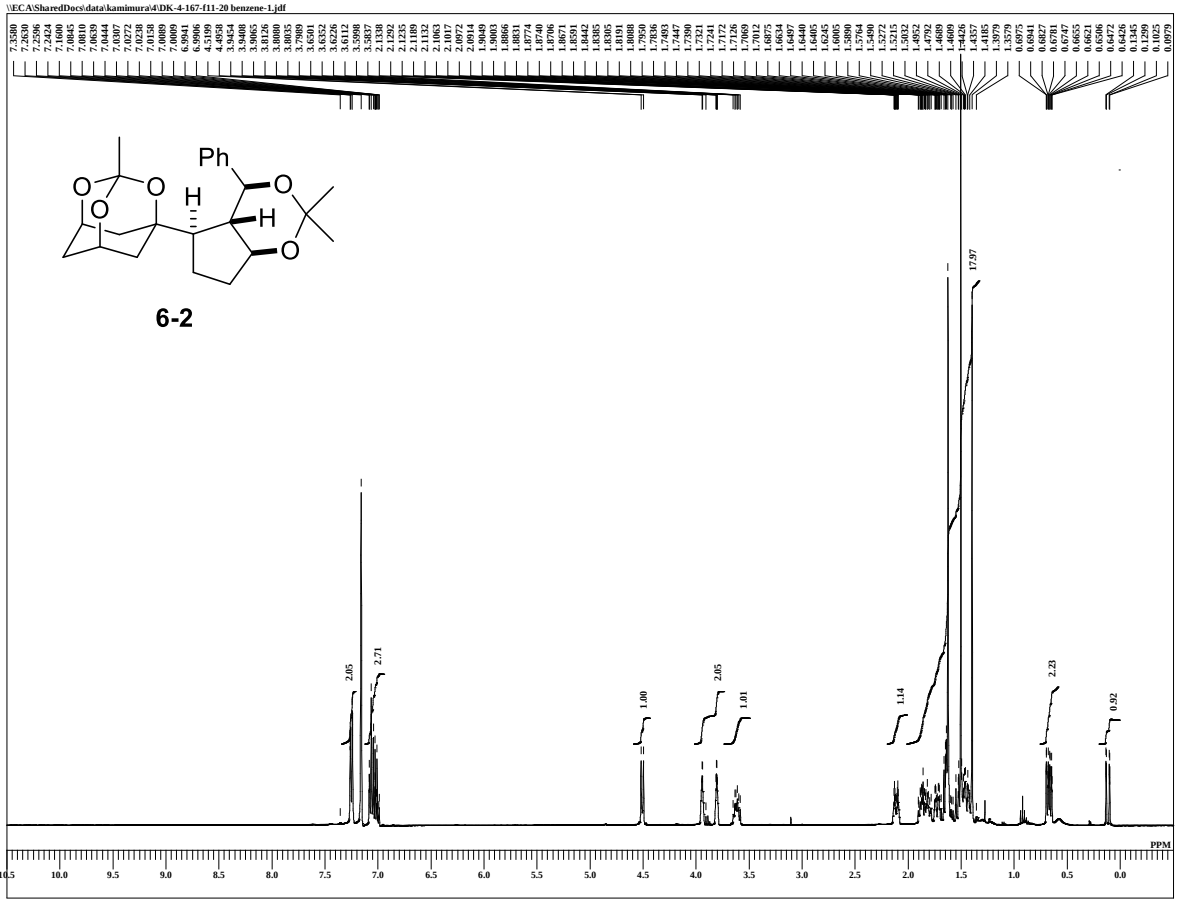
4-49

DFILE DK-5-199-f9-13 13C-1.jdf
COMNT single pulse decoupled gat
DATIM 20-11-2013 11:23:07
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 57
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 57
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-5-199-f9-13 13C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 460.0 c
SLVNT CDCL3
EXREF 77.00 ppm

スペクトルデータ

single_pulse

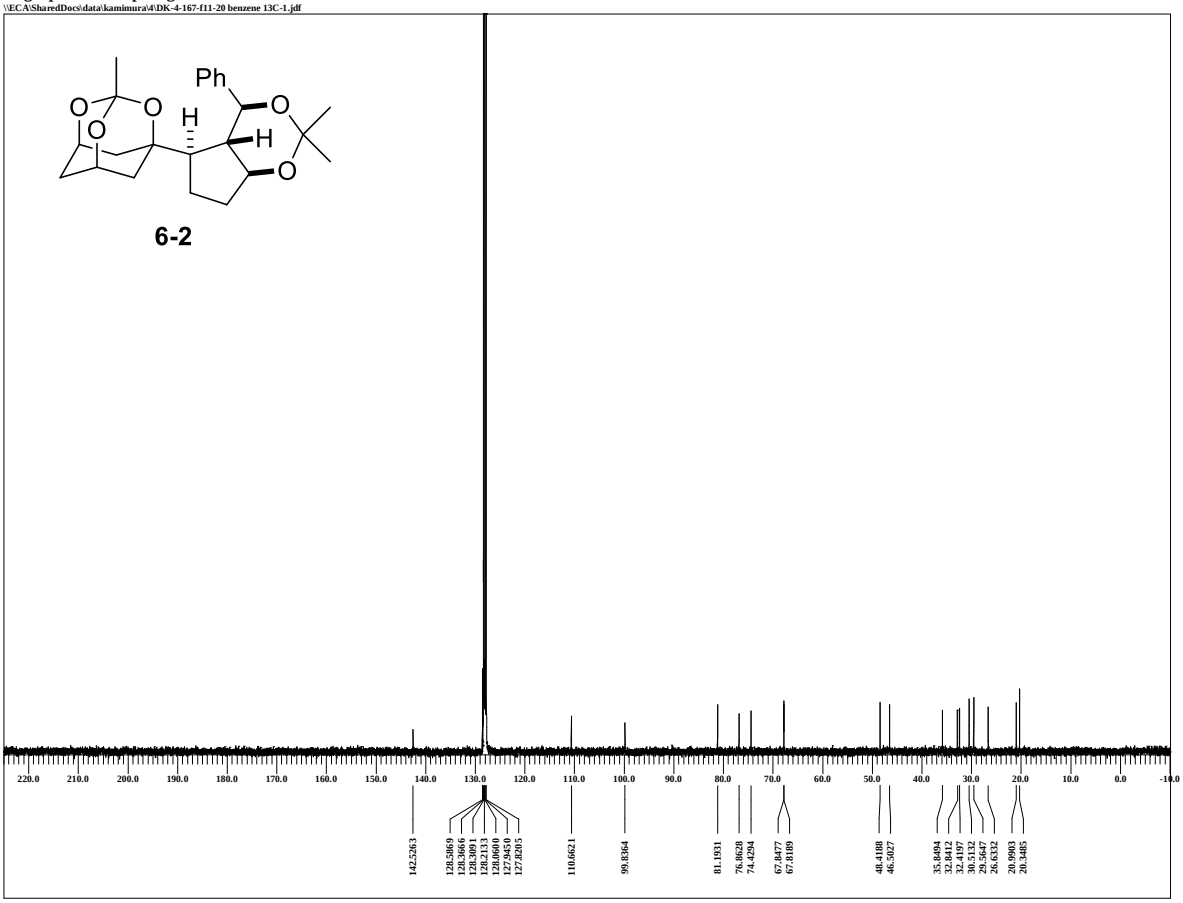
\\ECA\SharedDocs\data\kamimura\4\DK-4-167-f11-20 benzene-1.jdf



DFILE	DK-4-167-f11-20 benzene
COMNT	single_pulse
DATIM	10-07-2013 18:23:21
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	34
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-167-f11-20 benzene
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	24.3 c
SLVNT	C6D6
EXREF	7.16 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-167-f11-20 benzene 13C-1.jdf

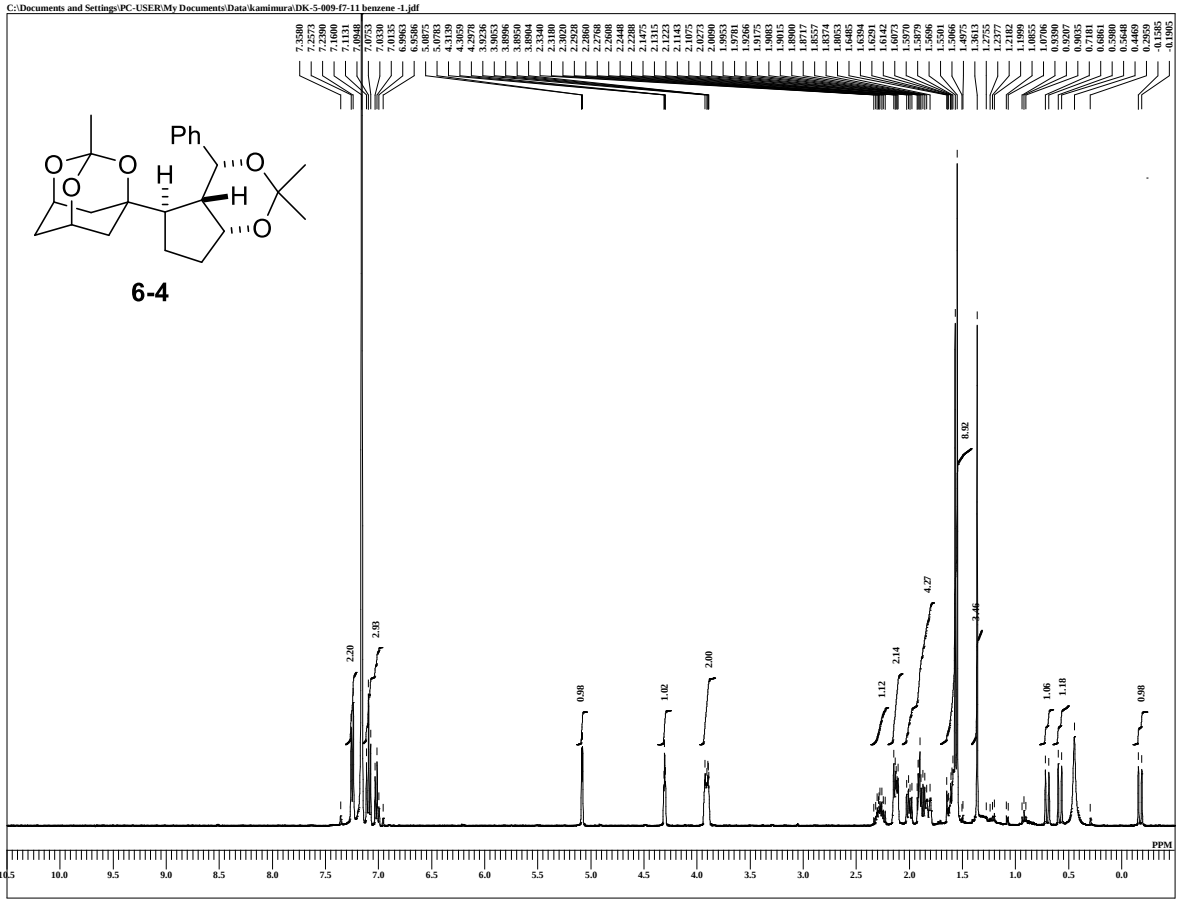


DFILE	DK-4-167-f11-20 benzene
COMNT	single pulse decoupled gat
DATIM	10-07-2013 19:02:48
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	2.90 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	113
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	113
ADBIT	16
RGAIN	58
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-167-f11-20 benzene
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	24.6 c
SLVNT	C6D6
EXREF	128.06 ppm

