remaining solution acidified and extracted with ether. The extract on evaporation gave succinic acid identified by mixed m.p. both of the acid and its *p*-bromophenacyl ester with authentic specimens.

Reduction of Catalytic Pentenol to Pentanol.—The alcohol (5 g.) was reduced at atmospheric pressure and room temperature using palladium oxide in methanol and hydrogen (1182 cc.). The product, b.p. 136–138°, n^{27}_{D} 1.4090, gave a 3,5-dinitrobenzoate, m.p. from petroleum ether 45–46°, and an α-naphthylurethan, m.p. 65.5–66.5°. It was identified as 1-pentanol by mixed m.p. of each of the two derivatives with authentic material.

Authentic 4-Pentenol.—Tetrahydrofurfuryl bromide was treated with sodium in ether as described by Paul.[10] The yield of product, b.p. 135–139°, was 70%. The dinitrobenzoate after two crystallizations from petroleum ether had m.p. 46–48°.

Anal. Calcd. for $C_{12}H_{12}N_2O_6$: C, 51.39; H, 4.32; N, 10.00. Found: C, 51.34; H, 3.84; N, 10.32.

Authentic 4-Pentenal.—The alcohol (5 g.) was added slowly to water (60 cc.), potassium dichromate (2 g.) and sulfuric acid (concentrated, 2 cc.) at 50–60°. The distillate gave a dinitrophenylhydrazone which after crystallizing from ethanol had m.p. 116–117.5°.

Pentanoic Acid, 5-Hydroxypentanoic Acid Lactone and Tetrahydrofurfuryl Pentanoate.—These substances were formed in appreciable amounts only with the 50 and 10 iron catalysts. Fractions (3) and (4) contained pentanoic acid, b.p. 180–190°, estimated by titration with standard alkali, extracted from fraction (4) by shaking with excess saturated sodium bicarbonate solution recovered by acidification and identified by preparation of the *p*-bromophenacyl ester. The product from 50 iron catalyst gave a derivative, m.p. 71–72°, mixed m.p. with authentic ester (m.p. 72–73.5°), 72–73.5° and the 10 iron catalyst, m.p. 73–74.5°, mixed m.p. 72.5–73.5°.

In the case of the 96 and 50 iron catalysts, the organic layer left after bicarbonate treatment contained the lactone. It was estimated either by back titration after shaking a sample with excess cold sodium hydroxide (*N*) or by adding liquid ammonia to a sample in toluene solution to precipitate the adduct. This compound served for identification and had m.p. 103.5–105° and 105–106°, respectively, from the two catalysts. The melting point was not depressed by admixture with authentic material (m.p. 108–109°). In the ammonia reaction, it was necessary to use freshly distilled material to ensure the presence of the monomeric lactone. The product, b.p. 220–240°, from the 96 iron catalyst went semi-solid after several months due to formation of the linear polymer. The polymer does not form the characteristic crystalline derivative with ammonia.

With the 50 iron catalyst, the ammonia method gave 12.6% and titration 12.6, 12.7% of the lactone.

After removal of pentanoic acid and the lactone by treatment with cold aqueous alkali the residue from the 50 iron catalyst had b.p. 230–245°. It went slowly into solution on refluxing with 20% sodium hydroxide. Extraction of this solution five times with chloroform gave tetrahydrofurfuryl alcohol, b.p. 175–180°, identified as 3,5-dinitrobenzoate, m.p. 69–72°, mixed m.p. with authentic (m.p. 81–84°), 70–73°. Re-extraction of the acidified aqueous layer with chloroform gave pentanoic acid, b.p. 180–190°, identified as the *p*-bromophenacyl ester. These reactions showed that the high boiling residue was tetrahydrofurfuryl pentanoate. It was quantitatively determined by back-titration after heating a sample (1 g.) with a standard solution of potassium hydroxide in diethylene glycol to boiling for 15 min. After prolonged alkali treatment, the material from the 10 iron catalyst gave a small fraction, b.p. 225–235°. This was probably impure tetrahydrofurfuryl tetrahydrofuryl ether since it gave the dinitrophenylhydrazone of 4-hydroxybutanal, m.p. 118.5–119°, identified by mixed m.p. but the precipitation was not quantitative.

