isolated uterus of a rabbit and also a guinea pig failed to give a single contraction. Fifty milligrams was dissolved in Wesson oil and fed to a pregnant guinea pig. After a few hours the pig developed the characteristic tremble. The following day she was much worse and died the following night, but without aborting the foetus.

A post mortem showed the lungs filled with blood, congested. The heart was flabby or soft and blood coagulated. The kidneys were congested and filled with coagulated blood. The large intestine and stomach were filled with gas; the liver was soft, brownish in color and very tender. The adrenal was enlarged and the outside walls of the uterus were very congested and inflamed. Five embryos measured approximately 3 inches (7 cm.) in length from crown to rump. The union between placenta discs was lost and embryos fell out as soon as the uterus was opened. If administered to anesthetized cats, this amorphous compound caused a fall in blood pressure. It was estimated that one milligram of this material has a depressor action equivalent to 0.002 mg. of histamine hydrochloride. By intravenous injection in mice, the lethal dose of the amorphous compound was found to be 22.5 mg. per kg.

Discussion

It may be definitely concluded that the active constituent in this compound is not an ergot alkaloid, as the experimental data show the base could not be combined with a weak acid, nor did it respond to the alkaloidal test characteristic of the ergot alkaloids. Methods for the isolation of alkaloids of ergot of rye by Thompson,[6] Stuart,[5] Arthur Stoll and Ernst Burckhardt,[7] Smith, Sidney and Timmis,[8] Tswett perfected by Kuhn, Winterstein, and Karrer,[9] have been used in this work, but all were of no value thus far.

Hydrolysis with enzymes leads one to believe it is not a glucoside.

It was thought at one time the fungus contained the same alkaloids as ergot of wheat or rye, but the pharmacological data prove this to be an erroneous idea.

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(6) Marvin R. Thompson, *J. Am. Pharm. Assoc.*, **24**, Nos. 1-3 (1935).

(7) Arthur Stoll and Ernst Burckhardt, U. S. Patent 2,080,954.

(8) Sidney, Smith and G. M. Timmis, English Patent 460,387.

(9) M. Tswett perfected by R. Kuhn, A. Winterstein and P. Karrer, French patent, démandé le 9 janvier 1935, à 16h 25m, à Paris.

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On the Hydration of Dihydropimaric Acid

BY TORSTEN HASSELSTROM AND BURT L. HAMPTON

In recent communications we described the formation of a lactone on hydration of dihydroabietic acid present in heat-treated rosin [Hasselstrom, U. S. Patents 2,121,032, 2,121,033 (1938); Hasselstrom, Brennan and McPherson. THIS JOURNAL, **60,** 1267 (1938); Hasselstrom and McPherson, *ibid.*, **60,** 2340 (1938)].

We have now been able to prepare a similar lactone by hydration of dihydropimaric acid, m. p. 241–243° (corr.), $(\alpha)_D$ +19.2°, obtained on recrystallization of hydrogenated rosin (Staybellite A-2, by courtesy of the Hercules Powder Company) with methanol according to the procedure described in a previous paper [Hasselstrom and Bogert, THIS JOURNAL, **57,** 2118 (1935)]. The methyl ester of dihydropimaric acid was obtained by means of diazomethane, m. p. 78.5–79.5 (corr.). *Anal.* Calcd. for $C_{21}H_{34}O_2$: C, 79.2; H, 10.8. Found: C, 78.8; H, 10.8. Ruzicka and Frank (*Helv. Chim. Acta*, **15,** 1297 (1932)] have recorded the melting point 79–80° for the methyl ester of the dihydropimaric acid.

A mixture of 3.5 g. of dihydropimaric acid and 40 cc. of sulfuric acid, sp. gr. 1.84, was stirred intermittently for about twenty minutes at 5°, then poured onto cracked ice. The mixture was extracted with ether, the ether solution washed with water, with dilute potassium hydroxide solution and with water. After drying with anhydrous sodium sulfate, the ether was evaporated and the residue, a yellow oil, was dissolved in hexane. On standing, 1.6 g. of white crystals separated which, after recrystallization from acetone, melted constantly at 143–144° (corr.). *Anal.* Calcd. for $C_{20}H_{32}O_2$: C, 78.9; H, 10.5. Found: C, 78.9; H, 10.6; $(\alpha)_D$ −40° (in ethanol). In a mixed melting point test with an authentic sample of the lactone of hydroxytetrahydroabietic acid a depression was observed, the melting point of the mixture being 102–112° (corr.).

