into the reaction flask. This rate of reduction in volume is related definitely to the rate of polymerization of isoprene. Other experiments in which catalyst was omitted showed no change in the level of the meniscus; experiments in which the U-tube was open to the air showed complete evaporation of the isoprene outward in three and a half days. These facts show that the aluminum chloride determines the evaporation rate. The isoprene chamber and catalyst flask were maintained at 24–26° for a period of one thousand hours. Readings were taken at intervals, the percentage polymerized was calculated based on the original length of the liquid isoprene column, and the flask was shaken at intervals to keep the catalyst in a finely divided state. At the end of the reaction less than 3% remained as liquid isoprene polymer in the side arm. This was viscous, high boiling and smelled of terpenes. The catalyst mass was disintegrated, neutralized with ammonia and filtered. The clear filtrate was distilled at a temperature of 200° at 2 mm. This gave the soluble resin. The cake on the filter was completely soluble in dilute hydrochloric acid, indicating absence of insoluble polymer and the presence of inorganic material.

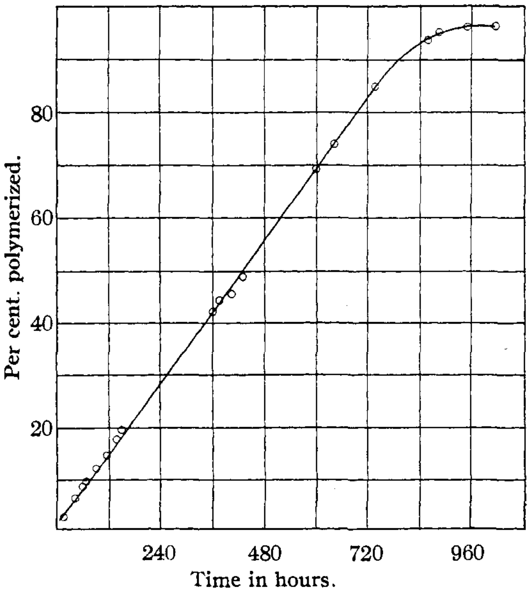

Fig. 2.

Figure 2 indicates the rate of polymerization and vaporization of isoprene. Table I is a relative comparison of the results obtained from both liquid[3] and vapor phase reaction.

TABLE I

Yields	Vapor phase	Liquid phase
Soluble polymer, %	98	12
Insoluble polymer, %	0	84
Molecular weight of soluble polymer in benzene	1300	600
Acid value	0.7	0.4
Iodine value	176	202

Summary.—Vapor phase polymerization of isoprene produces only soluble polymer; insoluble polymer is not formed. The molecular weight of this new polymer is approximately 1300. The molecular weight of the soluble polymer is approximately double that obtained by the usual liquid phase polymerization.

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[3] Thomas and Carmody, THIS JOURNAL, **54**, 2480 (1932).

Preparation of *p*-Phenyltriphenylcarbinol, and the Existence of a Metastable Form

BY DANIEL B. CLAPP AND AVERY A. MORTON

p-Phenyltriphenylcarbinol was first prepared by Schlenk[1] from *p*-iododiphenyl, magnesium, and benzophenone. The preparation of this substance according to the method of Morton and Stevens,[2] using sodium instead of magnesium, has proved to be simpler and more economical. The yield of the carbinol is excellent, and *p*-chlorodiphenyl is substituted for the more expensive *p*-iododiphenyl.

In the first experiment, the carbinol was isolated, after three recrystallizations from ligroin, as white, prismatic needles; m. p. 112–113°. The carbinol prepared according to Schlenk melted at 136°. The form melting at 112–113° was shown to be pure by a cooling-curve determination on the melt, and to be a metastable form of *p*-phenyltriphenylcarbinol by its conversion into the higher melting form by heating at 100° for several hours, or by repeated crystallization from high boiling solvents. Also, the metastable form with acetyl chloride gave *p*-phenyltriphenylchloromethane,[1] m. p. 146–147°, and with phenol *p*-hydroxy-*p'*-phenyltetraphenylmethane,[3] m. p. 182°, both derivatives being identical, respectively, with those prepared from the carbinol melting at 136°.

In all subsequent preparations of the carbinol, only the stable high melting form was isolated,

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[1] Schlenk, *Ann.*, **368**, 298 (1909).

[2] Morton and Stevens, THIS JOURNAL, **53**, 4028 (1931).

[3] Boyd and Hardy, *J. Chem. Soc.*, 1006 (1929).

and all attempts to convert this form into the metastable form were unsuccessful.