COLUMBUS 10, OHIO RECEIVED FEBRUARY 16, 1951

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(10) R. Paul, *Bull. soc. chim.*, [5] **2**, 745 (1935).

[CONTRIBUTION FROM AVERY LABORATORY OF THE UNIVERSITY OF NEBRASKA]

Studies in the Furan Series. Reactions with Chloral

BY JOE R. WILLARD AND CLIFF S. HAMILTON

Furan and 2-methylfuran have been treated with chloral in the presence of zinc chloride to yield 1-(2-furyl)- and 1-(5-methylfuryl-2)-2,2,2-trichloroethanol, respectively, and the corresponding acetates have been prepared. N,N-Diethyl-2-furamide has been treated with chloral in the presence of concentrated sulfuric acid to give 1-(5-N,N-diethylfuramido-2)-2,2,2-trichloroethanol. Repeated attempts with 2-furanilide with chloral have been unsuccessful. The first furan analog of DDT, 1,1-bis-(5-carbomethoxyfuryl-2)-2,2,2-trichloroethane, has been prepared by condensation of methyl 2-furoate with chloral. The dehydrochlorination and hydrolysis of the latter compound are described. Ultraviolet absorption spectra of representative compounds are presented.

Since the advent of DDT (1,1-bis-(*p*-chlorophenyl)-2,2,2-trichloroethane) as a contact insecticide,[1] many analogous compounds have been prepared and tested; however, little attention has been given, with the exception of the thiophene analogs,[2] to preparation of related compounds containing heterocyclic rings. Therefore, a study of the preparation of some furan analogs was undertaken.

Due to the sensitivity to inorganic acids, which is characteristic of the furan nucleus when stabilizing negative substituents are not present,[3] the usual method of preparation of DDT-type compounds; *viz.*, the condensation of the aromatic hydrocarbon with chloral in the presence of concentrated or fuming sulfuric acid[2,4] would not be applicable to the furan analogs. Studies by Hartough and Kosak[5] indicated that catalytic amounts of anhydrous zinc chloride were effective catalysts in the acylation of furan. While catalytic amounts of zinc chloride were ineffective, furan and 2-methylfuran were found to add to chloral in the presence of molecular quantities of zinc chloride to form the very unstable 1-(2-furyl)-2,2,2-trichloroethanol (I) and 1-(5-methylfuryl-2)-2,2,2-trichloroethanol (III). As was observed by Peters and Fischer[6] in studies on alkyl furylcarbinols, these carbinols could not be esterified by many of the usual acylating agents, such as acetyl chloride, benzoyl chloride and benzoyl chloride in pyridine. Treatment of the carbinols with acetic anhydride in pyridine at room temperature resulted in the

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(1) Muller, U. S. Patent 2,329,074; *C. A.*, **38**, 1056 (1944).

(2) Metcalf and Gunther, THIS JOURNAL, **69**, 2579 (1947); Truitt, Mattison and Richardson, *ibid.*, **70**, 79 (1948); Feeman, Dove and Amstutz, *ibid.*, **70**, 3136 (1948).

(3) Elderfield, "Heterocyclic Compounds," Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1950, p. 142.

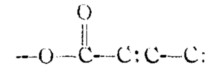
(4) Haskelburg and Lavie, THIS JOURNAL, **69**, 2267 (1947); Stephenson and Waters, *J. Chem. Soc.*, 333 (1946).

(5) Hartough and Kosak, THIS JOURNAL, **69**, 1012 (1947).

(6) Peters and Fischer, *ibid.*, **52**, 2079 (1930).

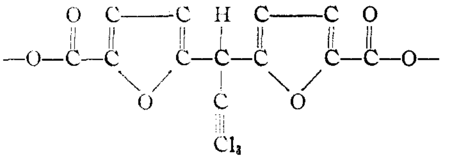
formation of the relatively stable acetates. 1-(2-Furyl)-2,2,2-trichloroethanol was also prepared by the base-catalyzed addition of chloroform to 2-furaldehyde according to the method of Howard.[7]