スペクトルデータ

single_pulse

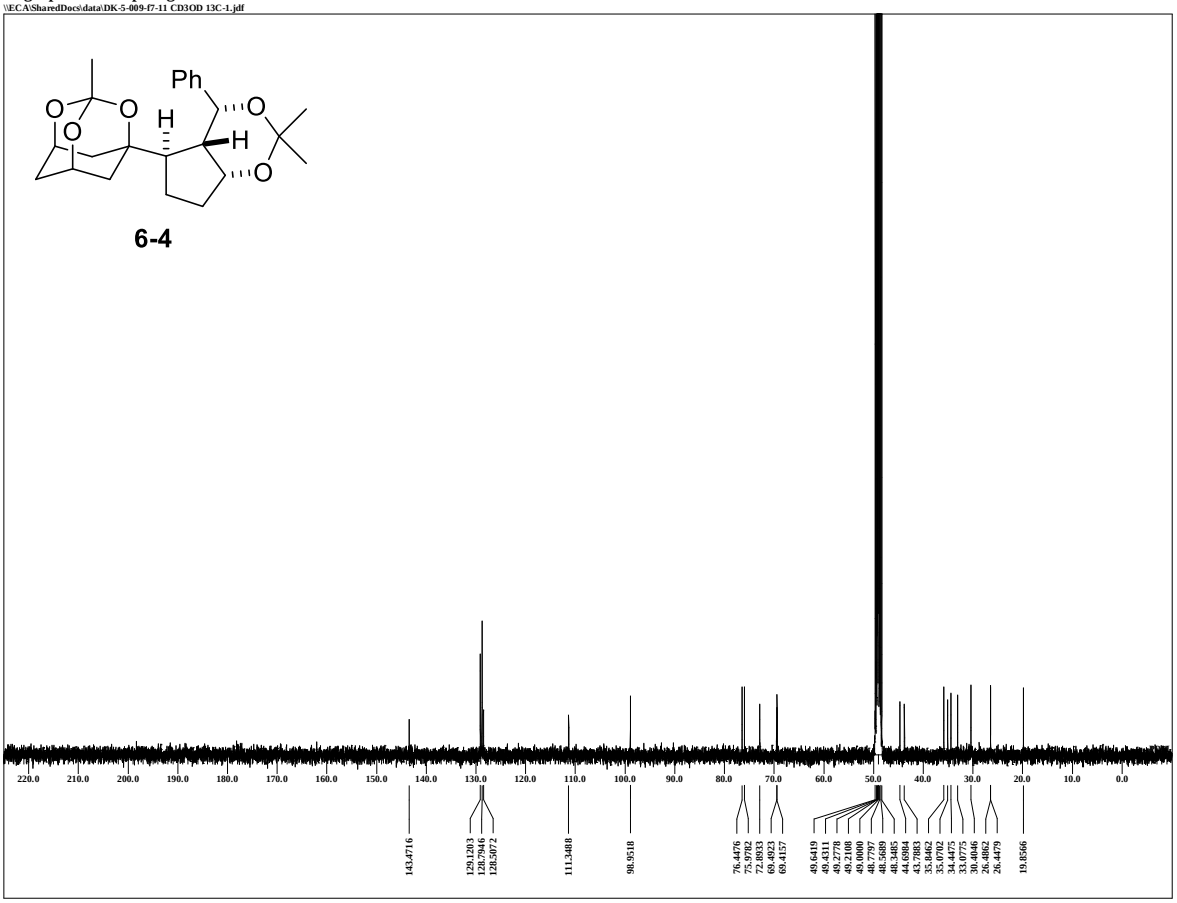
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-5-009-f7-11 benzene -1.jdf




```
DFILE    DK-5-009-f7-11 benzene -
COMNT    single_pulse
DATIM    04-08-2013 16:59:28
MENUF
OBNUC    1H
OFR        395.88 MHz
OBFRQ      395.88 MHz
OBSET        6.28 KHz
OBFIN        0.87 Hz
PW1          6.50 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        16384
SPO          16384
TIMES        16
DUMMY        1
FREQU      7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM        2.2073 sec
PD           2.0000 sec
SCANS        16
ADBIT        16
RGAIN        44
BF           0.01 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-5-009-f7-11 benzene -
SF
LKSET        13.20 KHz
LKFIN        69.6 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        23.1 c
SLVNT    C6D6
EXREF        7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\DK-5-009-f7-11 CD3OD 13C-1.jdf

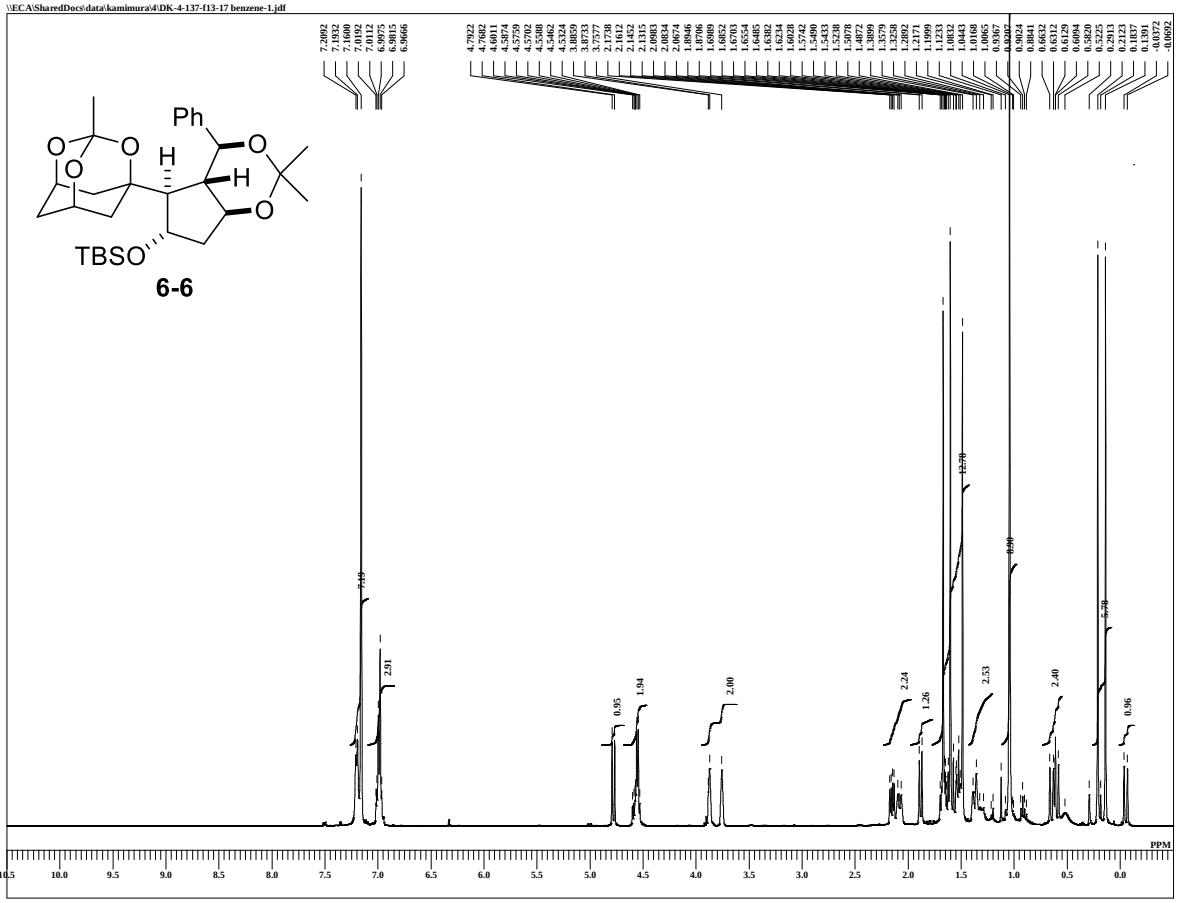



```
DFILE    DK-5-009-f7-11 CD3OD 1
COMNT    single pulse decoupled gat
DATIM    06-08-2013 09:32:11
MENUF
OBNUC    13C
OFR        99.55 MHz
OBFRQ      99.55 MHz
OBSET        5.13 KHz
OBFIN        0.98 Hz
PW1          2.90 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        32768
SPO          32768
TIMES        1200
DUMMY        4
FREQU      31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM        1.0486 sec
PD           2.0000 sec
SCANS        1200
ADBIT        16
RGAIN        60
BF           1.00 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-5-009-f7-11 CD3OD 1
SF
LKSET        13.00 KHz
LKFIN        35.6 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        23.7 c
SLVNT    CD3OD
EXREF        49.00 ppm
```

スペクトルデータ

single_pulse

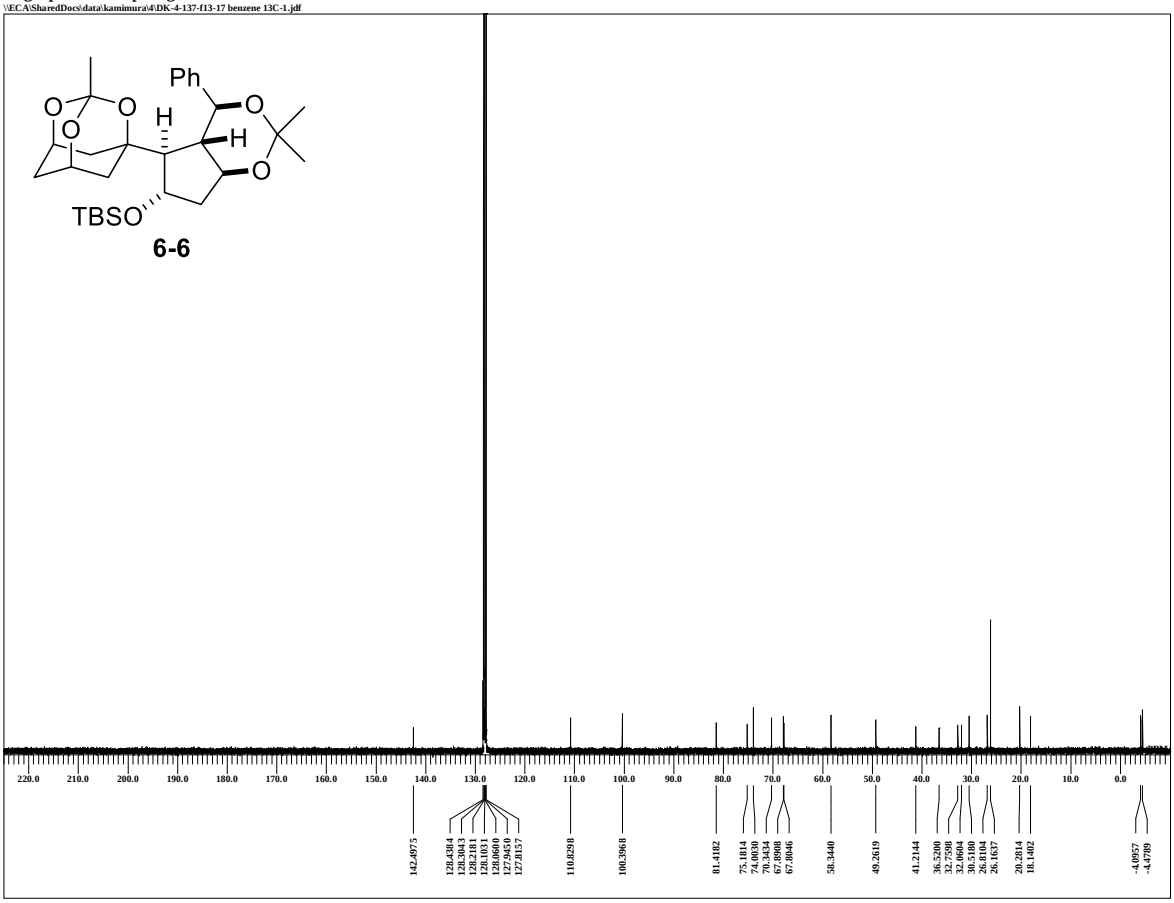
\\ECA\SharedDocs\data\kamimura\4\DK-4-137-f13-17 benzene-1.jdf




```
DFILE   DK-4-137-f13-17 benzene
COMNT   single_pulse
DATIM   07-07-2013 09:26:46
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.50 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       16384
SPO         16384
TIMES          32
DUMMY           1
FREQU    7422.80 Hz
FLT        30000 Hz
DELAY      16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS          32
ADBIT          16
RGAIN          34
BF          0.01 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW        147 usec
IRATN          79
DFILE   DK-4-137-f13-17 benzene
SF
LKSET     13.20 KHz
LKFIN      69.6 Hz
LKLEV          0
LGAIN          0
LKPHS          0
LKSIG          0
CSPED       0 Hz
FILDC
FILDF
CTEMP      21.4 c
SLVNT   C6D6
EXREF      7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-137-f13-17 benzene 13C-1.jdf

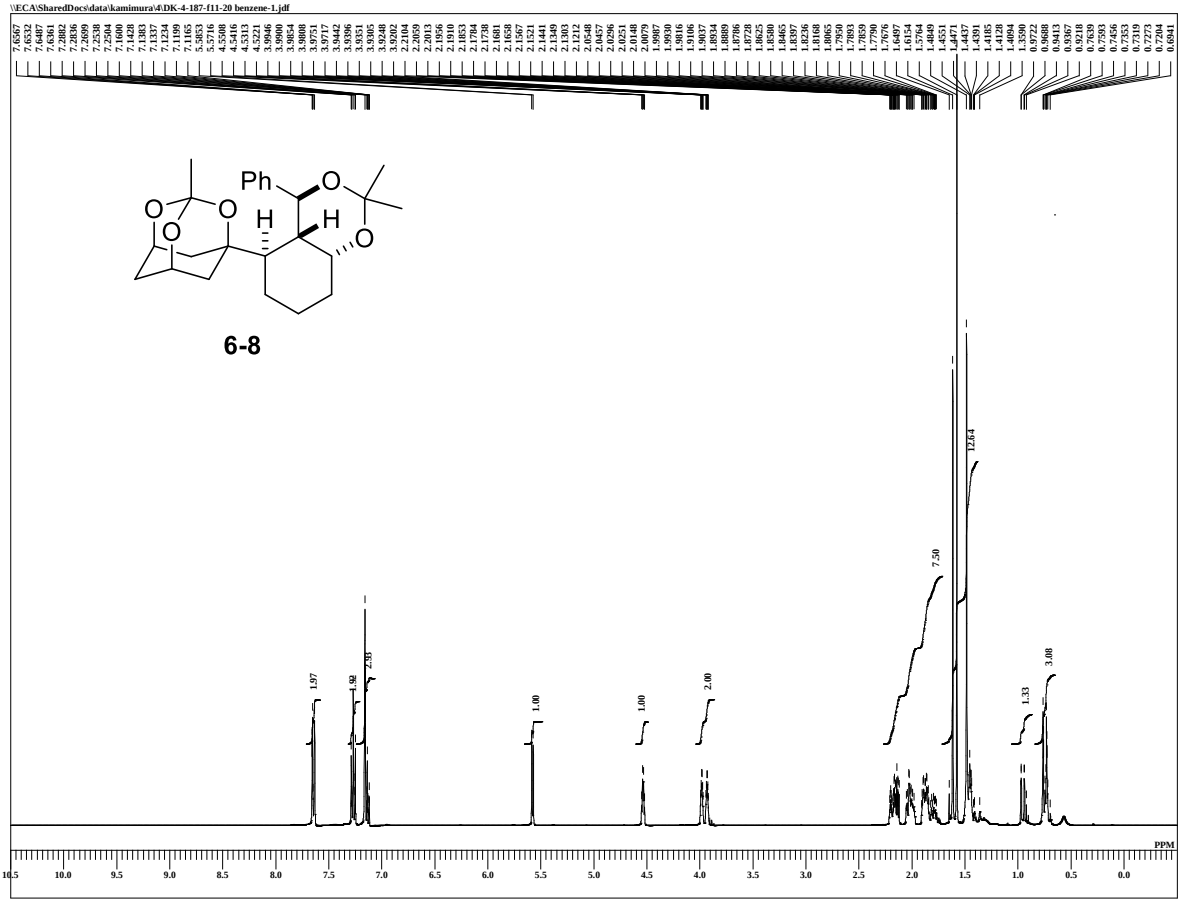



```
DFILE   DK-4-137-f13-17 benzene
COMNT   single pulse decoupled gat
DATIM   07-07-2013 10:07:44
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
PW1        2.90 usec
DEADT      0.00 usec
PREDL      0.00000 msec
IWT        1.0000 sec
POINT      65536
SPO        65536
TIMES        500
DUMMY          4
FREQU   31250.00 Hz
FLT       125000 Hz
DELAY     20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS        500
ADBIT         16
RGAIN         58
BF         0.01 Hz
T1         0.00
T2         0.00
T3       100.00
T4       100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET      6.28 KHz
IRFIN      0.87 Hz
IRRPW       115 usec
IRATN         79
DFILE   DK-4-137-f13-17 benzene
SF
LKSET     13.20 KHz
LKFIN      69.6 Hz
LKLEV          0
LGAIN          0
LKPHS          0
LKSIG          0
CSPED       0 Hz
FILDC
FILDF
CTEMP      21.5 c
SLVNT   C6D6
EXREF     128.06 ppm
```

