

Monolupine Dihydrochloride.—Five grams of monolupine in 15 cc. of acetone treated with 5 cc. of concd. hydrochloric acid (excess) produced a sirupy precipitate which was redissolved by the addition of just enough alcohol. After standing in an ice box for two hours the mixture solidified to a mush of crystals, which was filtered, washed with acetone and recrystallized from alcohol-acetone as fine white plates. Dried over calcium chloride the m. p. was 115–116° with rapid heating. Slower heating gave various results due to decomposition. When heated at 100° the dihydrochloride loses 2 moles of water and 1 mole of hydrochloric acid being converted into the monohydrochloride. The loss varies with the efficiency of the drying over calcium chloride.

Anal. Calcd. for $B \cdot 2HCl \cdot 2H_2O$, (367.2): Cl, 19.33; moisture (1 HCl plus $2H_2O$), 19.74%. Found: Cl, 19.6; moisture, 20.24, 20.25. (a)_D²⁰ -120.3° in water, $c = 2.1620$, $l = 2$, $a = -5.20^\circ$.

Monolupine Hydrochloride.—Prepared by heating the dihydrochloride at 110° to constant weight as white powder, very hygroscopic, m. p. 280°. It could not be recrystallized.

Anal. Calcd. for $B \cdot HCl$, (294.7): Cl, 12.05. Found: Cl, 12.06, 12.09.

Monolupine Gold Chloride.—To a solution of the dihydrochloride in water was added an excess of gold chloride solution. The yellow curdy precipitate was redissolved by heating and the filtered solution was set aside. On cooling a mass of yellow needles separated. These were filtered off, and dried; m. p. 167–168° (dec.). The mother liquor deposited a film of metallic gold after standing for twenty-four hours.

Anal. Calcd. for $B \cdot 2HAuCl_4 \cdot 3H_2O$, (992.6): Au, 39.73; H_2O , 5.44. Found: Au, 39.70; H_2O , 5.46.

Monolupine Methiodide.—Two grams of monolupine in 5 cc. of acetone mixed with 2 cc. of methyl iodide deposited crystals after two days of standing. These were recrystallized from alcohol twice and dried when the m. p. was constant at 257°.

Anal. Calcd. for $B \cdot CH_3I \cdot H_2O$ (418.2): I, 30.33; H_2O , 4.30. Found: I, 30.83, 30.85; H_2O , 4.06, 4.03.

Summary

Lupinus caudatus Kellogg contains 0.44 to 0.45 % of a new alkaloid, monolupine, $C_{16}H_{22}ON_2$, that closely resembles anagryne. Chemically this plant is distinct from *L. palmeri*.

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NOTES

The Preparation of Platinum Oxide for Catalytic Hydrogenations

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Platinum oxide for catalytic hydrogenations can be prepared more conveniently from ammonium chloroplatinate than from chloroplatinic acid by the well-known procedure of Adams.¹ By adding an excess of ammonia to a solution of chloroplatinic acid, ammonium chloroplatinate is precipitated. This is the basis for a convenient method of recovering platinum in spent catalysts.² The amount of catalyst produced from a given weight of ammonium chloroplatinate is almost exactly one-half the weight of the ammonium salt and is therefore very easily calculated. Ammonium chloroplatinate is not hygroscopic and is therefore weighed more easily than chloroplatinic acid. In starting from the ammonium salt, no water is used, and hence no spattering occurs in heating the mixture to the fusion temperature.

By the new procedure a given weight of ammon-

(1) Adams, "Organic Syntheses," John Wiley and Sons, Inc., New York, N. Y., Collective Vol. I, 1932, p. 452.

(2) Baldeschwieler and Mikeska, *THIS JOURNAL*, **57**, 977 (1935).

ium chloroplatinate is well mixed with ten times its weight of powdered sodium nitrate, and the mixture is heated gradually to the fusion point. During this process much gas is evolved, due presumably to the decomposition of ammonium nitrate, but the evolution is gentle and no spattering occurs. The fused mixture is held at 500° for twenty-five to thirty minutes and the platinum oxide is isolated according to Adams' directions. From 3.0 g. of the salt was obtained 1.51 g. of platinum oxide, no different in general appearance or activity from that prepared in the usual way. This experiment has been duplicated in several other laboratories and shortens the procedure for converting spent catalyst to platinum oxide by 25% or more.

The following data which compare the rates of hydrogenation of maleic acid and of benzaldehyde using catalysts prepared by the original (I) and by the modified (II) procedures were communicated by Dr. E. L. Baldeschwieler and are reported with his permission.³

(3) Catalyst II used in these experiments was prepared by adding ammonium chloroplatinate in small portions to fused sodium nitrate at 350° rather than by the more convenient procedure described above since this method was developed later.