The lactone of the hydroxytetrahydropimaric acid was then saponified with a 10% solution of butyl alcoholic potassium hydroxide. After removal of the alcohol by steam distillation, the potassium salt of the hydroxy acid was dissolved in water and some unsaponified lactone removed by filtration. The alkali solution was made acid with dilute acetic acid and the product recrystallized from acetone, m. p. 143–144° (corr.). This product was insoluble in alkali and did not lower the melting point in the mixed melting point test with the original lactone, showing that the intermediately formed hydroxytetrahydropimaric acid had been transformed into the original lactone.

This investigation is being continued.

G. & A. LABORATORIES, INC.
SAVANNAH, GEORGIA RECEIVED MARCH 10, 1939

A Modification of Bettendorff's Arsenic Test. II. Catalyzed by Mercury[1]

BY W. BERNARD KING AND F. E. BROWN

In a previous paper[2] the authors reported that traces of mercuric chloride hastened markedly the reduction of arsenic compounds with stannous chloride. They also found that the time required for the appearance and development of the arsenic suspension was a function of the concentration of mercuric chloride in the solution. This behavior was utilized to determine the concentration of mercuric chloride in concentrations as low as 2×10^{-8} molar mercuric chloride. The authors, following the example of other investigators, used the terms "inductor" and "its inductive effect" without questioning the correctness of these terms. In the following report, data are presented which show that the modified Bettendorff test is catalytic and not induced.

Reagents and Procedure

The Preparation of Mercurous and Mercuric Chloride Solutions of Identical Concentrations.—By means of a microbalance a few milligrams of mercurous chloride was weighed out, dissolved in concentrated hydrochloric acid and diluted with the same solvent until the solution was 9×10^{-7} molar. One hundred cubic centimeters of this solution was thoroughly saturated with chlorine gas which was passed through some of the same solvent before going into the sample. The chlorine was allowed to stand overnight in contact with the mercurous chloride solution, after which the excess was swept out completely by a current of hydrogen chloride gas. It required about two hours for the greenish yellow color of the chlorine to disappear. Another sample of this 9×10^{-7} molar mercurous chloride solution was then saturated with hydrogen chloride gas in order to have both the oxidized and non-oxidized portions at the same concentration of hydrogen chloride. The mercuric chloride solution, *i. e.*, the oxidized portion, and the mercurous chloride solution, the non-oxidized portion, were then compared with regard to their catalytic effects.

The data reported were secured by the use of 50-cc. Nessler tubes. Two comparison tubes A and B were employed. The time at which a changing suspension becomes darker than an unchanging standard is more easily determined than the time at which reduction is complete. This required of course that the unchanging standard be less dense than the completely reduced experimental sample. The values obtained are shown in Table I. The times required to match the colors or densities of reference solutions A and B are expressed in minutes and seconds.

TABLE I

A COMPARISON BETWEEN THE CATALYTIC EFFECTS OF MERCUROUS AND MERCURIC CHLORIDE SOLUTIONS OF IDENTICAL CONCENTRATION

Tube	Cc. $HgCl$	Cc. $HgCl_2$	Cc. HCl	Cc. $SnCl_2$	Cc. As_2O_3	Time for reduction A	B
1	10	0	35.5	2.5	2	3:40	6:55
2	0	10	35.5	2.5	2	3:45	7:00
3	10	0	35.5	2.5	2	3:36	6:58
4	0	10	35.5	2.5	2	3:37	7:00
5	10	0	35.5	2.5	2	3:30	6:56
6	0	10	35.5	2.5	2	3:36	6:30
7	0	0	45.5	2.5	2	11:00	19:40

The concentration of the arsenious oxide solution was 0.001 *M*, of the stannous chloride approximately 8.0 *M*. Standard A consisted of completely reduced arsenious oxide in which the final concentration after dilution was 0.00001 *M*. Standard B had a final concentration which was double that of A.

The results show quite conclusively that mercuric and mercurous chloride solutions have equal catalytic power in the reduction of arsenic compounds with stannous chloride. They also suggest that the actual catalyst is the mercury atom formed by the reduction of the mercury salt.

Confirmation of the probability that free mercury was the catalytic agent, and not the mercury ions or its salts was established by a simple experiment. All that needed to be changed in the previous experiments was the order of adding the reagents. Instead of adding stannous chloride last, as had been the practice, it was added just after putting in the mercury salts, causing them to be reduced to free mercury. The arsenious oxide solution was added last. A glance at the values as shown in Table II shows that com-

(1) Original manuscript received May 9, 1938.

(2) King and Brown, *Ind. Eng. Chem., Anal. Ed.*, **5**, 168 (1933).