スペクトルデータ

single_pulse

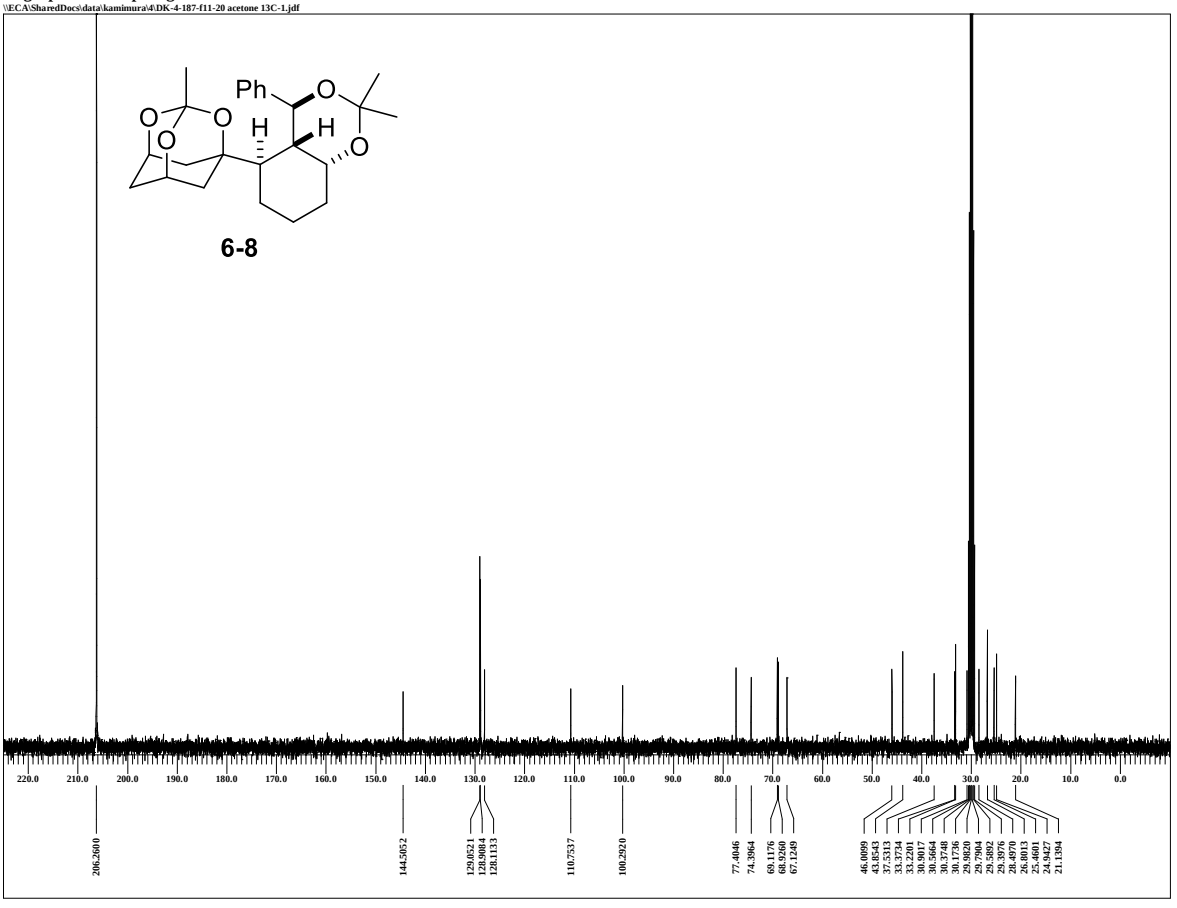
\\ECA\SharedDocs\data\kamimura\4\DK-4-187-f11-20 benzene-1.jdf




```
DFILE   DK-4-187-f11-20 benzene
COMNT   single_pulse
DATIM   25-07-2013 09:04:19
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.50 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       16384
SPO         16384
TIMES       8
DUMMY       1
FREQU       7422.80 Hz
FLT         30000 Hz
DELAY       16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS       8
ADBIT       16
RGAIN       32
BF          0.01 Hz
T1          0.00
T2          0.00
T3          100.00
T4          100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       115 usec
IRATN       79
DFILE   DK-4-187-f11-20 benzene
SF
LKSET       13.20 KHz
LKFIN       69.6 Hz
LKLEV       0
LGAIN       0
LKPHS       0
LKSIG       0
CSPED       0 Hz
FILDC
FILDF
CTEMP       23.1 c
SLVNT   C6D6
EXREF       7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-187-f11-20 acetone 13C-1.jdf

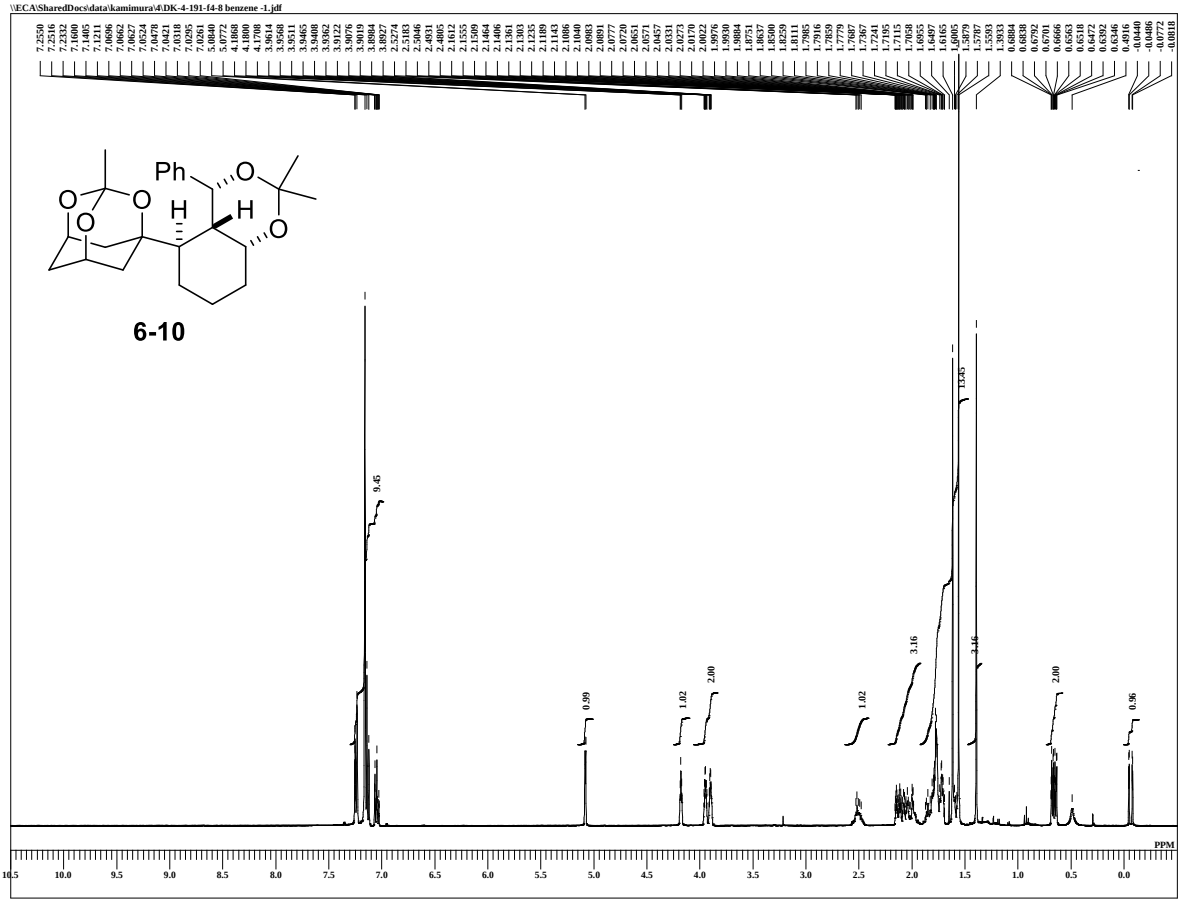