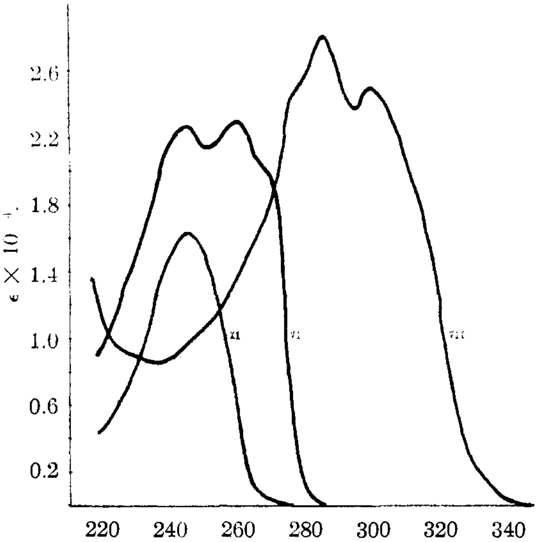
Because attempts to condense these carbinols with furan and 2-methylfuran resulted only in extensive decomposition of the carbinols, attention was shifted to the study of the condensation of derivatives of furoic acid with chloral. This latter investigation was also of interest since Lauger,[8] as a result of studies on naturally occurring-substances and related compounds, concluded that the grouping



$$-O-\overset{\overset{O}{\|}}{C}-C{:}C-C{:}$$

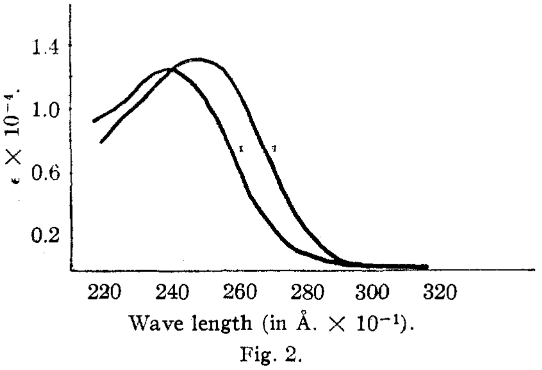
is very important in bestowing insecticidal properties on such compounds. Consideration of the molecule





Wave length (in Å. × 10^{-1}).

Fig. 1.



Wave length (in Å. × 10^{-1}).

Fig. 2.

indicates the presence of two of these "diene-ester" groupings.

Condensation of methyl 2-furoate with chloral in the presence of concentrated sulfuric acid gave the desired DDT analog, 1,1-bis-(5-carbomethoxyfuryl-2)-2,2,2-trichloroethane (VI). Use of milder condensing agents such as zinc chloride, phosphoric acid, chlorosulfonic acid in carbon tetrachloride, etc., resulted in no reaction. Although this compound was stable at the melting point and gave no precipitate with ethanolic silver nitrate, treatment with ethereal piperidine resulted in immediate separation of the theoretical amount of piperidine hydrochloride and formation of 1,1-bis-(5-carbomethoxyfuryl-2)-2,2-dichloroethene (VII). Similar treatment of DDT caused separation of less than 10% of the theoretical amount of piperidine hydrochloride after 48 hours. Attempts to oxidize either VI or VII using chromium trioxide in glacial acetic acid resulted only in extensive decomposition.

As expected, the action of ethanolic potassium hydroxide on VI caused dehydrochlorination and hydrolysis to give 1,1-bis-(5-carboxyfuryl-2)-2,2-dichloroethene (VIII). However, hydrolysis in a mixture of concentrated hydrochloric acid and dioxane also resulted in dehydrochlorination to produce 1-(5-carbomethoxyfuryl-2)-1-(5-carboxyfuryl-2)-2,2-dichloroethene (IX).

Repeated attempts to condense 2-furanilide with chloral have been unsuccessful; however, reaction of N,N-diethyl-2-furamide (X) with chloral by a modification of the procedure of Burger[9] produced 1-(5-N,N-diethylfuramido-2)-2,2,2-trichloroethanol (V). Attempts to condense this carbinol with methyl 2-furoate or N,N-diethyl-2-furamide have been unsuccessful.

