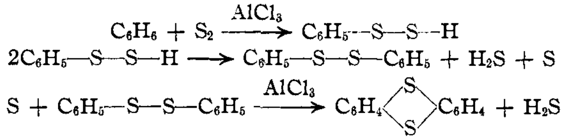
The hydrogen disulfide decomposed, however, as rapidly as it was formed

$$2NO_2—C_6H_4—S—S—H \longrightarrow NO_2—C_6H_4—S—S—C_6H_4—NO_2 + S + H_2S$$

In view of this result it seems likely that the correct explanation of the formation of thianthrene from sulfur, benzene and aluminum chloride is



$$C_6H_6 + S_2 \xrightarrow{AlCl_3} C_6H_5—S—S—H$$

$$2C_6H_5—S—S—H \longrightarrow C_6H_5—S—S—C_6H_5 + H_2S + S$$

That is, it is not necessary to assume, as was done by Dougherty and Hammond,[1] that the diphenyl disulfide was formed from diphenyl sulfide and sulfur. The reaction would undoubtedly take the easier course as outlined in the above equations.

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The Electron Diffraction Investigation of Some Higher Aliphatic Halides

By J. A. A. Ketelaar and K. J. Palmer

The van der Waals cohesion forces acting between different molecules is without doubt also active between different parts of the same molecule when, as in the higher aliphatic hydrocarbons, due to the rotation around the C–C bond, these parts can come close together without an appreciable increase of energy.[1] Consequently these molecules will tend to have a bent or even a more or less spherical form rather than that of a stretched chain.

In order to test this by a direct method, we have taken electron diffraction photographs of 1,10-dibromodecane, 1,5-dibromopentane, and 1,4-diiodobutane. These photographs show only a very few diffuse rings and much background; this appearance can be accounted for by the interactions between halogen and the two nearest carbon atoms and gives no indication of the halogen–halogen interaction, which would have a large effect in case the halogen–halogen distances were constant. The interpretation of these more or less negative results seems to be that the chains are indeed not in the stretched form nor in any one contracted form, but that they occupy at the temperature of the experiment a large number of configurations so that no constant large interatomic distances are present. This temperature effect will no doubt also be shown by other large molecules.

We are indebted to the Experimental Station of du Pont de Nemours, Wilmington, Delaware, to Dr. J. van Alphen, Leyden, and to Dr. H. de Vries Robles, Amsterdam, for providing us with samples of the substances used in this investigation.

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[1] Even if free rotation is not possible as indicated by the results of J. D. Kemp and K. S. Pitzer, *J. Chem. Phys.*, **4**, 749 (1936), [This Journal, **59**, 276 (1937)], and of J. B. Howard, *Phys. Rev.*, **51**, 53 (1937), this consideration will remain true, as equilibrium positions differing by a rotation over 120° can be reached, thus leading to a large number of possible configurations.

The Isolation of Sucrose from Soybeans

By H. R. Kraybill, R. L. Smith and E. D. Walter

Street and Bailey[1] have reported the presence of sucrose in soybeans based on an increased reducing value of an extract of the beans following hydrolysis with invertase. Since invertase hydrolyzes other sugars in addition to sucrose, this does not give conclusive evidence of the presence of sucrose. Other workers[2,3] have reported the difficulty in crystallizing the sugars from soybean extract. Tanret[4] reported the isolation of sucrose from soybeans but he did not give supporting data characterizing the substance isolated. We have isolated a crystalline substance from soybeans and identified it as sucrose.

In experiments[5,6] on the extraction of soybeans with different solvents it was noted that a crystalline substance was deposited on the sides of the flask when acetone was used as the solvent. The impure substance collected from the sides of the flask gave negative tests for nitrogen and a positive Molisch test. The substance failed to reduce Fehling's solution.

A larger quantity of the substance was obtained by extracting the beans with petroleum ether to

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[1] J. P. Street and E. M. Bailey, *J. Ind. Eng. Chem.*, **7**, 853–858 (1915).

[2] Block, *Bull. sci. pharmacol.*, **14**, 536–551 (1907).

[3] Sadakichi and Sato, Technology Reports, **2**, No. 2 (1921), Imperial University, Sendai, Japan.

[4] Georges Tanret, *Bull. soc. chim.*, **13**, 176–182 (1913).

[5] R. L. Smith, Master of Science Thesis, Purdue University, May, 1931.