```
DFILE   DK-4-187-f11-20 acetone
COMNT   single pulse decoupled gat
DATIM   25-07-2013 12:07:22
MENUF
OBNUC   13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET       5.13 KHz
OBFIN       0.98 Hz
PW1         2.90 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       32768
SPO         32768
TIMES       63
DUMMY       4
FREQU       31250.00 Hz
FLT         125000 Hz
DELAY       20.50 usec
ACQTM       1.0486 sec
PD          2.0000 sec
SCANS       63
ADBIT       16
RGAIN       60
BF          1.00 Hz
T1          0.00
T2          0.00
T3          100.00
T4          100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       115 usec
IRATN       79
DFILE   DK-4-187-f11-20 acetone
SF
LKSET       12.90 KHz
LKFIN       59.1 Hz
LKLEV       0
LGAIN       0
LKPHS       0
LKSIG       0
CSPED       0 Hz
FILDC
FILDF
CTEMP       23.5 c
SLVNT   ACETN
EXREF       206.26 ppm
```

スペクトルデータ

single_pulse

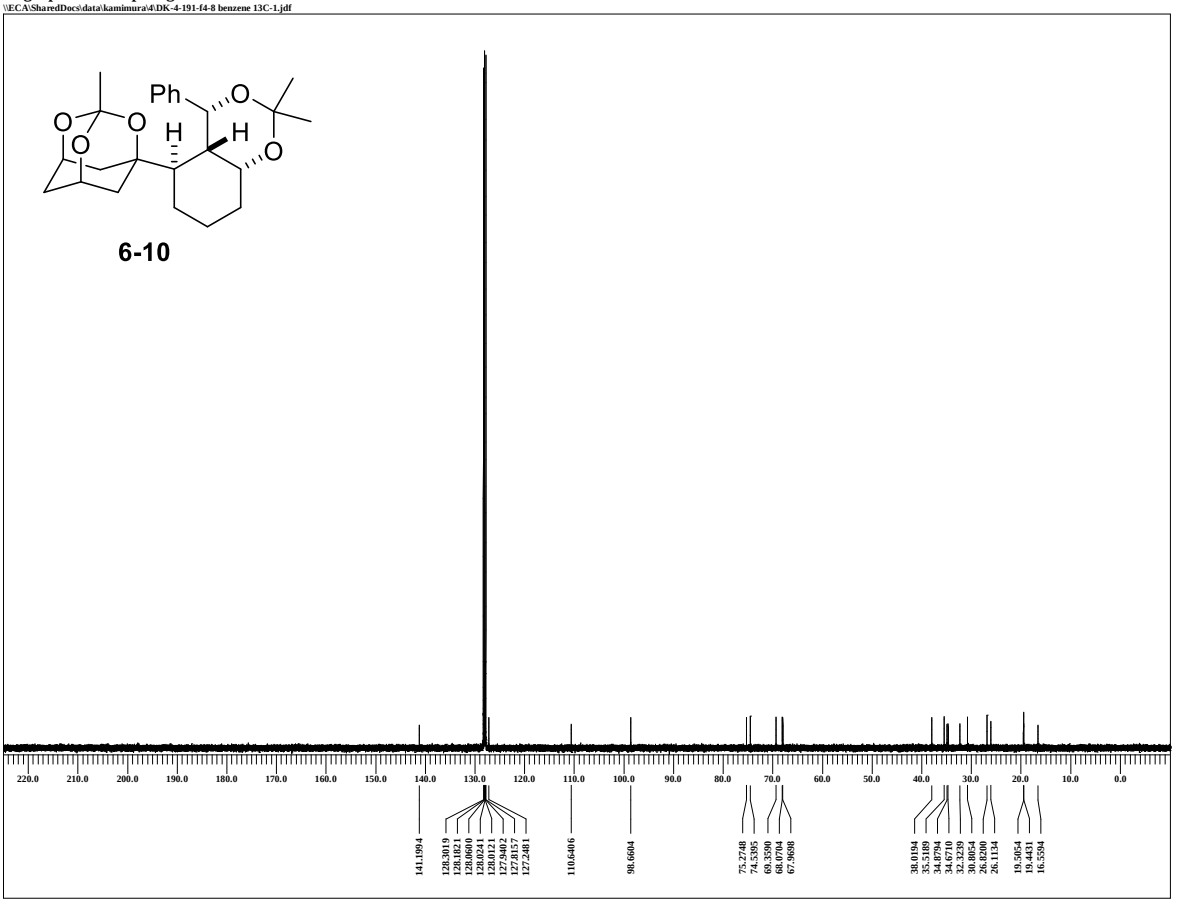
\\ECA\SharedDocs\data\kamimura\4\DK-4-191-f4-8 benzene -1.jdf



DFILE	DK-4-191-f4-8 benzene -1
COMNT	single_pulse
DATIM	26-07-2013 16:33:33
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	36
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-191-f4-8 benzene -1
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	24.1 c
SLVNT	C6D6
EXREF	7.16 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-191-f4-8 benzene 13C-1.jdf

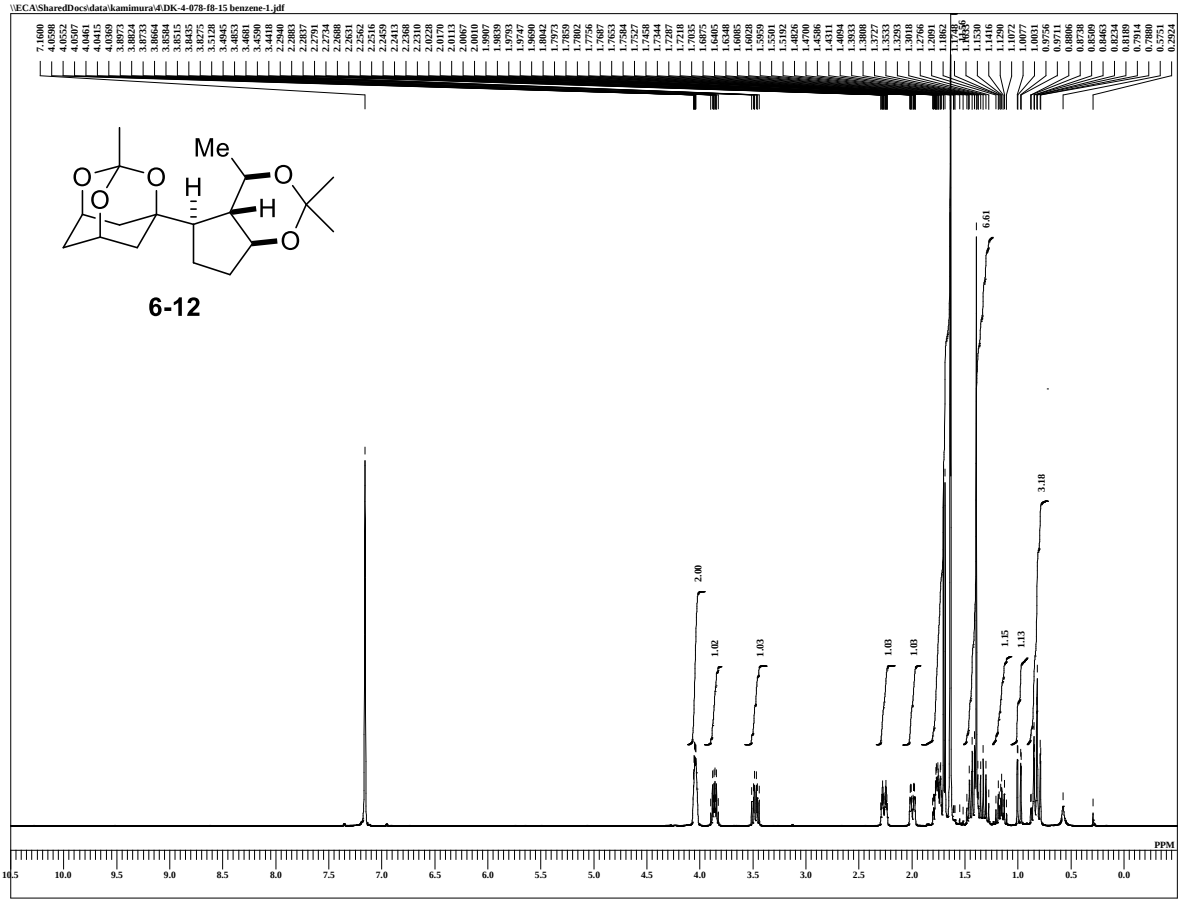


DFILE	DK-4-191-f4-8 benzene 13
COMNT	single pulse decoupled gat
DATIM	26-07-2013 16:43:32
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	2.90 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	131072
SPO	131072
TIMES	182
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	182
ADBIT	16
RGAIN	58
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-191-f4-8 benzene 13
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	24.3 c
SLVNT	C6D6
EXREF	128.06 ppm

スペクトルデータ

single_pulse

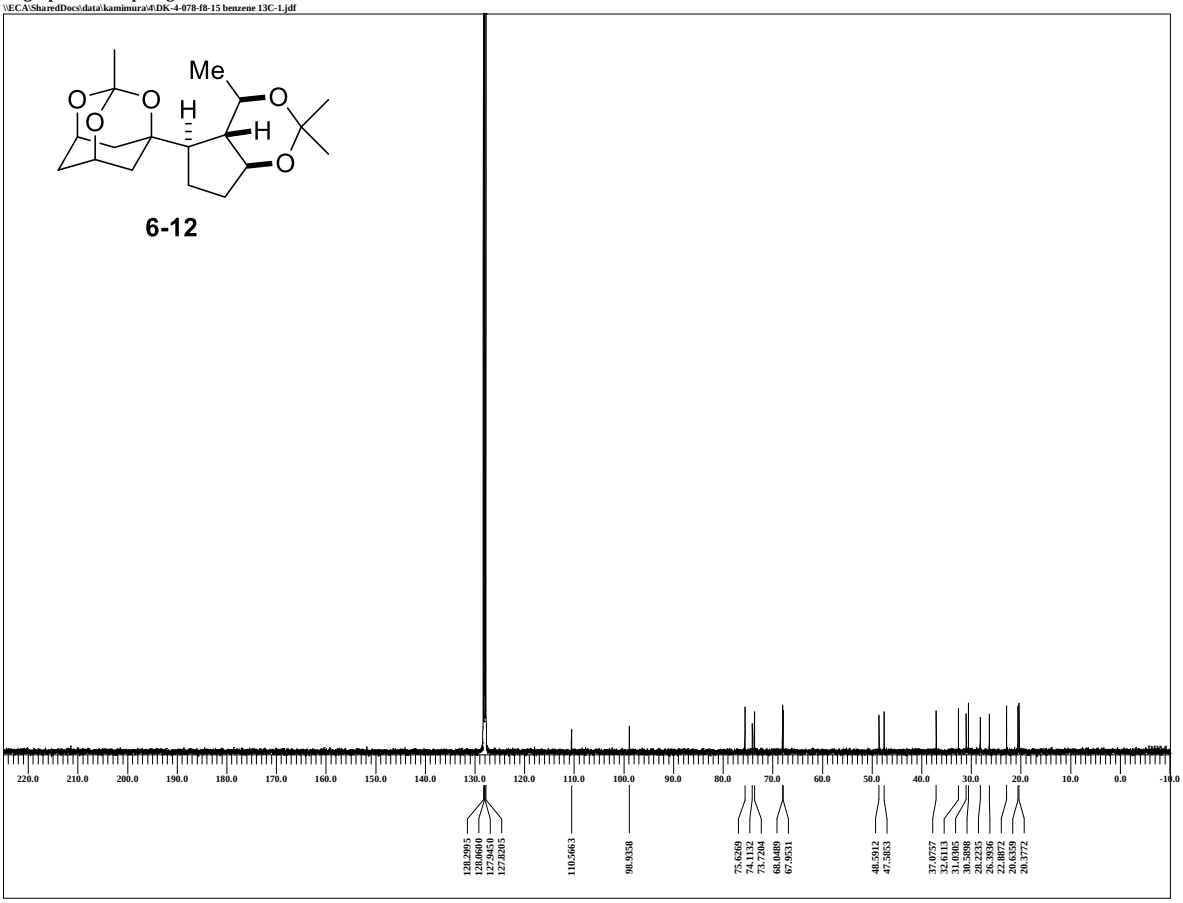
\\ECA\SharedDocs\data\kamimura\4\DK-4-078-f8-15 benzene-1.jdf



DFILE	DK-4-078-f8-15 benzene-1
COMNT	single_pulse
DATIM	08-06-2013 18:17:24
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.38 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	36
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-078-f8-15 benzene-1
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.8 c
SLVNT	C6D6
EXREF	7.16 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-078-f8-15 benzene 13C-1.jdf

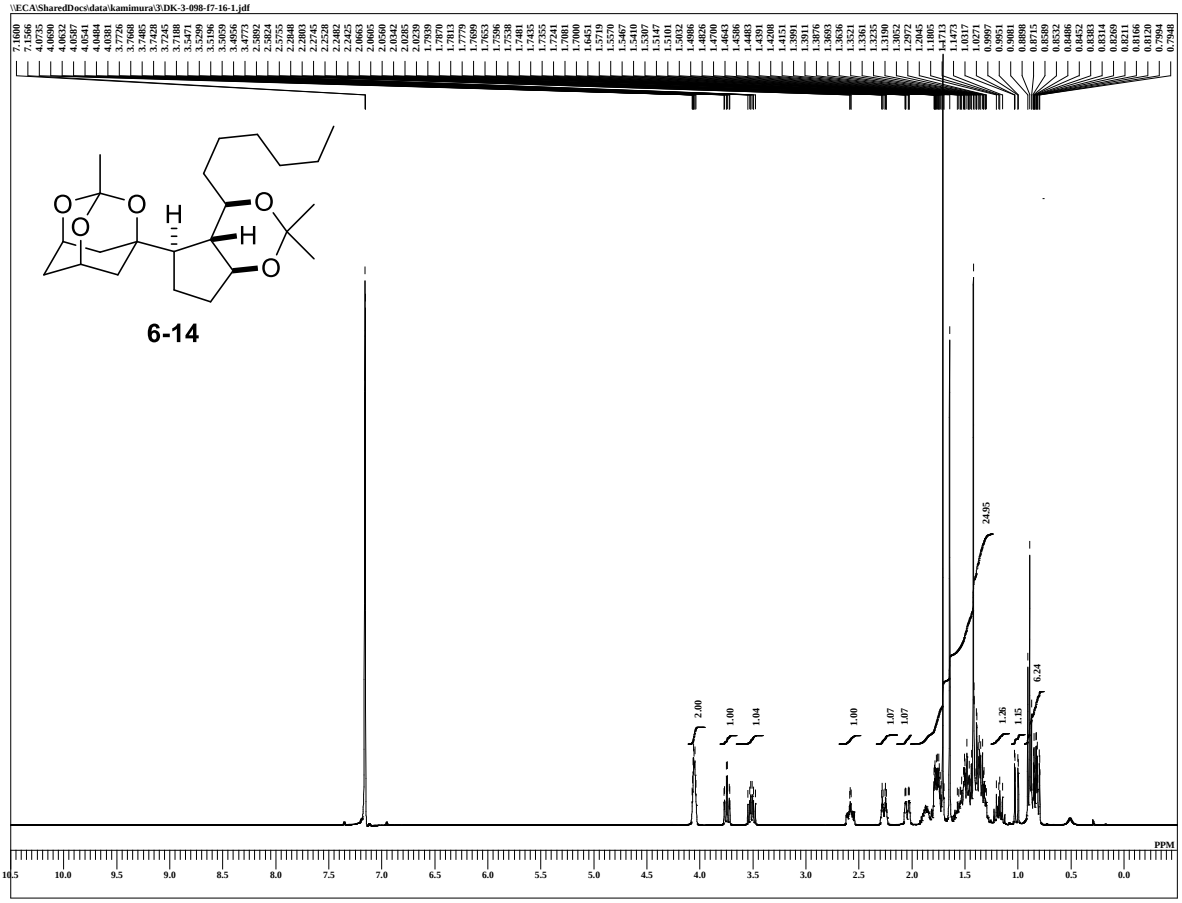


DFILE	DK-4-078-f8-15 benzene 1
COMNT	single pulse decoupled gat
DATIM	08-06-2013 18:24:36
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	123
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	123
ADBIT	16
RGAIN	58
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-078-f8-15 benzene 1
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.9 c
SLVNT	C6D6
EXREF	128.06 ppm

スペクトルデータ

single_pulse

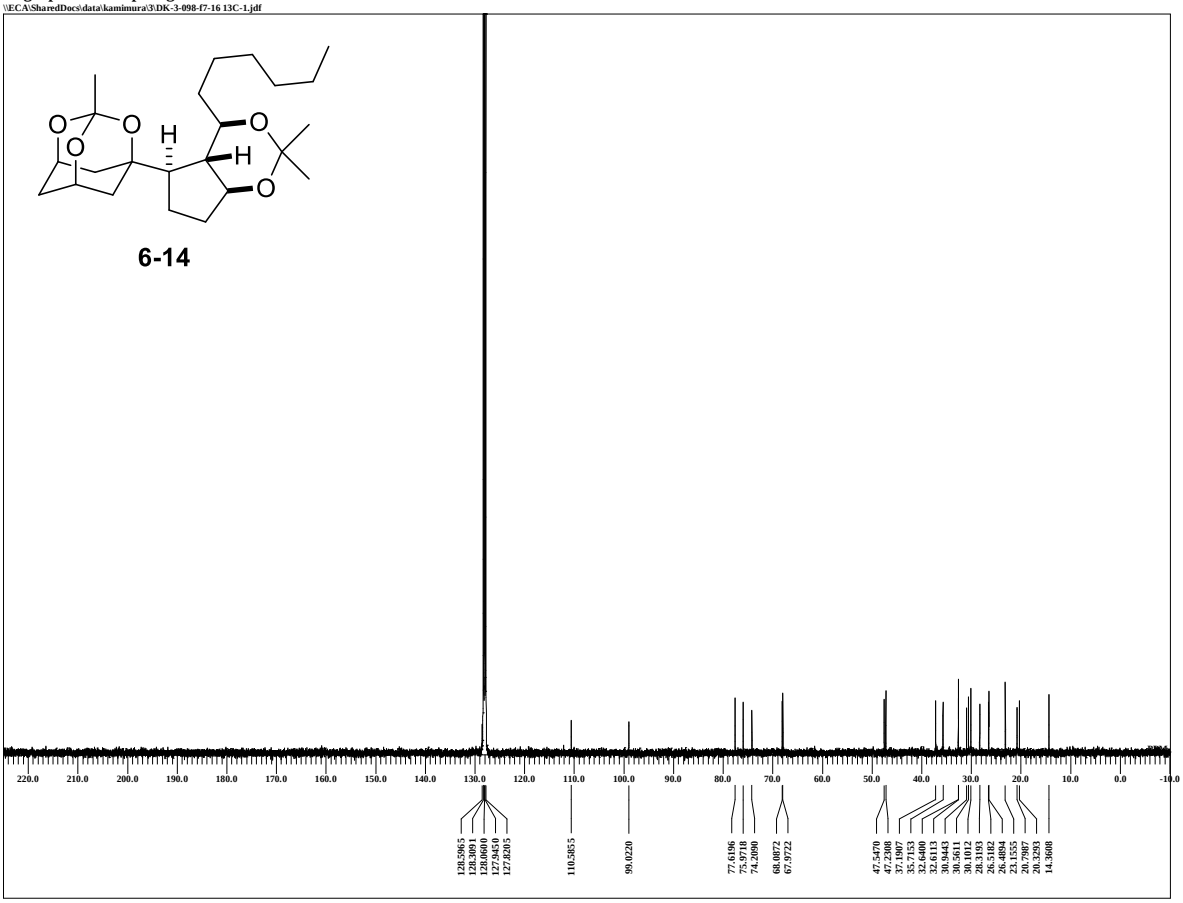
\\ECA\SharedDocs\data\kamimura\3\DK-3-098-f7-16-1.jdf




```
DFILE    DK-3-098-f7-16-1.jdf
COMNT    single_pulse
DATIM    01-02-2013 17:43:53
MENUF
OBNUC    1H
OFR        395.88 MHz
OBFRQ      395.88 MHz
OBSET        6.28 KHz
OBFIN        0.87 Hz
PW1          6.38 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        16384
SPO          16384
TIMES        8
DUMMY        1
FREQU        7422.80 Hz
FLT          30000 Hz
DELAY        16.68 usec
ACQTM        2.2073 sec
PD           2.0000 sec
SCANS        8
ADBIT        16
RGAIN        36
BF           0.01 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-3-098-f7-16-1.jdf
SF
LKSET        13.20 KHz
LKFIN        69.6 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        20.4 c
SLVNT    C6D6
EXREF        7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\3\DK-3-098-f7-16 13C-1.jdf

