PREPARATION OF SODIUM NITROPRUSSIDE.

By F. S. Hyde.

Received November 18, 1896.

THE literature referring to this compound is usually more or less incomplete in regard to the details of its preparation; but, unless it is prepared in the pure state, its use as a delicate reagent is somewhat impaired.

Place sixty grams pulverized potassium ferrocyanide in a medium-sized evaporating dish, and pour on it a solution consisting of 150 grams concentrated nitric acid (sp. gr. 1.42) diluted with 100 cc. of water. Perform the operation under a hood or in a draft of air. When the evolution of gases has ceased, evaporate on a water-bath with occasional stirring until a few drops of the liquid, mixed with water in a test-tube, no longer give a blue color with ferrous sulphate solution but, instead, a dark greenish cloud.

It may be necessary to continue the evaporation for an hour or more before the final test is obtained. However, the liquid which has a dark reddish brown color, should be evaporated to about one-half of its original bulk to insure success. Allow to cool slowly over night, when crystals of potassium nitrate, blackened with impurities separate out in stellated masses or needles.

Decant the liquid from the crystals (which are rejected) and neutralize it by stirring in dry sodium carbonate, producing a greenish brown froth and a red solution. This will require some patience on account of frothing; heat on the water-bath, filter and wash. Evaporate the red filtrate to one-half of its bulk or more, and allow it to crystallize by slow cooling on the water-bath.

The crystallization consists mostly of crusts of sodium and potassium nitrates, together with little red needles of nitroprusside. Pour off the mother liquor and treat the impure crystals with as little water as possible just enough to dissolve the nitrates and leave the red crystals. Then with this nitrate solution in the same dish, wash or "pan out" (with a rotary motion) the impurities from the crystals.

These red crystals may be recrystallized by dissolving in a small quantity of distilled water and evaporating the deep-red

solution in a beaker to small bulk, or better still until there is an incipient formation of crystals.

By slow cooling, as before, clusters of beautiful red rhombic needles are produced. Pour off the mother liquor and dry the crystals on filter paper.

The yield averages about twenty-five per cent. by weight of the ferrocyanide used. In one experiment, the writer obtained seventeen grams of the nitroprusside from sixty grams ferrocyanide, or over twenty-eight per cent. yield. Very little is gained by treating the mother liquors for an extra yield, especially in the case of small amounts.

Sodium nitroprusside ($Na_2Fe(NO)Cy_5 + 2H_2O$) has no melting point. When heated in a glass tube, the crystal gives off moisture and becomes black without disintegration. It is slowly soluble in cold water, but easily soluble in hot water. It is best known as a very delicate reagent for sulphur in the form of alkaline sulphides, producing violet colored solutions which gradually lose color on standing.

Certain organic compounds also react with sodium nitroprusside. For instance, with a weak aqueous solution of nitroprusside made slightly alkaline with caustic soda, formaldehyde gradually produces a dark amber tint; acetic aldehyde gives a blood-red color, becoming darker on standing, and finally violet after two or three hours; acetone produces a blood-red solution fading gradually to orange-red; and benzaldehyde gives no coloration.

IMPROVEMENTS IN THE COLORIMETRIC TEST FOR COPPER.

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Received November 9, 1896.

THE modifications of Heine's "blue test" described in some of the text-books[1] recently issued, admit of some improvement in speed of operation, ease of manipulation (in presence of the large amounts of iron, alumina and silica in poor slags or tailings), and finally in the making of the standard colors from solution of pure copper.

Having made a study of the process, and having had it in use

[1] Furman's Manual of Practical Assaying, *1895*, p. 159; Kerl's Metallurgy of Copper; Peter's Modern Copper Smelting, *1895*, p. 65.