The ultraviolet absorption spectra of methyl 2-furoate (XI), 1,1-bis-(5-carbomethoxyfuryl-2)-2,2,2-trichloroethane (VI), 1,1,-bis-(5-carbomethoxyfuryl-2)-2,2-dichloroethene (VII), N,N-diethyl-2-furamide (X) and 1-(5-N,N-diethylfuramido-2)-2,2,2-trichloroethanol (V) have been measured using a Beckman model DU photoelectric quartz spectrophotometer. Results of these measurements are presented in Figs. 1 and 2.[10]

Experimental

1-(2-Furyl)-2,2,2-trichloroethanol (I) **Method A.**—To a well-stirred mixture of 29.5 g. (0.2 mole) of chloral and 273 g. (0.4 mole) of furan in 100 ml. of glacial acetic acid was added 27.4 g. (0.2 mole) of freshly fused zinc chloride. Stirring was continued for 1.5 hours without external cooling, an equal volume of water added to the mixture and the mixture steam distilled. The steam distillate was neutralized by the addition of solid potassium bicarbonate and extracted with ether. The combined extracts were dried over magnesium sulfate and distilled under reduced pressure to yield 18.3 g. (0.088 mole, 44%) of pale yellow oil distilling between 114–122° under 11–12 mm. Redistillation gave a very pale yellow oil, b.p. 118.5–120° (12 mm.), 123–124° (15 mm.); $n^{25}D$ 1.5289, $n^{30}D$ 1.5274; d^{25}_4 1.510; molar refractivity, calcd. 44.52, found 44.77. This oil solidified on cooling to a white solid which melted at 33–35°.

Anal. Calcd. for $C_6H_5O_2Cl_3$: C, 33.35; H, 2.34. Found: C, 33.49; H, 2.74.

Method B.—According to the procedure of Howard,[7] 8.0 g. of solid potassium hydroxide was added in portions to a

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[7] Howard, THIS JOURNAL, **47**, 455 (1925); see also Bergmann, Ginsberg and Lavie, *ibid.*, **72**, 5012 (1950).

[8] Lauger, Martin and Muller, *Helv. Chim. Acta*, **27**, 892 (1944).

[9] Burger, Graef and Bailey, THIS JOURNAL, **68**, 1725 (1946).

[10] For ultraviolet spectra of other furan compounds see Raffauf, *ibid.*, **72**, 753 (1950).

vigorously stirred mixture of 66 g. (0.69 mole) of 2-furaldehyde and 120 g. (1.0 mole) of dry chloroform, 30 minutes being required to complete the addition. Stirring was continued for 1.5 hours. Two hundred ml. of ether was added to the mixture, the suspended solid removed by filtration and the filtrate concentrated by flash distillation of the ether–chloroform mixture. After removal of 2-furaldehyde, 12.5 g. of yellow oil was collected boiling at 112–121° under 14 mm.; $n^{25}\text{D}$ 1.5292.

1-(2-Furyl)-2,2,2-trichloroethyl Acetate (II).—The procedure described above (Method A) was repeated twice using 0.2 mole of chloral, 0.4 mole of furan and 0.2 mole of zinc chloride in each preparation.[11] To the combined crude products was added a mixture of 100 g. of acetic anhydride and 100 g. of pyridine. After 42 hours at room temperature, the mixture was poured into 400 ml. of cold water and the solution neutralized with solid sodium bicarbonate. The aqueous mixture was extracted with four 50-ml. portions of chloroform and the combined extracts subjected to distillation. Forty-three grams (42% based on furan used) of crude II was obtained. Redistillation gave a product which distilled at 135–137° (23 mm.); $n^{25}\text{D}$ 1.4984; d^{26}_4 1.407; molar refractivity, calcd. 53.91, found 53.72.

Anal. Calcd. for $C_8H_7O_3Cl_3$: C, 37.31; H, 2.74. Found: C, 37.56; H, 2.71.

1-(5-Methylfuryl-2)-2,2,2-trichloroethanol (III).—The procedure described for I resulted in extensive decomposition and very low yields when applied to 2-methylfuran. Thus the following procedure was developed.

To a mixture of 29.5 g. (0.2 mole) of chloral and 32.6 g. (0.4 mole) of 2-methylfuran in 100 ml. of dry ether was added a suspension of 41.1 g. (0.3 mole) of anhydrous zinc chloride in 100 ml. of dry ether, 30 minutes being required to complete the addition. The mixture was heated at the reflux temperature for 80 minutes, and allowed to stand at room temperature for 3.5 hours. The cool mixture was diluted with 100 ml. of ether and the solution was washed successively with saturated aqueous sodium chloride solution, 2.5% aqueous sodium carbonate and water, dried over magnesium sulfate and distilled through a 10-inch Vigreux column. The yellow oil, weighing 22.8 g., distilled at 126–132° (14–15 mm.).