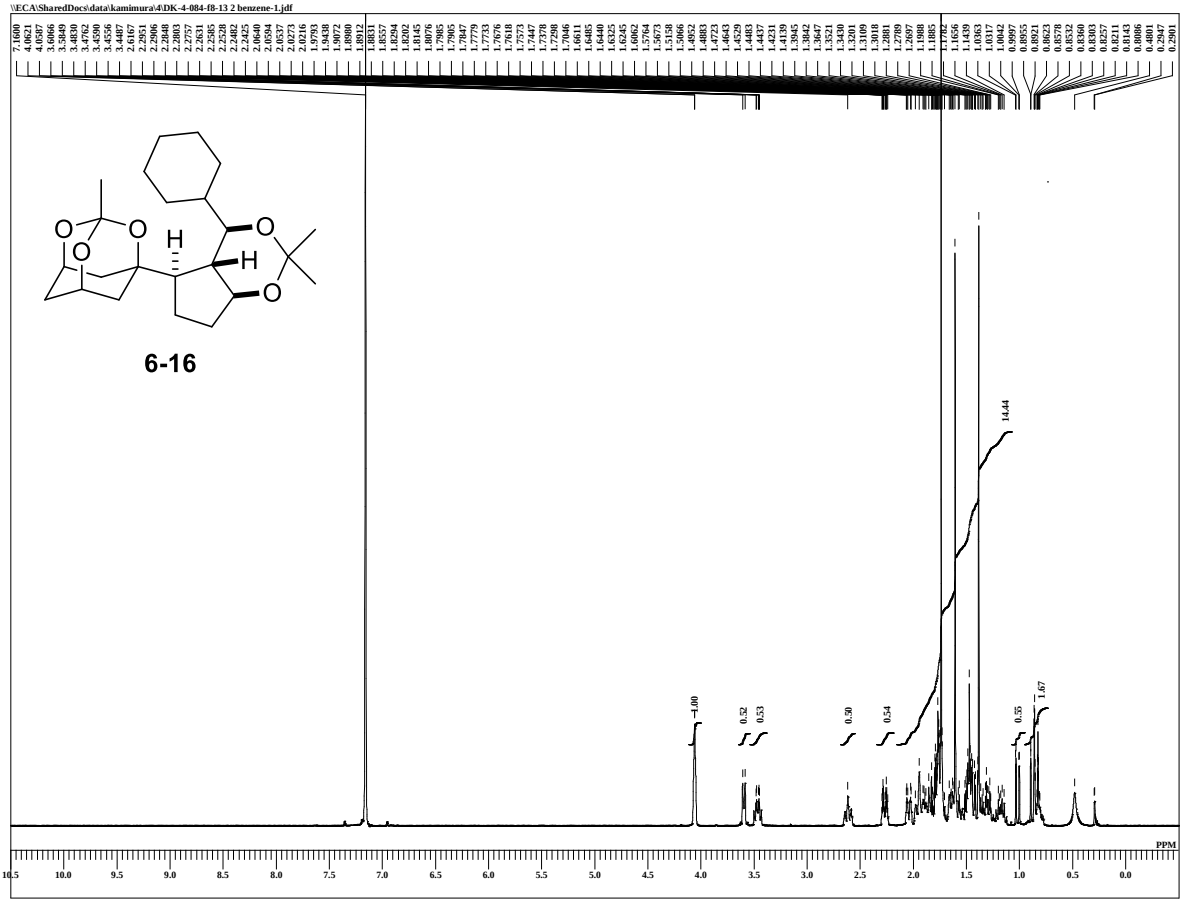



```
DFILE    DK-3-098-f7-16 13C-1.jdf
COMNT    single pulse decoupled gat
DATIM    01-02-2013 17:55:15
MENUF
OBNUC    13C
OFR        99.55 MHz
OBFRQ      99.55 MHz
OBSET        5.13 KHz
OBFIN        0.98 Hz
PW1          3.25 usec
DEADT        0.00 usec
PREDL        0.00000 msec
IWT          1.0000 sec
POINT        32768
SPO          32768
TIMES        203
DUMMY        4
FREQU        31250.00 Hz
FLT          125000 Hz
DELAY        20.50 usec
ACQTM        1.0486 sec
PD           2.0000 sec
SCANS        203
ADBIT        16
RGAIN        58
BF           1.00 Hz
T1           0.00
T2           0.00
T3           100.00
T4           100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR        395.88 MHz
IRSET        6.28 KHz
IRFIN        0.87 Hz
IRRPW        115 usec
IRATN        79
DFILE    DK-3-098-f7-16 13C-1.jdf
SF
LKSET        13.20 KHz
LKFIN        69.6 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED        0 Hz
FILDC
FILDF
CTEMP        20.9 c
SLVNT    C6D6
EXREF        128.06 ppm
```

スペクトルデータ

single_pulse

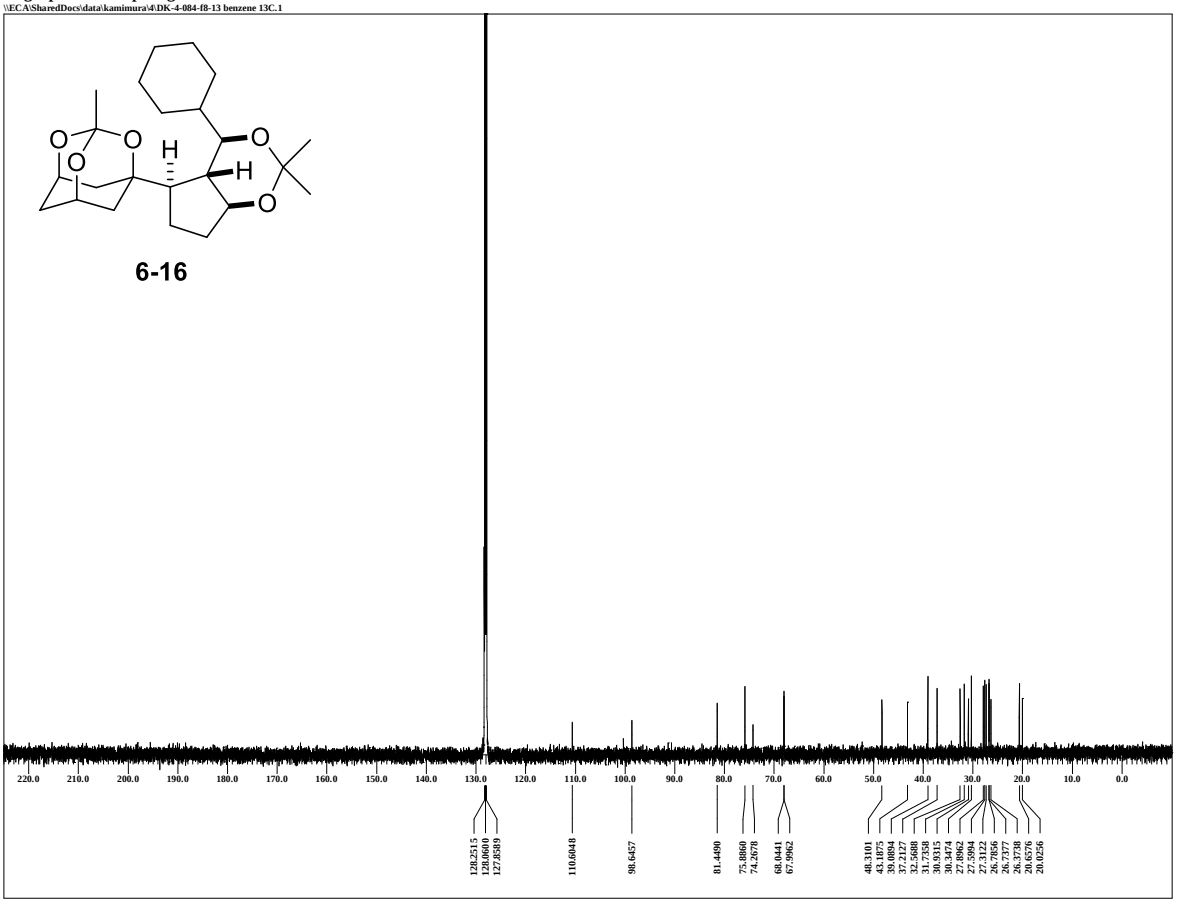
\\ECA\SharedDocs\data\kamimura\4\DK-4-084-f8-13 2 benzene-1.jdf




```
DFILE   DK-4-084-f8-13 2 benzene
COMNT   single_pulse
DATIM   28-07-2013 13:43:33
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         6.50 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       16384
SPO         16384
TIMES       8
DUMMY       1
FREQU     7422.80 Hz
FLT         30000 Hz
DELAY       16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS       8
ADBIT       16
RGAIN       38
BF          0.01 Hz
T1          0.00
T2          0.00
T3          100.00
T4          100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       147 usec
IRATN       79
DFILE   DK-4-084-f8-13 2 benzene
SF
LKSET     13.20 KHz
LKFIN       69.6 Hz
LKLEV       0
LGAIN       0
LKPHS       0
LKSIG       0
CSPED       0 Hz
FILDC
FILDF
CTEMP       22.7 c
SLVNT   C6D6
EXREF       7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-084-f8-13 benzene 13C.1

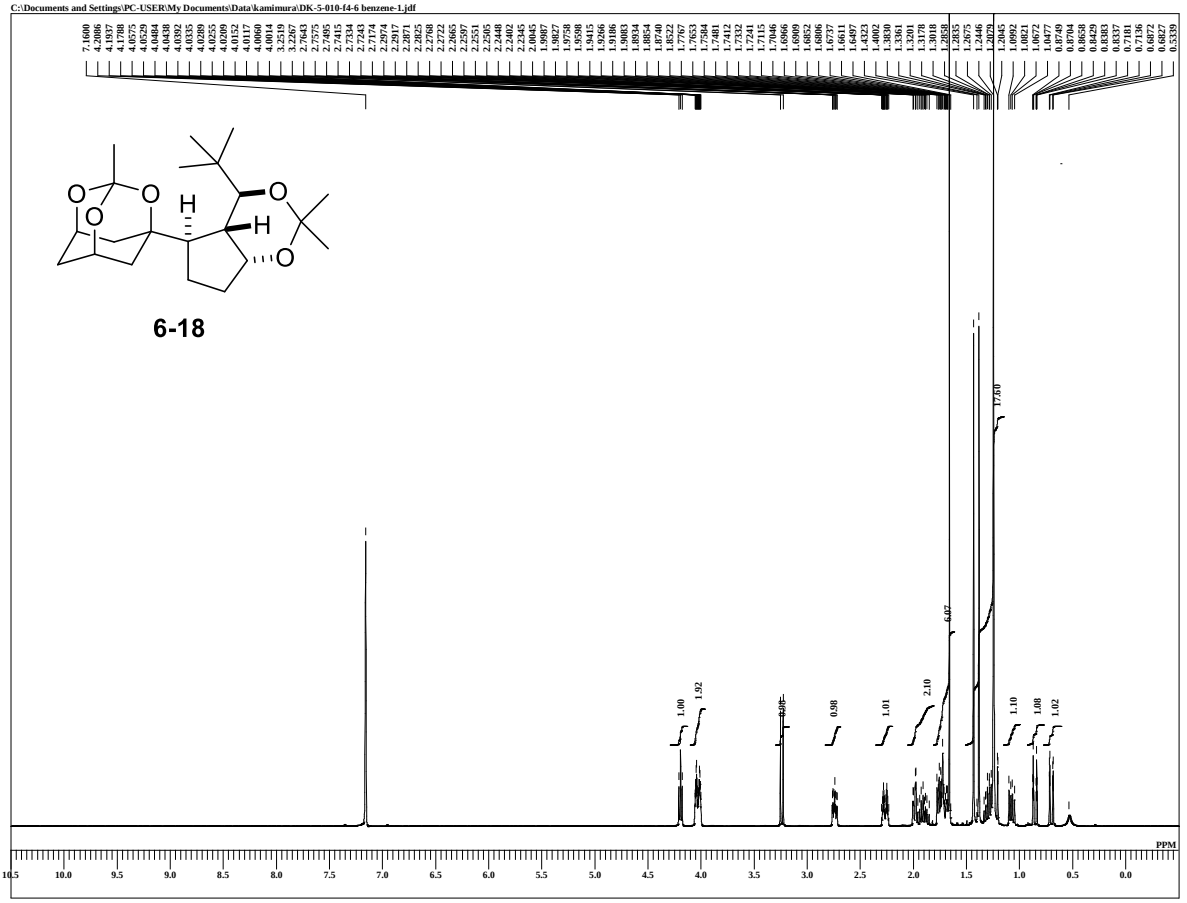



```
DFILE   DK-4-084-f8-13 benzene 1
COMNT   single pulse decoupled gat
DATIM   15-06-2013 16:38:52
MENUF
OBNUC   13C
OFR       124.51 MHz
OBFRQ     124.51 MHz
OBSET       3.45 KHz
OBFIN       6.00 Hz
PW1         3.70 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       32768
SPO         32768
TIMES       315
DUMMY       4
FREQU     39062.50 Hz
FLT       157000 Hz
DELAY       20.80 usec
ACQTM       0.8389 sec
PD          2.0000 sec
SCANS       315
ADBIT       16
RGAIN       50
BF          1.00 Hz
T1          0.00
T2          0.00
T3          100.00
T4          100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       495.13 MHz
IRSET       4.38 KHz
IRFIN       9.64 Hz
IRRPW       92 usec
IRATN       79
DFILE   DK-4-084-f8-13 benzene 1
SF
LKSET     748.40 KHz
LKFIN       90.6 Hz
LKLEV       0
LGAIN       0
LKPHS       0
LKSIG       0
CSPED       0 Hz
FILDC
FILDF
CTEMP       21.3 c
SLVNT   C6D6
EXREF       128.06 ppm
```

スペクトルデータ

single_pulse

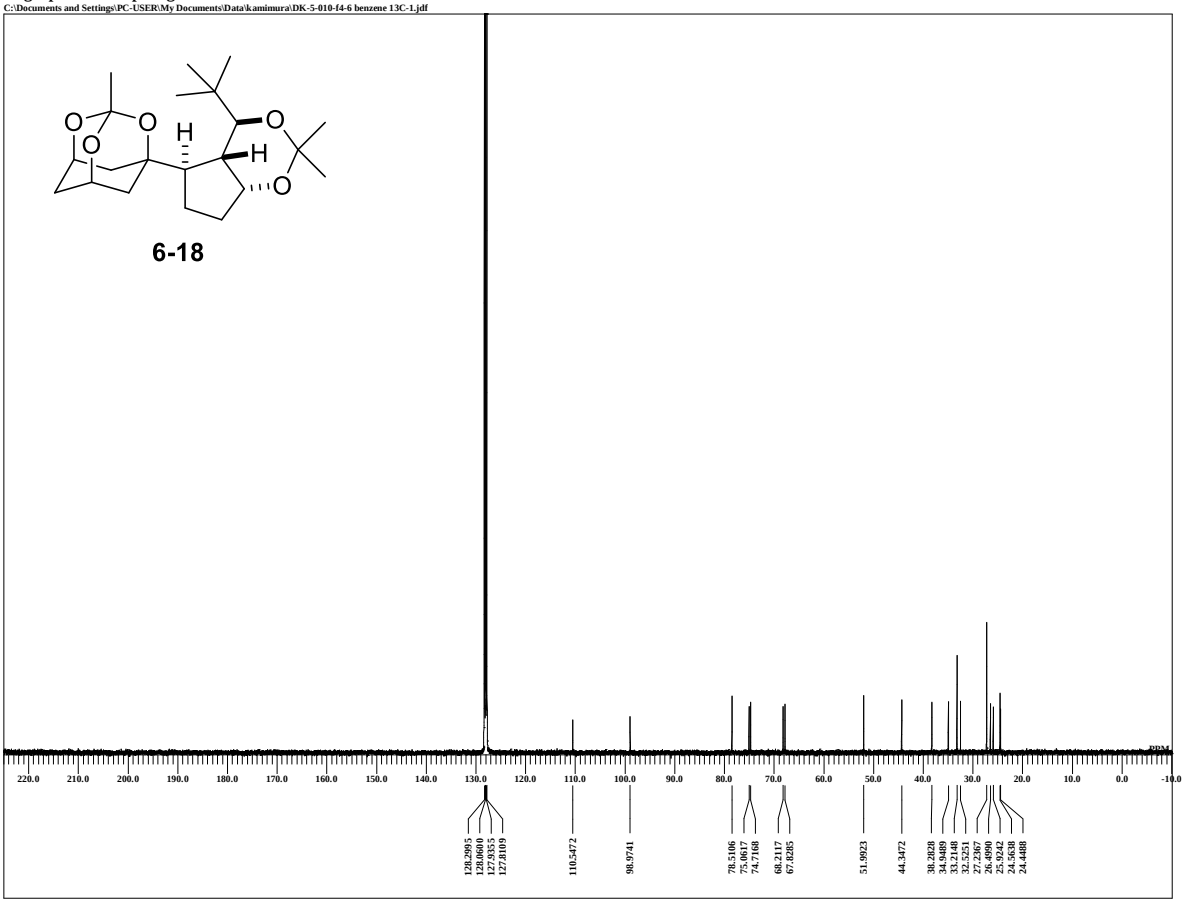
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-5-010-f4-6 benzene-1.jdf




