Preparation of Pure Alkali Nitrites.

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When pure sodium or potassium nitrite is wanted, it is customary to prepare silver nitrite from crude alkali nitrite, and convert this again into alkali nitrite by means of sodium or potassium chloride. The crude nitrite must be nearly free from sulphate and, either before or after adding the silver nitrate to it, nitric acid must be added till all hydroxide and carbonate are neutralised. The silver nitrite is got in the most convenient form for washing by precipitating it from concentrated solutions. Silver chloride is very sensibly soluble in a concentrated solution of alkali nitrite, and when the solution is no longer clouded by the addition of more alkali chloride, it already contains this salt in excess. Therefore, somewhat large dilution is necessary and this entails, of course, much evaporation afterwards.

The silver nitrite process is evidently not a very satisfactory one, and when sodium nitrite is wanted pure, it is better to recrystallise, three times over, the commercial 96 per cent. sodium nitrite, well draining each time on the suction funnel. A concentrated solution of the crude salt should be left to clear from lead-turbidity for two days or be filtered cold through a fine filter. The lead carbonate is more soluble in the hot nitrite solution than in the cold. After separating the lead,

the solution should be fully neutralised with nitric acid, before evaporation for crystallising. Potassium nitrite is too soluble and deliquescent to be conveniently purified in a similar way.

A most satisfactory and simple process for preparing either sodium or potassium nitrite, when the pure hydroxide or carbonate is at command, is to saturate this with red fumes under appropriate conditions. That nitrites can be thus obtained is known to every chemist, was known to Gay-Lussac in 1816, and was described by Fritzsche in 1840, but it has hitherto been stated and believed that much nitrate is then unavoidably formed along with the nitrite. That is a mistake, and therefore this note is published. If obvious precautions of the simplest kind are taken, so little nitrate, if any, is formed, as to be hardly detectable with certainty in presence of so much nitrite. Consequently, if the quantity of pure alkali taken is known, a solution of given strength in nitrite is perhaps better prepared in this way than in any other.

Avoiding so far as practicable, the use of cork and caoutchouc, nitrous gases, from nitric acid and starch or arsenious oxide, are passed into the concentrated solution of the hydroxide or carbonate until the alkali is quite neutralised. Sodium carbonate alone is somewhat inconvenient, because of its sparing solubility, but this may be circumvented by adding it, finely divided and in sufficient quantity, to its own saturated solution just before passing the gases, and by often shaking the vessel during their absorption. To prevent free access of air, the nitrite is prepared in a flask with its mouth kept loosely closed, while the gases are passing. Cooling the flask is not necessary. The strength of the nitric acid and the temperature of the generation of the nitrous gases must be so regulated that just

a little nitric oxide is in excess of the nitric peroxide, and therefore is passing unabsorbed, as a guarantee that the latter does not act upon the solution in absence of its equivalent of the former and thus produce some nitrate. To free the gases from volatilising nitric acid they may be passed through a bottle or tube, either empty or packed loosely with cotton. The finished solution must be almost neutral; if acid, it is to be boiled till neutral, before exposing it to the air. A concentrated solution of alkali nitrite dissolves a little nitrous acid without decomposing it, as water alone would. To get the salt in the solid state or to crystallise out the sodium nitrite, where it is necessary to be sure of absence of all nitrate in it, the solution may be freely evaporated, even at a boiling heat, without decomposing or oxidising it.

The alkali nitrites have been very imperfectly described, and need examination. In the mean time, some points in their description are here given. Sodium nitrite and potassium nitrite are distinctly though faintly yellow and give markedly yellow solutions in a little water. They are very slightly alkaline to litmus. At 15° sodium nitrite, 5 parts, requires water, 6 parts, to dissolve it. Potassium nitrite is soluble in about one-third of its weight of water. Sodium nitrite melts at 271° (mercury thread immersed). Sodium nitrite is moderately deliquescent, remaining dry in winter-cold weather. Potassium nitrite is exceedingly deliquescent. This nitrite is got in very small, thick, prismatic crystals, but sodium nitrite crystallises in very thin, flattened prisms, often very large. Sodium nitrite is well known to be anhydrous; not so, potassium nitrite, crystals of which are reputed to contain half a molecule of water. I have examined small, but distinct and separate, crystals, taken from

the upper part of some kilos. of the commercial salt, which had become well drained by long standing. They were removed in very dry weather and weighed, and then found to lose hardly one per cent. on fusion. The anhydrous character of the salt was further ascertained by heating a cake of minute crystals, prepared by myself, which had been pressed, under cover, between porous tiles, in cold dry air. The loss of weight on heating, much above 100°, was a little over one per cent., and the percentage of potassium was 45.30, instead of 45.88, required for the anhydrous salt.

Somewhat remarkably, the point as to hydration of potassium nitrite was examined independently in the same year, 1863, by Lang and by Hampe, with identical results, indicating the composition expressed by $(KNO_2)_2,OH_2$. But then both chemists made the determination upon a magma of indistinct crystals, dried up in the vacuum-desiccator.