This product was redistilled under an atmosphere of nitrogen gas in apparatus which had been carefully washed to remove traces of acid. It decomposed very rapidly even at room temperature and no analysis was possible. The following properties were observed for our best sample: b.p. 124–126° (7 mm.), 134–137° (15 mm.); $n^{25}\text{D}$ 1.5269; d^{26}_4 1.421; molar refractivity, calcd. 49.07, found 49.63.

Use of chloroform as solvent gave yields quite comparable with those when ether was employed.

1-(5-Methylfuryl-2)-2,2,2-trichloroethyl Acetate (IV).—The procedure described above for the preparation of III was repeated and to the crude product was added a mixture of 50 g. of pyridine and 50 g. of acetic anhydride. After 44 hours at room temperature, the reaction mixture was treated as described for II. Fourteen grams (26% based on methylfuran) of IV was obtained. Redistillation gave a sample having the following properties: b.p. 137–139° (15 mm.), 147–151° (23 mm.); $n^{25}\text{D}$ 1.5010; d^{26}_4 1.351; molar refractivity, calcd. 58.53, found 59.16.

Anal. Calcd. for $C_9H_9O_3Cl_3$: C, 39.81; H, 3.64. Found: C, 40.01; H, 3.80.

N,N-Diethyl-2-furamide (X).—A solution of 48 g. (0.66 mole) of diethylamine in 200 ml. of dry ether was added to a solution of 40 g. (0.325 mole) of 2-furoyl chloride in 200 ml. of dry ether. Distillation of the solution after removal of the solid by filtration gave 47 g. (0.28 mole, 87%) of X which distilled at 243° (743 mm.); $d^{25.5}_4$ 1.068; $n^{25}\text{D}$ 1.5048.

Anal. Calcd. for $C_9H_{13}O_2N$: C, 64.65; H, 7.835. Found: C, 64.60; H, 7.828.

1-(5-N,N-Diethylfuramido-2)-2,2,2-trichloroethanol (V).—By a modification of the procedure of Burger, *et al.*,[9] a mixture of 20 g. (0.12 mole) of X and 20 g. (0.135 mole) of chloral was added over a period of 1.0 hour, to 300 ml. of thoroughly chilled (10°) concentrated sulfuric acid. The red mixture was stirred for 2.0 hours, set aside at room temperature for 4.0 days, and was poured onto 400 g. of crushed ice. The sticky mass was extracted with five 100-ml. volumes of carbon tetrachloride and the combined extracts dried over magnesium sulfate. After separation of the drying agent, the solution was concentrated to one-half and petroleum ether (Barnsdall N-6) was added until the solution remained cloudy after vigorous swirling. The mixture was chilled for three days. A total of 14.5 g. (0.046 mole, 38.3% based on X) of pale yellow solid melting at 91–93.5° was obtained. Recrystallization from a mixture of 3 parts (by volume) of carbon tetrachloride and 1 part of petroleum ether increased the melting point to 95–96°.[12]

Anal. Calcd. for $C_{11}H_{14}O_3NCl_3$: C, 42.00; H, 4.485. Found: C, 42.07; H, 4.502.

1,1-Bis-(5-carbomethoxyfuryl-2)-2,2,2-trichloroethane (VI).—According to the procedure of Dinelli and Marini[13] for the condensation of methyl 2-furoate with formaldehyde, 100 ml. of concentrated sulfuric acid was added over a period of 15 minutes to a mixture of 50.5 g. (0.4 mole) of methyl 2-furoate and 33.5 g. (0.2 mole) of chloral hydrate while the mixture was stirred vigorously. The temperature increased rapidly to 84° where it remained only momentarily before beginning to decrease. Thirty minutes after addition of acid had been completed, the entire mixture was poured into 400 g. of crushed ice. The water–acid mixture was decanted and the heavy mass was leached repeatedly with boiling water. When the water extracts were no longer acidic, the insoluble residue was dissolved in 400 ml. of purified benzene. The benzene–water mixture was distilled and the water-free residue was dissolved in a minimum amount of hot ethanol. The solution was treated with decolorizing charcoal until the solution was light yellow in color. A total of 52 g. (0.136 mole, 68%) of crude VI, melting point 97–101°, was obtained. Recrystallization from ethanol increased the melting point to 103.5–104°. Titration of an ethanolic solution of VI with 0.01 *N* sodium hydroxide solution gave a neutral equivalent of 384 (VI requires 381.6).