```
DFILE   DK-5-010-f4-6 benzene-1.
COMNT   single_pulse
DATIM   04-08-2013 12:57:19
MENUF
OBNUC   1H
OFR     395.88 MHz
OBFRQ   395.88 MHz
OBSET   6.28 KHz
OBFIN   0.87 Hz
PW1     6.50 usec
DEADT   0.00 usec
PREDL   0.00000 msec
IWT     1.0000 sec
POINT   16384
SPO     16384
TIMES   8
DUMMY   1
FREQU   7422.80 Hz
FLT     30000 Hz
DELAY   16.68 usec
ACQTM   2.2073 sec
PD      2.0000 sec
SCANS   8
ADBIT   16
RGAIN   32
BF      0.01 Hz
T1      0.00
T2      0.00
T3      100.00
T4      100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR     395.88 MHz
IRSET   6.28 KHz
IRFIN   0.87 Hz
IRRPW   147 usec
IRATN   79
DFILE   DK-5-010-f4-6 benzene-1.
SF
LKSET   13.20 KHz
LKFIN   69.6 Hz
LKLEV   0
LGAIN   0
LKPHS   0
LKSIG   0
CSPED   0 Hz
FILDC
FILDF
CTEMP   22.7 c
SLVNT   C6D6
EXREF   7.16 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-5-010-f4-6 benzene 13C-1.jdf

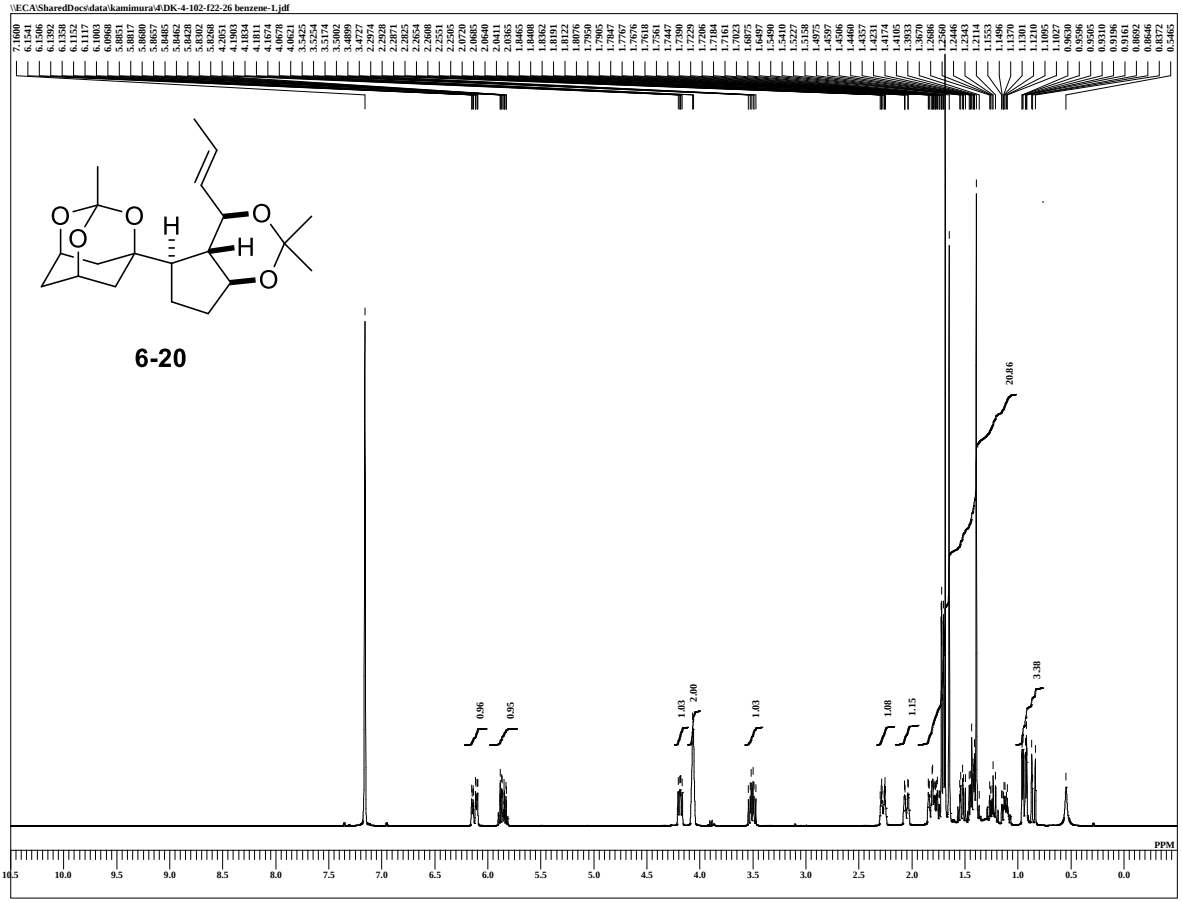



```
DFILE   DK-5-010-f4-6 benzene 13
COMNT   single pulse decoupled gat
DATIM   04-08-2013 13:13:12
MENUF
OBNUC   13C
OFR     99.55 MHz
OBFRQ   99.55 MHz
OBSET   5.13 KHz
OBFIN   0.98 Hz
PW1     2.90 usec
DEADT   0.00 usec
PREDL   0.00000 msec
IWT     1.0000 sec
POINT   32768
SPO     32768
TIMES   300
DUMMY   4
FREQU   31250.00 Hz
FLT     125000 Hz
DELAY   20.50 usec
ACQTM   1.0486 sec
PD      2.0000 sec
SCANS   300
ADBIT   16
RGAIN   60
BF      1.00 Hz
T1      0.00
T2      0.00
T3      100.00
T4      100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR     395.88 MHz
IRSET   6.28 KHz
IRFIN   0.87 Hz
IRRPW   115 usec
IRATN   79
DFILE   DK-5-010-f4-6 benzene 13
SF
LKSET   13.20 KHz
LKFIN   69.6 Hz
LKLEV   0
LGAIN   0
LKPHS   0
LKSIG   0
CSPED   0 Hz
FILDC
FILDF
CTEMP   22.9 c
SLVNT   C6D6
EXREF   128.06 ppm
```

スペクトルデータ

single_pulse

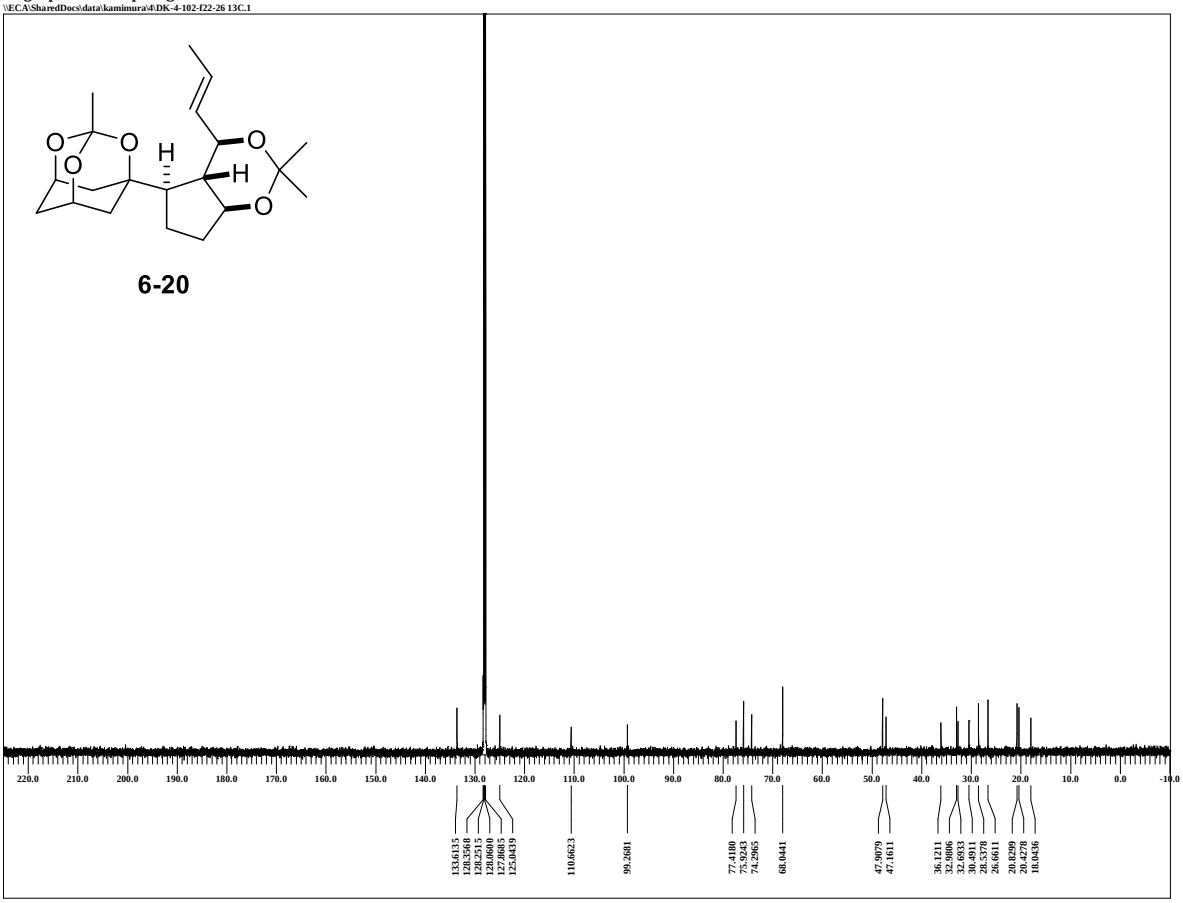
\\ECA\SharedDocs\data\kamimura\4\DK-4-102-f22-26 benzene-1.jdf




```
DFILE   DK-4-102-f22-26 benzene
COMNT   single_pulse
DATIM   14-06-2013 17:50:56
MENUF
OBNUC   1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET       6.28 KHz
OBFIN       0.87 Hz
PW1         3.00 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       16384
SPO         16384
TIMES       16
DUMMY       1
FREQU     7422.80 Hz
FLT         30000 Hz
DELAY      16.68 usec
ACQTM       2.2073 sec
PD          2.0000 sec
SCANS       16
ADBIT       16
RGAIN       30
BF          0.01 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR       395.88 MHz
IRSET       6.28 KHz
IRFIN       0.87 Hz
IRRPW       115 usec
IRATN       79
DFILE   DK-4-102-f22-26 benzene
SF
LKSET      13.20 KHz
LKFIN      69.6 Hz
LKLEV      0
LGAIN      0
LKPHS      0
LKSIG      0
CSPED      0 Hz
FILDC
FILDF
CTEMP      21.6 c
SLVNT   C6D6
EXREF      7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-102-f22-26 13C.1

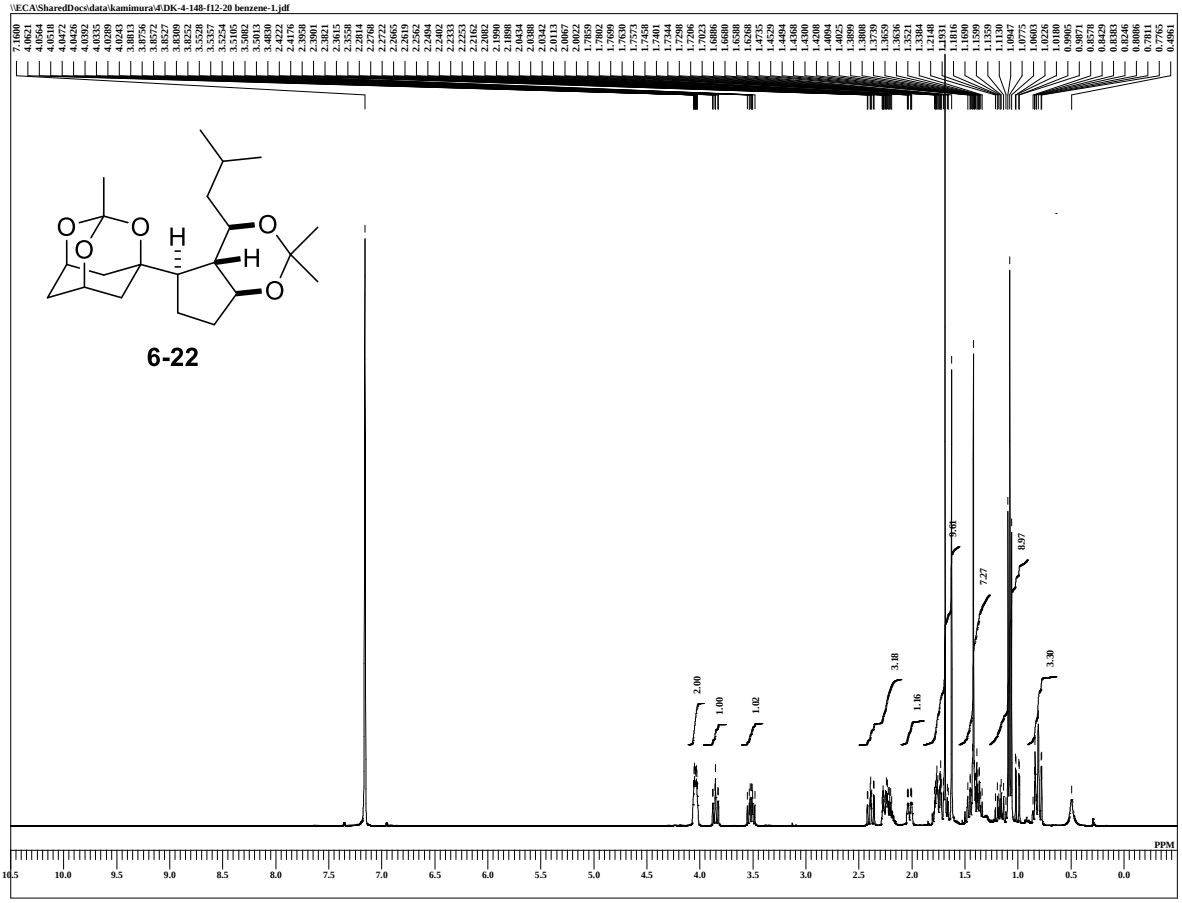