Experimental

In a 2-liter flask fitted with mercury-seal mechanical stirrer and reflux condenser, were placed 150 g. (0.8 mole) of *p*-chlorodiphenyl (Eastman Kodak Co. Techn.), 145 g. (0.8 mole) of *p*-benzophenone, and 525 cc. of anhydrous benzene. The mixture was heated until all of the solid had dissolved. Forty-five grams (0.2 mole) of sodium sand was then added, in small portions, through the top of the condenser, each portion being washed down with a little dry benzene. Just enough heat was applied to keep the reaction mixture refluxing gently. The time required for the addition of the sodium was two hours. The reaction mixture was then refluxed for two hours longer. After cooling, the unreacted sodium was destroyed by the addition of methanol. The mixture was then hydrolyzed with dilute hydrochloric acid, the benzene layer separated, and the carbinol isolated by removal of the benzene. After repeated recrystallization from ligroin (b. p. 90–120°) 180 g. of the carbinol was obtained, m. p. 135–136°; yield 67%.

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CAMBRIDGE, MASS.
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A Calcium Chloride Compound of *α-d*-Galactose[1]

BY RAYMOND M. HANN AND C. S. HUDSON

Although crystalline calcium chloride addition compounds of several aldose sugars have been obtained, one containing *d*-galactose as a component does not appear to have been described. The present note summarizes the experimental details leading to the isolation of *α-d*-galactose calcium chloride trihydrate.

Ten grams of *α-d*-galactose was dissolved in 16 cc. of water and 16 g. of calcium chloride dihydrate (2 moles) was added. Upon slowly concentrating the thin sirup in a desiccator, spontaneous crystallization occurred in seven months in a yield of 15 g. The substance may be recrystallized by solution in three parts of 90% alcohol containing 10% of calcium chloride dihydrate, separating in groups of clear colorless elongated prisms which melt at 129–130° (corr.). It must be preserved in a dry atmosphere to prevent deliquescence. Analysis and the course of mutarotation indicate that the compound is *α-d*-galactose calcium chloride trihydrate.

Anal. Calcd. for $C_6H_{12}O_6 \cdot CaCl_2 \cdot 3H_2O$: Ca, 11.6; Cl, 20.5; galactose, 52.2. Found: Ca, 11.7; Cl, 20.4; galactose (by hypoiodite) 52.2.

MUTAROTATION OF *α-d*-GALACTOSE·$CaCl_2 \cdot 3H_2O$ IN WATER

Concentration 2.0173 g. in 25 cc. solution; tube length, 2 dm.; $T = 20 \pm 0.5°$.

Time after making soln., min.	$[\alpha]^{20}D$	$[\alpha]^{20}D$ based on galactose	$k_1 + k_2$	$k_1 + k_2$ pure galactose
2.7	+75.82°	+145.3°	....	....
3.8	74.65°	143.0°	0.0143	....
5.8	72.82°	139.5°	.0133	0.0127
7.8	71.43°	136.9°	.0121	.0117
8.9	70.57°	135.2°	.0121	.0118
10.9	69.28°	132.7°	.0116	.0107
14.1	67.67°	129.7°	.0108	.0105
25.0	62.42°	119.6°	.0101	.0096
38.4	57.92°	114.9°	.0094	.0092
48.0	56.13°	107.5°	.0094	.0089
73.0	50.19°	96.2°	.0092	.0086
∞	42.68°	81.8°	....	....

The extrapolated initial rotation of the galactose in each case was about +151° and the change in rate as mutarotation progressed was quite similar. The somewhat high equilibrium rotation, based on galactose, is probably due to the presence of calcium chloride.

NATIONAL INSTITUTE OF HEALTH
U. S. PUBLIC HEALTH SERVICE
WASHINGTON, D. C.
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[1] Publication authorized by the Surgeon General, U. S. Public Health Service.

Monolayers and Multilayers of Chlorophyll

BY IRVING LANGMUIR AND VINCENT J. SCHAEFER

Monolayers of chlorophyll[1] spread on a water surface from a benzene solution may be deposited in successive layers on a solid plate to form multilayers.

A benzene solution of chlorophyll illuminated by blue light shows red fluorescence. During the spreading of a drop of the solution on a water surface fluorescence disappears as soon as the benzene has evaporated.

Chlorophyll monolayers on water behave as two-dimensional liquids until subjected to a pressure of more than 20 dynes cm.$^{-1}$, when they become quite viscous. The *p*H of the water has apparently no effect on the viscosity below this pressure.

The absolute viscosities[2] of chlorophyll mono-

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[1] Made by Dr. A. Stoll of Basel and obtained from Dr. J. Franck of the Physics Department of Johns Hopkins University.

[2] Irving Langmuir and Vincent J. Schaefer, THIS JOURNAL, not yet published.