Anal. Calcd. for $C_{21}H_{18}O_6$: C, 68.85; H, 4.92. Found: C, 68.53; H, 4.90.

A mixture of equal parts of this substance and the acetate of chrysophanic acid-9-anthranol obtained from chrysarobin,[7] m. p. 239.6–240°, melted at 237.8–238.2°, showing that the compounds were identical.

Chrysophanic Acid.—A solution of 0.065 g. of chrysophanic acid-9-anthrone, obtained from aloe-emodin-9-anthrone, in 4 cc. of glacial acetic acid was treated with 0.036 g. of chromic acid in a little water. The mixture was warmed on the steam-bath for a half hour, diluted with water and cooled; yield, 0.02 g. of orange powder, m. p. 190.5–192°, from alcohol, using "Norit."

Anal. Calcd. for $C_{15}H_{10}O_4$: C, 70.87; H, 3.94. Found: C, 71.29; H, 3.80.

A mixture of equal parts of this substance and chrysophanic acid prepared from chrysarobin, m. p. 193–194°, melted at 191–192.5°, proving that the compounds were identical.

Summary

The product obtained from aloin by hydrolysis with an aqueous solution of borax is 1,8-dihydroxy-9-anthrone-3-carbinol.

ST. LOUIS, MISSOURI RECEIVED FEBRUARY 26, 1934

[CONTRIBUTION FROM THE INSECTICIDE DIVISION, BUREAU OF CHEMISTRY AND SOILS]

Lonchocarpic Acid, a New Compound from a Species of Lonchocarpus

BY HOWARD A. JONES

In the course of an investigation of plants for their rotenone content numerous species of *Derris* and *Lonchocarpus* have been studied in the Insecticide Division. A recent sample of root material of an unknown species of *Lonchocarpus* from Venezuela gave no qualitative color test for rotenone.[1] When subjected to the usual carbon tetrachloride extraction used in determining rotenone,[2] about 2.3% of crystalline material readily separated from the extract. This material, purified by recrystallization from acetone and carbon tetrachloride, had a melting point of 201° and a methoxyl content of 7.0%.

A larger sample of the same root was then obtained from the same source, and the bark and inner portion of the root were separated. These portions were extracted separately with acetone, the extracts were evaporated to small volume, and the material was caused to crystallize by the addition of carbon tetrachloride and subsequent cooling. The bark yielded 3.7% crystalline material and 7.2% total extractives, while the inner portion of the root gave 1.5% crystalline material and 2.4% total extractives. The average for the whole root based on the proportion of bark to inner portion was 2.1% crystalline material and 3.7% total extractives. The crystalline material separated more readily and in an apparently purer state from the extract of the bark than from that of the inner portion of the root.

The crystalline material from the inner portion of the root was recrystallized, but could not be obtained in a satisfactorily pure condition. The melting point of the recrystallized material was 197°.

The crystalline material from the bark as first separated had a melting point of 199°. It was recrystallized once from acetone and carbon tetrachloride and then from acetone alone. A melting point of 201.5° was obtained on this purified material. (The compound also occasionally exhibited a melting point of 220–221°, probably indicating dimorphism.) A further recrystallization from amyl acetate did not change the melting point.

The pure material is readily soluble in acetone and chloroform, sparingly soluble in benzene and amyl acetate, and only slightly soluble in carbon tetrachloride and petroleum ether. It is optically inactive in chloroform solution. It contains no nitrogen. The analytical figures for carbon, hydrogen and methoxyl together with the results of the titration of the material with alkali indicate an empirical formula of $C_{26}H_{26}O_6$, on the assumption that the compound is a monocarboxylic acid with one methoxyl group. The name "lonchocarpic acid" has been given to the compound.

Experimental

Preparation of Lonchocarpic Acid.—Five hundred grams of ground root of *Lonchocarpus sp.* was extracted with acetone. The extract was evaporated to about 300 cc. and carbon tetrachloride added while hot until precipitation began. The extract was then cooled in the refrigerator for several days. The crystalline material was separated, the filtrate evaporated to about one-half the volume and again cooled. A second crop of crystals was obtained.

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(1) Jones and Smith, *Ind. Eng. Chem., Anal. Ed.*, **5**, 75 (1933).

(2) Jones, *ibid.*, **5**, 23 (1933).