```
DFILE   DK-4-102-f22-26 13C.1
COMNT   single pulse decoupled gat
DATIM   14-06-2013 17:25:27
MENUF
OBNUC   13C
OFR       124.51 MHz
OBFRQ     124.51 MHz
OBSET       3.45 KHz
OBFIN       6.00 Hz
PW1         3.70 usec
DEADT       0.00 usec
PREDL       0.00000 msec
IWT         1.0000 sec
POINT       32768
SPO         32768
TIMES       130
DUMMY       4
FREQU     39062.50 Hz
FLT         157000 Hz
DELAY      20.80 usec
ACQTM       0.8389 sec
PD          2.0000 sec
SCANS       130
ADBIT       16
RGAIN       48
BF          1.00 Hz
T1          0.00
T2          0.00
T3        100.00
T4        100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR       495.13 MHz
IRSET       4.38 KHz
IRFIN       9.64 Hz
IRRPW       92 usec
IRATN       79
DFILE   DK-4-102-f22-26 13C.1
SF
LKSET      748.40 KHz
LKFIN      90.6 Hz
LKLEV      0
LGAIN      0
LKPHS      0
LKSIG      0
CSPED      0 Hz
FILDC
FILDF
CTEMP      21.6 c
SLVNT   C6D6
EXREF      128.06 ppm
```

スペクトルデータ

single_pulse

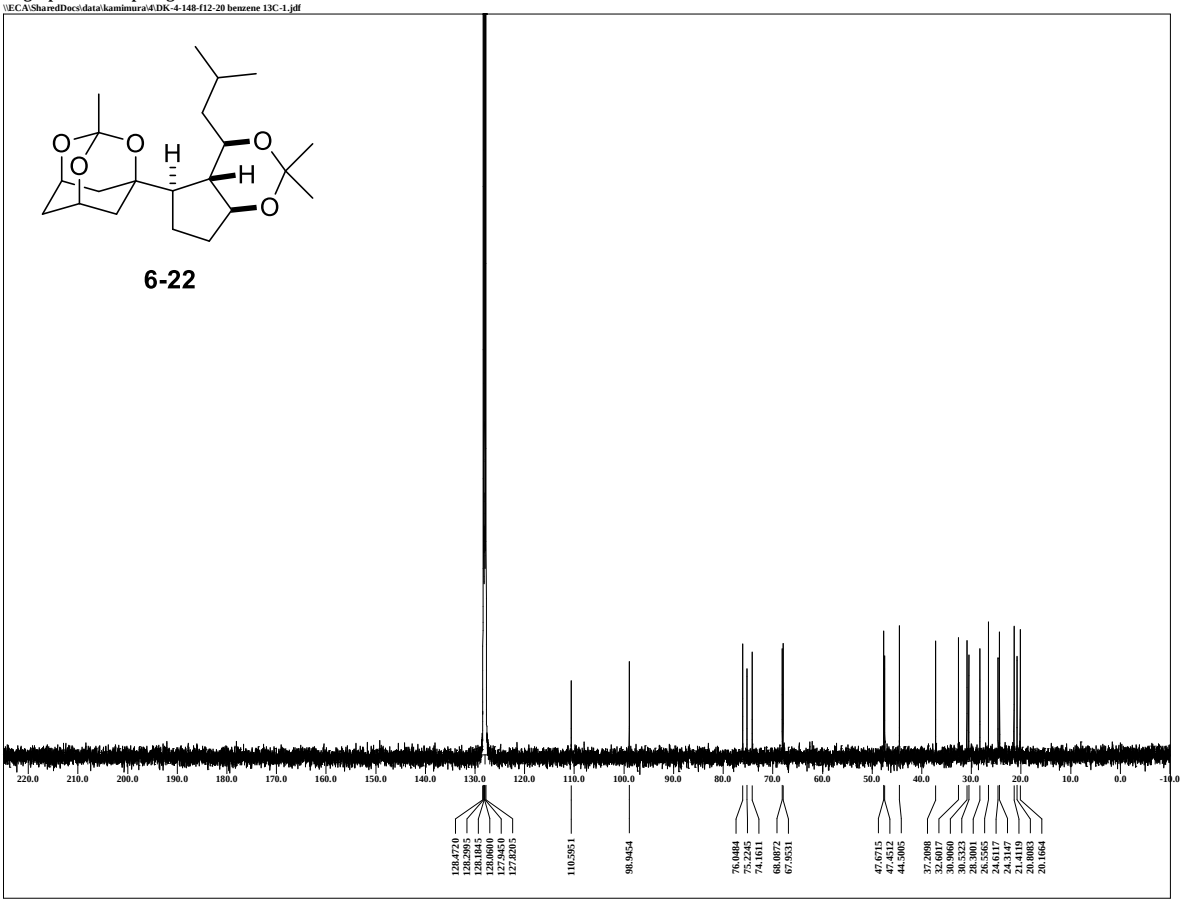
\\ECA\SharedDocs\data\kamimura\4\DK-4-148-f12-20 benzene-1.jdf



DFILE	DK-4-148-f12-20 benzene
COMNT	single_pulse
DATIM	07-07-2013 12:09:38
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	32
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	32
ADBIT	16
RGAIN	38
BF	0.01 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	147 usec
IRATN	79
DFILE	DK-4-148-f12-20 benzene
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.7 c
SLVNT	C6D6
EXREF	7.16 ppm

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-148-f12-20 benzene 13C-1.jdf

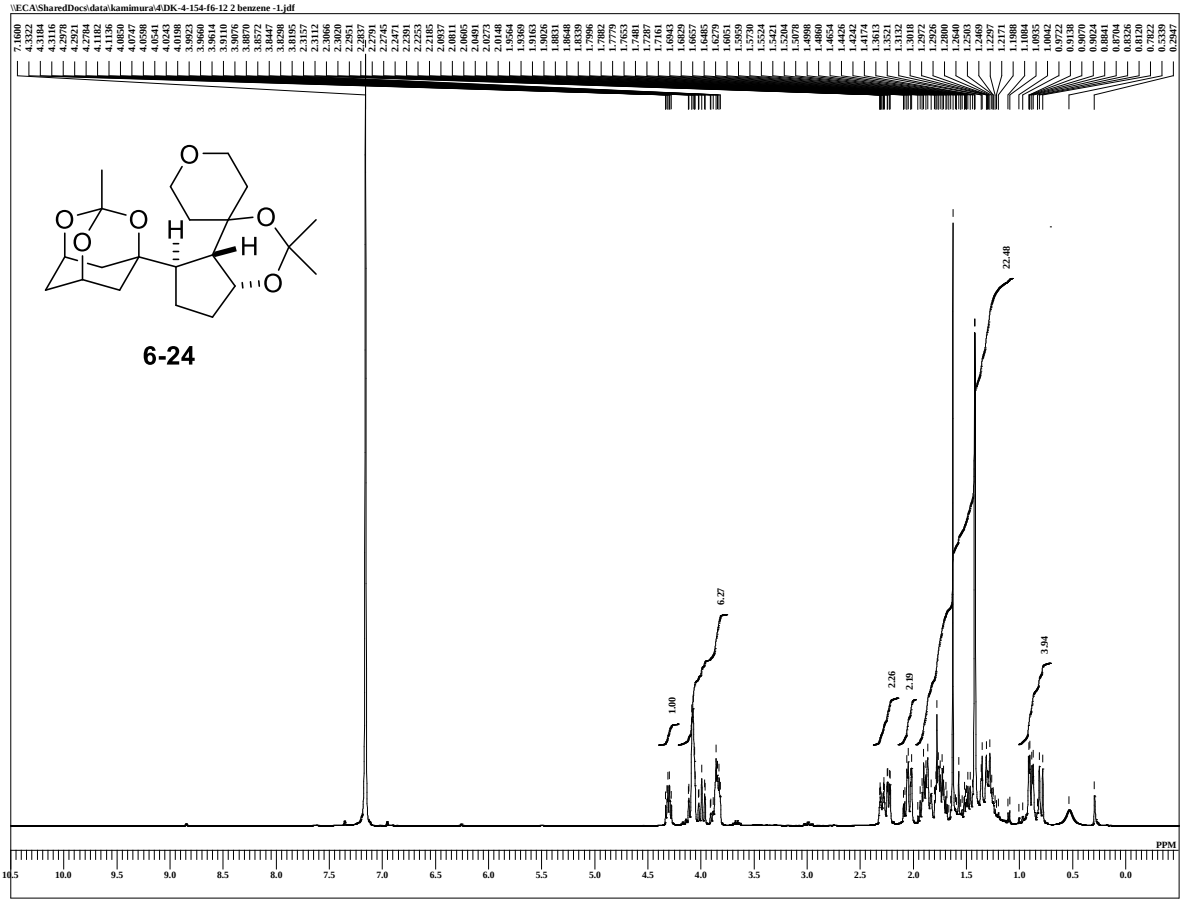


DFILE	DK-4-148-f12-20 benzene
COMNT	single pulse decoupled gat
DATIM	07-07-2013 12:30:36
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	2.90 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	400
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	400
ADBIT	16
RGAIN	58
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	DK-4-148-f12-20 benzene
SF	
LKSET	13.20 KHz
LKFIN	69.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	21.9 c
SLVNT	C6D6
EXREF	128.06 ppm

スペクトルデータ

single_pulse

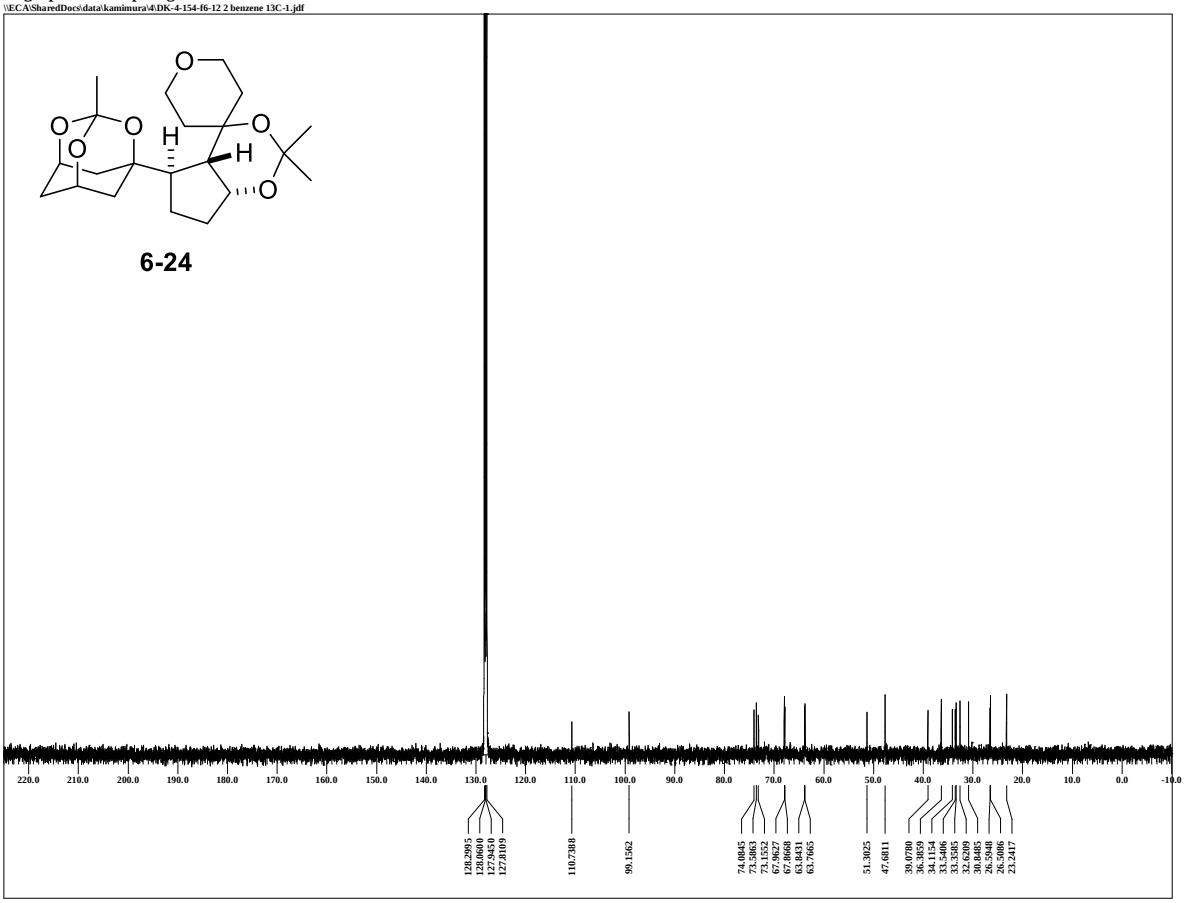
\\ECA\SharedDocs\data\kamimura\4\DK-4-154-f6-12 2 benzene -1.jdf