Anal. Calcd. for $C_{14}H_{11}O_6Cl_3$: C, 44.06; H, 2.90. Found: C, 43.98; H, 2.78.

1,1-Bis-(5-carbomethoxyfuryl-2)-2,2-dichloroethene (VII).—To a solution of 3.82 g. (0.01 mole) of VI in 150 ml. of anhydrous ethyl ether was added a solution of 0.85 g. (0.01 mole) of piperidine in 50 ml. of anhydrous ether. A solid separated immediately after mixing the solutions. Filtration gave 1.15 g. (0.0095 mole) of piperidine hydrochloride. Removal of the ether from the filtrate left 3.6 g. of crude VII, melting at 141–142°. Recrystallization from dioxane increased the melting point to 144.5–145.5°.

Anal. Calcd. for $C_{14}H_{10}O_6Cl_2$: C, 48.72; H, 2.92. Found: C, 48.48; H, 2.99.

1,1-Bis-(5-carboxyfuryl-2)-2,2-dichloroethene (VIII).—Ten grams of VI was added to a solution of 10 g. of potassium hydroxide in 100 ml. of absolute ethanol and the solution was heated under reflux for 1.5 hours. The hot mixture was poured into 400 ml. of cold water and the cold basic solution extracted with ether to remove unreacted ester. Acidification of the water solution with dilute sulfuric acid gave 5.5 g. (65%) of VIII, melting at 227–231° (decomposition). Recrystallization from dioxane increased the melting point to 239–244°. Titration of an ethanolic solution of VIII with 0.01 *N* sodium hydroxide solution gave a neutral equivalent of 170 (VIII requires 158.6).

Anal. Calcd. for $C_{12}H_6O_6Cl_2$: C, 45.45; H, 1.91. Found: C, 45.25; H, 2.05.

1-(5-Carbomethoxyfuryl-2)-1-(5-carboxyfuryl-2)-2,2-dichloroethene (IX).—Ten grams of VI was dissolved in 100 ml. of dioxane and to this solution was added 10 ml. of water and 10 ml. of concentrated hydrochloric acid. The mixture was heated under reflux for 3.0 hours. The dioxane was removed by distillation, the residue dissolved in ethanol, and the solution treated with activated charcoal. The decolorized solution was poured into excess sodium carbonate solution. The basic solution was extracted with ether to remove unhydrolyzed ester. Removal of the ether from the dried extracts gave 5.3 g. of VI (identified by mixture melting point with starting material).

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[11] Attempts to employ double these quantities resulted in overheating and extensive decomposition of the mixture.

[12] This ratio of solvents seemed critical. It was necessary to avoid warming the solutions, thus the solid was dissolved in the minimum amount of solvent at room temperature and crystallization induced by chilling the solution.

[13] Dinelli and Marini, *Gazz. chim. ital.*, **67**, 312 (1937); *C. A.*, **32**, 154 (1938).

The basic solution after extraction was acidified with hydrochloric acid and again extracted with ether. After drying over magnesium sulfate, the ether was removed from the extracts to give 3.4 g. (84% based on reacted VI) of IX, melting at 205–207°. Recrystallization from dioxane increased the melting point to 255–258°. Mixture with VIII depressed the melting point to 224–231°. Titration of an ethanolic solution of IX with 0.01 *N* sodium hydroxide solution gave a neutral equivalent of 329 (IX requires 331.1).

Anal. Calcd. for $C_{13}H_8O_6Cl_2$: C, 47.15; H, 2.44. Found: C, 47.08; H, 2.51.

