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·2HCl·2H₂O, (367.2): Cl, 19.33; moisture (1 HCl plus 2H₂O), 19.74%. Found: Cl, 19.6; moisture, 20.24, 20.25. (a)²⁹D -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·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·2HAuCl₄·3H₂O, (992.6): Au, 39.73; H₂O, 5.44. Found: Au, 39.70; H₂O, 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 $B \cdot CH_{3}I \cdot H_{2}O$ (418.2): I, 30.33; $H_{2}O$, 4.30. Found: I, 30.83, 30.85; $H_{2}O$, 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 anagyrine. Chemically this plant is distinct from L. palmeri.

WASHINGTON, D. C. RECEIVED MARCH 2, 1936

NOTES

The Preparation of Platinum Oxide for Catalytic Hydrogenations

BY WILLIAM F. BRUCE

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 1I 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.

Table	Ι		

A. Hydro	genation of Male	IC ACID	
	Total time, seconds		
Hydrogen absorbed, cc.	Catalyst I (original method)	Catalyst II (modified method)	
100	78	60	
200	123	98	
300	166	135	
400	208	173	
500	249	213	
600	288	253	
B. Hydro	ogenation of Benz	XALDEHYDE ⁴	
100	70	152	
200	125	239	
300	181	314	
4 00	2 40	388	
500	304	465	
600	372	545	

These results show that the catalyst prepared by the modified procedure was in this case somewhat better for the hydrogenation of maleic acid than that made in the usual way, while the reverse is true for the hydrogenation of benzaldehyde. Reference to the previous study of rates of hydrogenation by this same technique² shows that the catalysts reported here are even more active than those in the earlier work.

(4) The addition of a trace of $FeSO_4$ sometimes promoted the reaction and sometimes poisoned it.

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The Numbering of the Sparteine Molecule and its Derivatives

By JAMES FITTON COUCH

The constitution of sparteine as modified by Clemo and Raper [J. Chem. Soc., 644-645 (1933)] from the structure suggested by Ing [*ibid.*, 504-510 (1933)] may be rearranged to the following form, and the component atoms may then be



numbered in the fashion indicated beginning with a ring nitrogen atom in the conventional way. It would be more convenient in this particular case to start with carbon atom number 16 but it is better to adhere to the established rule. This arrangement of the sparteine formula which has been accepted by Prof. Clemo [personal communication] shows at once its symmetrical character and relationship to other lupine alkaloids and to cytisine and anagyrine. Lupanine becomes 2keto-sparteine, hydroxylupanine tentatively its 10-hydroxy derivative and anagyrine is 3,4,5,6tetradehydro-2-keto-sparteine. Lupinine may be formulated as



and cytisine



indicating a close relationship between these alkaloids since cytisine may be derived from (hypothetical) tetradehydro-2-keto-lupinine by condensation of methylamine across atoms 9 and 11 of the lupinine skeleton, atom 8 becoming atom 13 of cytisine. Sparteine may be theoretically derived from lupinine by condensation with piperidine in an analogous manner.

BUREAU OF ANIMAL INDUSTRY WASHINGTON, D. C. RECEIVED MARCH 2, 1936

Amino Alcohols Derived from 1,2,3,4-Tetrahydrodibenzofuran

BY RICHARD A. ROBINSON AND ERICH MOSETTIG

In logical connection with a study of derivatives of 4,5-phenanthylene oxide [Mosettig and Meitzner, THIS JOURNAL, 56, 2738 (1934)] the investigation of dibenzofuran derivatives was begun in this Laboratory in 1932 with the hope of finding in this series compounds resembling, particularly in their analgesic action, morphine. In our first paper dealing with dibenzofuran derivatives [Mosettig and Robinson, ibid., 57, 902 (1935)] we outlined in some detail the direction in which our further synthetic experiments were to proceed. In the meantime other research groups [Kirkpatrick and Parker, ibid., 57, 1123 (1935); Gilman, Smith, and Cheney, *ibid.*, 57, 2095 (1935)] have approached the synthesis of possibly analgesic and hypnotic substances in the dibenzofuran series in a way which is in part similar to ours; previous publications from these investigators had not indicated any intention of seeking morphine-like substances. In order to avoid unnecessary duplication of effort, some of our recent experimental results are submitted in this preliminary form.