[6] Annual Report Purdue Univ. Agr. Expt. Station, 1931, p. 54.

remove the fat and then extracting with acetone. After evaporation of the acetone the residue was extracted repeatedly with ether to remove traces of fat. The residue was then dissolved in a small quantity of water and treated with absolute alcohol; 1.4551 g. of a crystalline substance was obtained. From the specific rotation and reducing value following hydrolysis with invertase the substance was found to be sucrose.

The sample was dissolved in water and made to 25 ml. at 20°. The $[\alpha]^{20}\text{D}$ of the solution was found to be +66.45°. The theoretical value for sucrose is +66.48°. Five ml. of the solution was diluted to 500 ml. Twenty-five ml. aliquots containing 14.551 mg. of the substance were hydrolyzed with melibiase-free invertase. The reducing value was determined by a combination of the Quisumbing and Thomas and Bertrand methods. The average of five determinations was 14.72 mg. of sucrose; the theoretical amount was 14.55 mg.

Recently we have isolated sucrose from soybeans by two different methods.

In the first method soybean flakes were extracted with petroleum ether or with ether followed by extraction with 99% alcohol. The alcohol extract was concentrated on a steam-bath until sugar began to crystallize on the sides of the beaker and on a stirring rod standing in the solution. After standing for two days the crystals were filtered and washed with ether. A 5% solution of the first crop without recrystallization gave $[\alpha]^{20}\text{D}$ 65.81°. Refractive indices determined by means of the polarizing microscope gave $\alpha = 1.537$ $\beta = 1.565$, $\gamma = 1.57$. This is in agreement with the constants in Groth's "Chemische Krystallographie," Volume III, p. 448, namely, $\alpha = 1.5371$, $\beta = 1.5653$, $\gamma = 1.5705$. By this simple method sucrose can be obtained much easier and in larger yields than by the customary method in which lead acetate and barium hydroxide are used to clear the solution.

In the second method a 5-liter portion of an 80% alcohol extract of soybean flakes was concentrated before a fan to a thick sirup. About 2 liters of water was added and the solution was treated with lead acetate. The precipitate was filtered and the filtrate was treated with barium hydroxide. The lead and barium were removed with sulfuric acid. The solution was shaken with ether to remove acetic acid, and was then concentrated to a thick sirup. An equal volume of 99% alcohol was added and the sirup settled to the bottom. After stirring the sirup in the alcohol on the steam-bath the alcohol was decanted and let stand for several days. Crystals appeared in the alcohol extract. The first two fractions were combined and recrystallized. A 5% solution of these crystals gave a specific rotation of 66.57°. The refractive indices were the same as those of pure sucrose.

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Efficiency at 90° of Phosphorus Pentoxide as a Desiccant

BY DONALD A. LACOSS AND ALAN W. C. MENZIES

Information as to the efficiency of this desiccant at other than room temperatures appears to be absent from the literature. Accordingly, in order to avoid any mere assumptions about the matter, we made the experiment here described at a time when we contemplated desiccation experiments at temperatures near 100°.

A current of air was passed at the rate of about 650 cc. per hour through our apparatus train for thirty-five days, namely, about 558 liters in all. This entered successively (1) a 500-cc. calcium chloride tower, (2) a series of three helical type washing bottles containing sulfuric acid of such a concentration as to yield an aqueous pressure of about 1 mm.,[1] (3) a U-tube of about 80-cc. capacity charged with Baker "plugs" of purified phosphorus pentoxide in half-inch (1.25-cm.) layers separated by layers of ignited asbestos,[2] (4) an ordinary stoppered 30-cc. U-tube containing magnesium perchlorate, (5) a U-tube of purified phosphorus pentoxide charged like (3) and of size similar to (4), (6) a calcium chloride guard tube, (7) a calibrated gas meter and (8) an aspirating device. Connections for the weighing tubes (4) and (5) were lubricated ground joints. Tube (3) was maintained at 90° in a thermostat, while the rest of the train was at room temperature.

Tubes (4) and (5) were weighed from time to time against counterpoises which were handled in a like manner prior to weighing. Although over 0.5 g. of water had entered tube (3) as vapor, no change in weight was found in either (4) or (5).

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(1) *Cf.* E. M. Collins, *J. Phys. Chem.*, **37**, 1191 (1933).

(2) *Cf.* H. B. Baker, *J. Chem. Soc.*, **65**, 611 (1894).