Absorption Spectra.—Absorption spectra were measured using freshly prepared 1 × 10^{-4} molar solutions. Solvent for compounds VI, VII and XI was Eastman spectro grade 2,2,4-trimethylpentane; that for compounds V and X was heptane purified according to the procedure of Weissberger.[14]

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[14] Weissberger, "Physical Methods of Organic Chemistry," Vol. II, Interscience Publishers, Inc., New York, N. Y., 1946, p. 766.

LINCOLN, NEBRASKA RECEIVED APRIL 26, 1951

[CONTRIBUTION FROM THE VIRUS LABORATORY, UNIVERSITY OF CALIFORNIA]

Ultracentrifuge Studies on Tobacco Mosaic Virus

BY H. K. SCHACHMAN

In view of the recent progress in the theoretical and technical aspects of ultracentrifugation it seemed of interest to re-examine solutions of purified tobacco mosaic virus in order to obtain information about the uniformity of the virus particles. Experiments are described which demonstrate the existence of boundary sharpening for solutions containing rod-like particles. The importance of this effect in the analysis of the shape of boundaries is discussed in relation to the determination of homogeneity. Evidence from boundary spreading experiments is presented to show that the virus preparations examined in this work are more homogeneous than those examined earlier. The existence of an anomaly in the centrifugation of mixtures is described, and its use in the detection of small amounts of trailing components is demonstrated. The virus preparations used in the present study are shown to contain not more than about 1% of monodisperse $^3/_4$ length particles. The results of this work are discussed in relation to the recent electron microscope studies of Williams and co-workers which indicate that the virus particles in solution are uniform with respect to length.

Introduction

The uniformity of tobacco mosaic virus particles has been the subject of much study and controversy. In addition, considerable attention has been directed toward the establishment of the identity of virus activity with the rod-like particles isolated from virus diseased tobacco plants. To some extent, experimental data relating to these problems have been conflicting; and, further, even the same data have been variously interpreted by different investigators. Some investigators have suggested that the virus particles have different lengths, varying from about 35 to 1000 mμ, any one of which or most of which possess biological activity.[1,2,3] Other research workers contend that there is a minimum length particle of about 300 mμ which possesses biological activity.[4–8]

The first point of view is based primarily upon the observation that particles of widely varying length are seen in electron micrographs of presumably purified virus. In addition, different results have been obtained from time to time by different investigators and even by the same workers when different virus preparations are studied in the ultracentrifuge, the diffusion cell, viscometers of different types, and in apparatus used for measurements of double refraction of flow.[3,9] Most of these inconsistent physical chemical data were obtained in the period of years immediately following the isolation and crystallization of tobacco mosaic virus by Stanley.[10]

On the other hand, more recent studies on different methods of isolation accompanied by viscosity and sedimentation analyses of the purified virus have yielded consistent and reproducible results suggesting uniformity of the virus particles with respect to length. The problem of aggregation has been studied by viscosity techniques. Although there has been little progress in developing a theoretical explanation for this characteristic end-to-end aggregation, empirical methods have been found which appear to reduce substantially the tendency of the virus particles to aggregate.[11,12]

Despite the progress in virus research there still appears to be a discrepancy between the sharp boundaries obtained in the ultracentrifuge for purified virus and the rather broad distribution of particle lengths as observed in the electron microscope. In view of the development of experimental techniques for the purification and study of the virus as well as the increase in knowledge with regard to the theoretical interpretation of ultracentrifuge and other physical chemical data, re-examination of this problem seemed advisable. This paper presents the results of an attempt to obtain more quantitative information about the uniformity of the rod-like particles and the results are discussed in the light of the electron microscope studies of Williams and co-workers.[13,14]

Materials and Methods

The tobacco mosaic virus preparations used in this study were obtained from juice extracted from diseased Turkish

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[4] W. M. Stanley, *Handbuch der Virusforschung*, **1**, 477 (1938).

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[9] M. A. Lauffer, W. C. Price and A. W. Petre, *Adv. in Enzymology*, **9**, 171 (1949).

[10] W. M. Stanley, *Science*, **81**, 644 (1935).

[11] H. K. Schachman, THIS JOURNAL, **69**, 1841 (1947).

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[13] R. C. Williams and R. L. Steere, THIS JOURNAL, **73**, 2057 (1951).

[14] R. C. Williams, R. C. Backus and R. L. Steere, *ibid.*, **73**, 2062 (1951).