The first and second crops of crystals were combined and dried (m. p. 199°). Eighteen and seven-tenths grams of crystalline material was obtained. Sixteen and seven-tenths grams of this was dissolved in 450 cc. of hot acetone, charcoal added and the solution filtered while hot. Forty cc. of hot carbon tetrachloride was added to the filtrate and the solution cooled in the refrigerator for several days. The crystalline material, separated and dried, amounted to 5.5 g. A second crop of less pure material obtained from the filtrate amounted to 4.8 g. A portion of the first crop of crystals was dissolved in hot acetone and the solution cooled. The separated crystalline material was pure white, and after thorough vacuum drying had a melting point of 201.5°.

Anal. Calcd. for $C_{26}H_{26}O_6$: C, 71.86; H, 6.03; OCH_3, 7.14; mol. wt., 434. Found: (micro)[3] C, 71.63, 71.56; H, 6.17, 6.03; (semimicro) OCH_3, 7.19, 7.19; mol. wt. 452 (titn. phenolphthalein); phenol, negative; nitrogen, none.

The material crystallizes from amyl acetate in the form of short, columnar crystals with the following refractive indices:[4] $\alpha = 1.510$; $\beta = 1.718$; γ considerably greater than 1.77.

The plant material for this work was kindly furnished by Mr. Oswaldo Stelling of Caracas, Venezuela.

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[3] The writer is indebted to Mr. J. R. Spies of the Insecticide Division for the determination of carbon and hydrogen by micro-combustion.

[4] Refractive indices were determined by Mr. E. L. Gooden of the Insecticide Division.

NOTES

Crystalline Guaiazulene

BY KENNETH STEWART BIRRELL

In the course of an investigation at present being carried out on the hydrocarbon designated by Ruzicka[1] as S-guaiazulene, it was observed that the liquid (b. p. 176° (17 mm.)) is in reality supercooled and when once frozen in liquid air remains solid indefinitely.

The solid melted initially at 27–28° and after recrystallization from alcohol had a constant m. p. of 31.5°. The picrate of the purified substance melted at 122° as before.

The crystalline azulene has the appearance of small shining blue-violet plates. Crystallographic examination shows that the substance is biaxial with a small optic axial angle and the crystals are probably orthorhombic.

Axial ratios: $a:b:c = 0.56:1:1.34$

Interfacial angles:

100 ∧ 110	29°7′
110 ∧ $\bar{1}10$	121°46′
011 ∧ $0\bar{1}1$	106°30′ ± 1°

Habit: tabular, parallel to macropinacoid (100)

The hydrocarbon described by Ruzicka as Se-guaiazulene has been prepared and found to exhibit the same phenomenon. This substance shows the same melting point as the S-guaiazulene and gives no melting point depression on admixture with the latter. A comparison of the melting points of the picrates and of the styphnates of the two substances lends support to the view that they may be identical and not isomeric as stated by Ruzicka.[1] In some cases the Se-guaiazulene even after regeneration from the picrate may fail to crystallize. Here the melting point of the picrate remains low but gives no melting point depression with S-guaiazulene picrate. It seems probable that this behavior is connected with the efficiency of the Se dehydrogenation and that an obstinate impurity inhibits crystallization. With S-guaiazulene purification through the solid may advantageously replace purification through the picrate. It is hoped to extend this work to the other isomeric azulenes described by Ruzicka.

Acknowledgment is made to Prof. J. K. H. Inglis for helpful suggestions and to Dr. F. J. Turner of the Geology Department, who kindly worked out the crystallography.

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(1) Ruzicka and Haagan-Smit, *Helv. Chim. Acta*, **14**, 1104 (1931).

A Convenient Modification of the Kiliani Synthesis of Higher Carbon Acids (or their Lactones) from Reducing Sugars[1]

BY C. S. HUDSON, OLIVE HARTLEY AND CLIFFORD B. PURVES

The condensation of slightly alkaline, aqueous hydrogen cyanide[2] or of potassium or barium cyanide[3] with the carbonyl group of a reducing

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(1) Published by permission of the Director of the National Bureau of Standards and the Surgeon General of the U. S. Public Health Service.

(2) Kiliani, *Ber.*, **19**, 767 (1886).

(3) Rupp and Hoelzle, *Arch. Pharm.*, **253**, 404–415 (1915). See also the Höchster Farbwerke German Patents, 24,526 and 253,754.