```
DFILE    DK-4-154-f6-12 2 benzene
COMNT    single_pulse
DATIM    28-07-2013 11:43:08
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.50 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    32
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    32
ADBIT    16
RGAIN    38
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    147 usec
IRATN    79
DFILE    DK-4-154-f6-12 2 benzene
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.6 c
SLVNT    C6D6
EXREF    7.16 ppm
```

single pulse decoupled gated NOE

\\ECA\SharedDocs\data\kamimura\4\DK-4-154-f6-12 2 benzene 13C-1.jdf

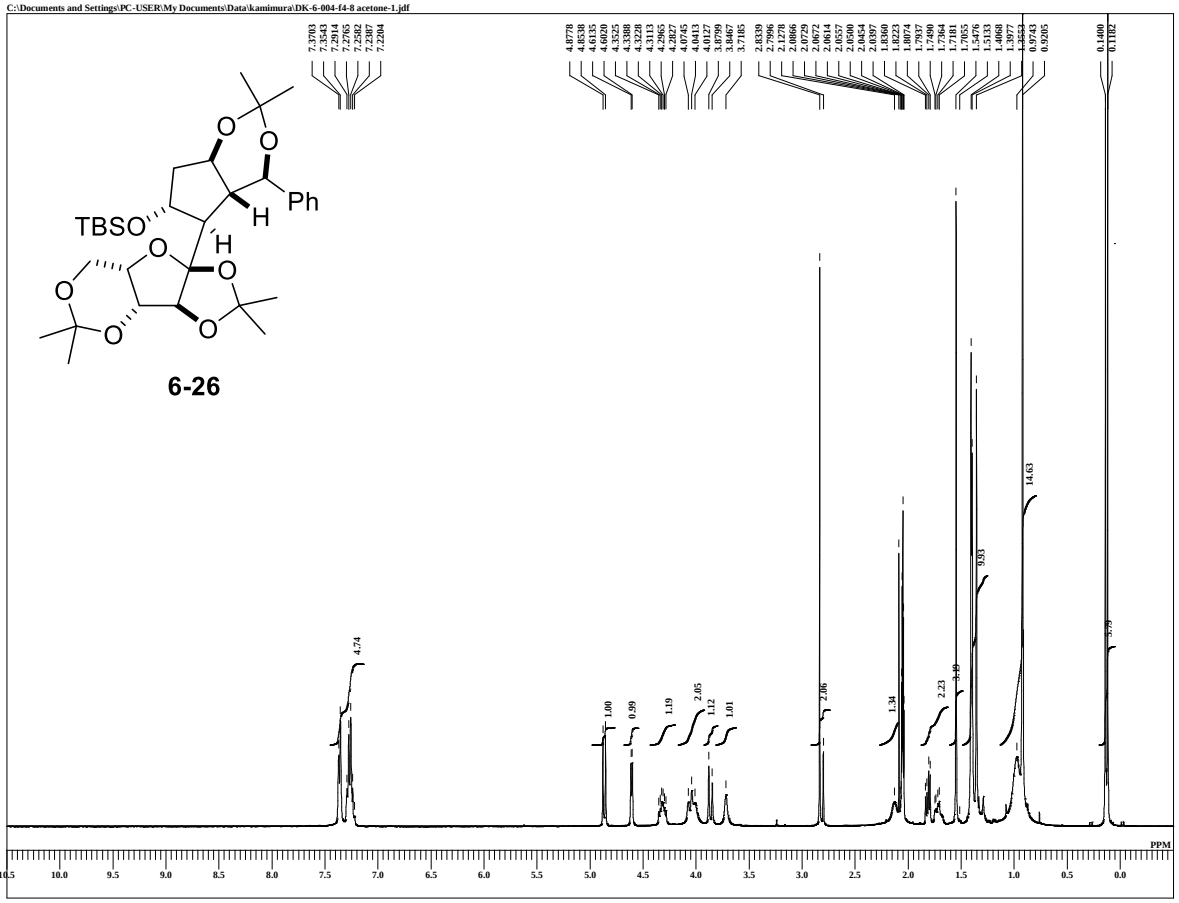



```
DFILE    DK-4-154-f6-12 2 benzene
COMNT    single pulse decoupled gat
DATIM    28-07-2013 11:53:57
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      2.90 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    200
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    200
ADBIT    16
RGAIN    58
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-4-154-f6-12 2 benzene
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.8 c
SLVNT    C6D6
EXREF    128.06 ppm
```

スペクトルデータ

single_pulse

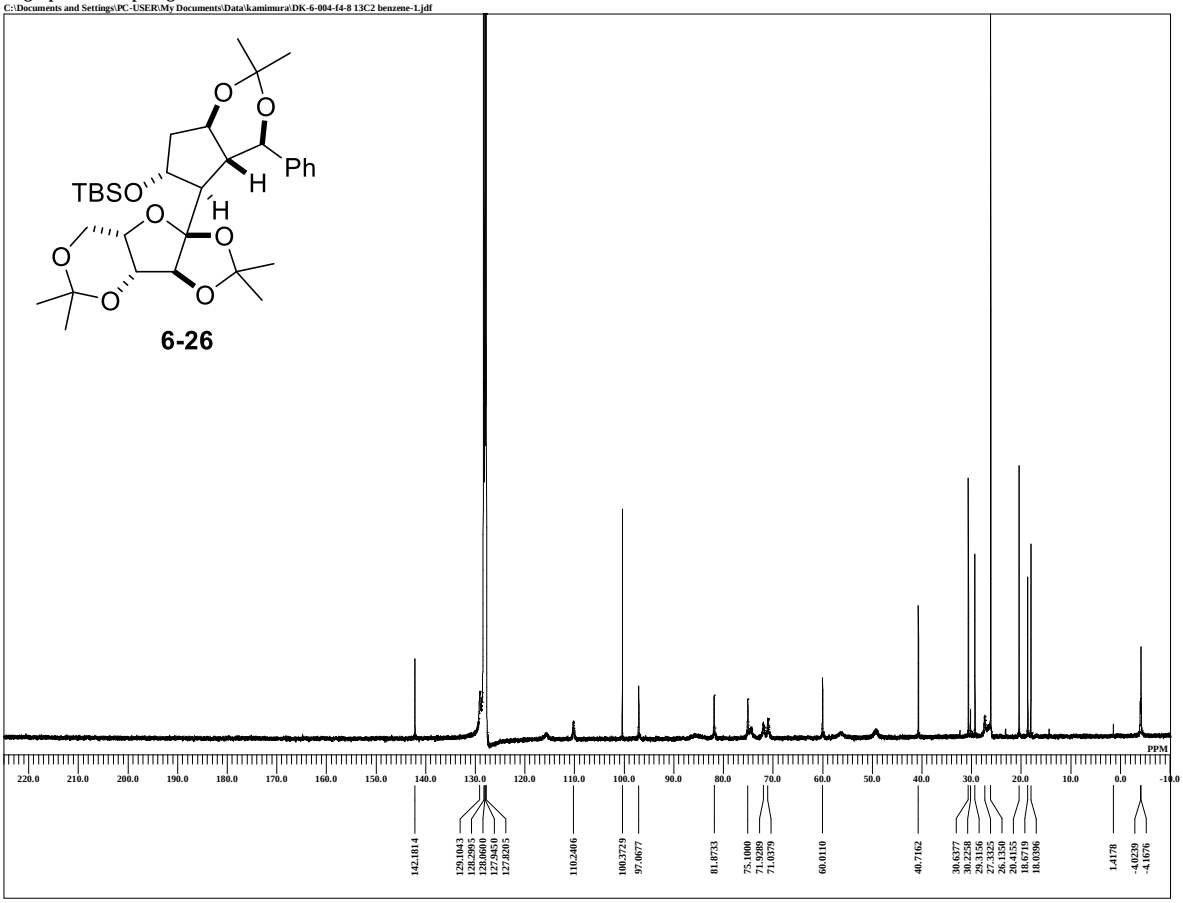
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-004-f4-8 acetone-1.jdf



DFILE DK-6-004-f4-8 acetone-1.j
COMNT single_pulse
DATIM 02-12-2013 08:13:58
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DK-6-004-f4-8 acetone-1.j
SF
LKSET 12.90 KHz
LKFIN 59.1 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT ACETN
EXREF 2.05 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-004-f4-8 13C2 benzene-1.jdf

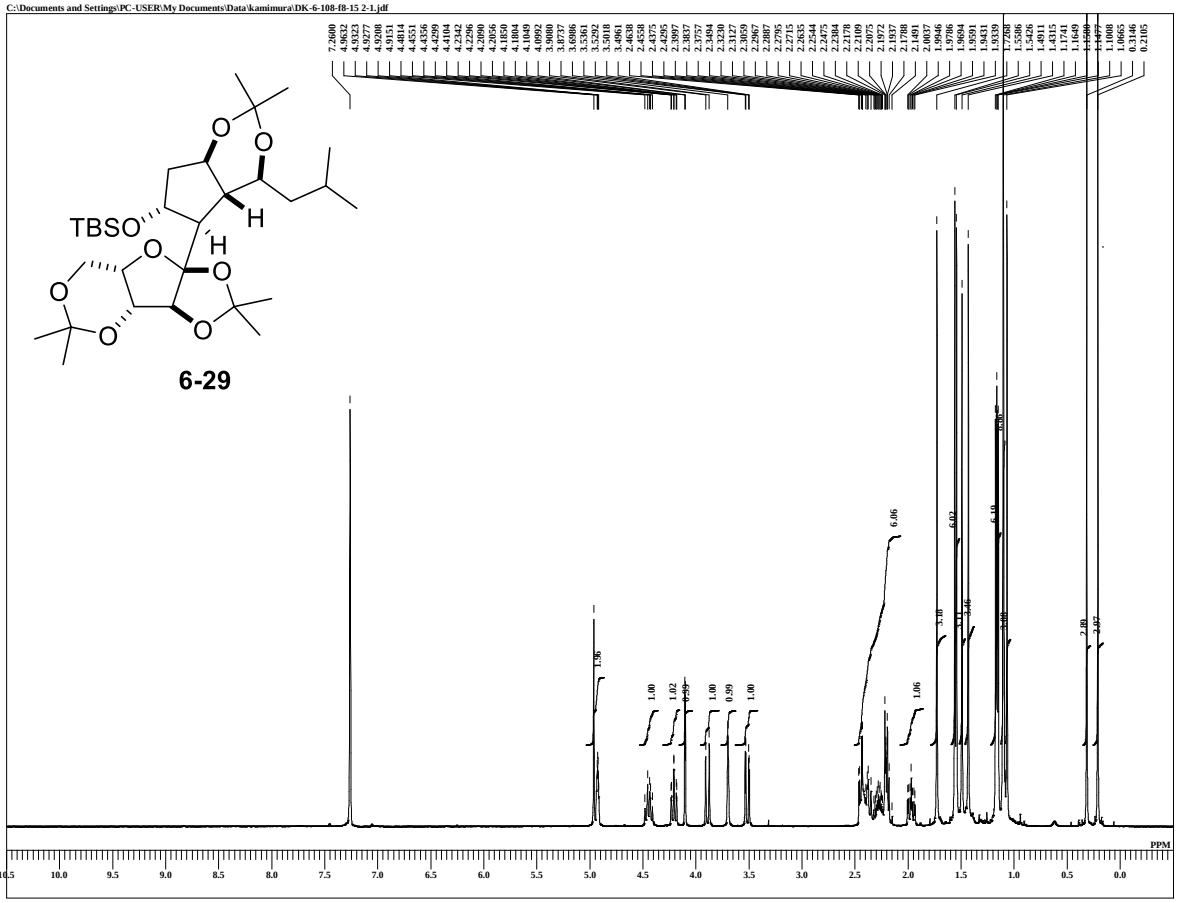


DFILE DK-6-004-f4-8 13C2 benz
COMNT single pulse decoupled gat
DATIM 02-03-2014 07:50:07
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 15000
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 15000
ADBIT 16
RGAIN 58
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DK-6-004-f4-8 13C2 benz
SF
LKSET 13.20 KHz
LKFIN 69.6 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.7 c
SLVNT C6D6
EXREF 128.06 ppm

スペクトルデータ

single_pulse

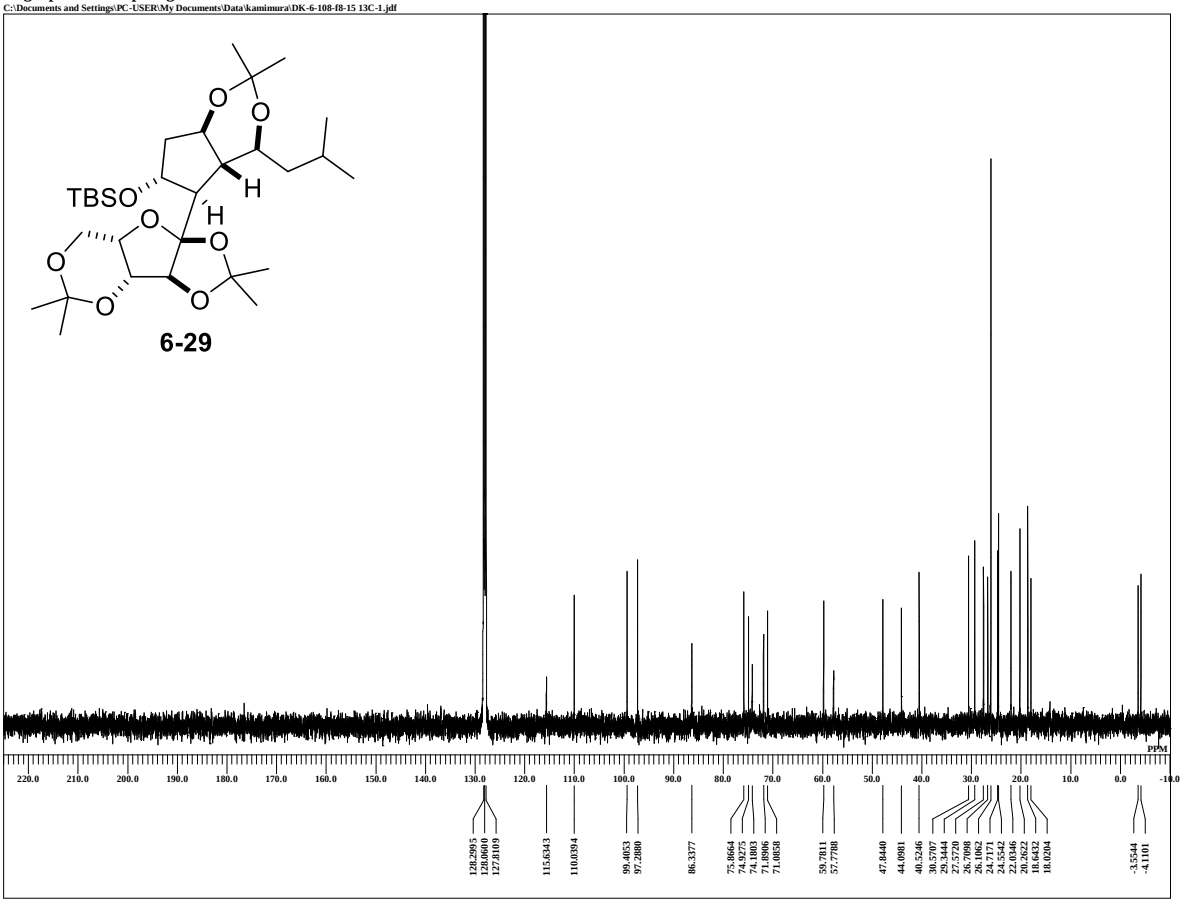
C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-108-f8-15 2-1.jdf




```
DFILE    DK-6-108-f8-15 2-1.jdf
COMNT    single_pulse
DATIM    18-02-2014 16:21:41
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    30
BF       0.10 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-6-108-f8-15 2-1.jdf
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.9 c
SLVNT    C6D6
EXREF    7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\My Documents\Data\kamimura\DK-6-108-f8-15 13C-1.jdf




```
DFILE    DK-6-108-f8-15 13C-1.jdf
COMNT    single pulse decoupled gat
DATIM    18-02-2014 11:20:51
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.03 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    151
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    151
ADBIT    16
RGAIN    58
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    DK-6-108-f8-15 13C-1.jdf
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    21.0 c
SLVNT    C6D6
EXREF    128.06 ppm
```