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.

(3) The writer is indebted to Mr. J. R. Spies of the Insecticide Division for the determination of carbon and hydrogen by microcombustion.

(4) Refractive indices were determined by Mr. E. L. Gooden of the Insecticide Division.

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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:

$100 \wedge 110$	29°7′
$110 \wedge \bar{1}10$	121°46′
$011 \wedge 0\bar{1}1$	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.

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

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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

(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.