

limited to the determination of small amounts of fluoride. A detailed study has not been made of the application of this technique to larger amounts using a much higher concentration of ferric ion in the reagent. The preliminary titrations indicate that such an application could be made, but the range we have chosen to study should be satisfactory for most of the fluoride determinations necessary.

3. *Accuracy*.—Although, as pointed out above, the accuracy is very good for the small amounts of fluoride determined, it should also be mentioned that the method would not be satisfactory for the accurate determination of fluoride in a substance whose fluoride content is greater than 10%, because in that case a 1% error would be appreciable, whereas for very small amounts of fluoride a 1% error is not important.

4. *Liquid junction*.—It is important to keep the total impurity molarity of the unknown fluoride solution below 0.01 *M*, else an appreciable liquid junction potential will cause fictitious results. In practice this is very easily done, by distilling the sample from impurities if in no other way. The distillate is much lower than 0.01 *M* in foreign ions.

It should be pointed out that the size of sample of an unknown should be adjusted so that the amount of fluoride in the final concentration cell is between 0.1 and 30 mg. per liter, preferably greater than 10 mg. per liter, since the percentage error is smaller for larger amounts of fluoride.

The procedure we have described corresponds

to the "Standard Solution" method for chloride used by Furman and Low.³ An attempt was made to apply the "small excess" method³ to the determination of fluoride, but with no success. The e. m. f. of the cell, with fluoride in both sides, seemed to be dependent upon the difference in fluoride concentration between the two sides rather than the ratio of the two concentrations. Because this "small excess" method offered no promising results, we abandoned a further study of it in favor of the procedure we have given.

The material presented in this paper gives indication that the technique described should be applicable to the determination of a number of other substances, such as aluminum, phosphate, sulfate, citrate or tartrate, by slight modifications of the procedure. It is hoped that further study will confirm this prediction.

Summary

A technique has been described for the determination of fluoride rapidly and accurately by means of a simple concentration cell. Interference of other ions can be eliminated entirely, since the procedure is applicable to the determination of fluoride in the distillate obtained in separating fluoride from all impurities. The range over which the method has been applied is 0.2 to 60 mg. per liter in a 5-cc. sample which is taken for analysis. Results are given showing the method's applicability to water analysis and to the determination of fluoride in phosphate rock.

AMHERST, MASS.

RECEIVED JUNE 13, 1939

NOTES

Dibenzyl Sebacate

BY R. E. BURNETT AND J. J. RUSSELL

The new compound dibenzyl sebacate was synthesized recently in this Laboratory for use in another investigation.¹ Sebacic acid was esterified with benzyl alcohol, the crude ester then distilled at 0.5 mm. pressure, and the middle fraction recrystallized several times from its melt. The following are properties of the

(1) Verhoek and Marshall, unpublished results.

pure dibenzyl sebacate.

Temp., °C.	Refractive index n_D (Abbe)	Density d_4^4
30.0	1.5171	1.055(6)
35.0	1.5152	1.051(9)
40.0	1.5133	1.048(1)

Colorless plates, practically odorless; melting point (from cooling curve), 28.3°; boiling point (in Claisen flask), 257° (uncorr.) at 4 mm.

RESEARCH LABORATORY
GENERAL ELECTRIC CO.
SCHENECTADY, N. Y.

RECEIVED FEBRUARY 21, 1939

On the Conversion of *l*-Abietic Acid into a *d*-Abietic Acid

BY TORSTEN HASSELSTROM AND JOHN D. MCPHERSON

The addition of hydrogen chloride to abietic acid produces dichlorodihydroabietic acid which is crystalline and easily purified.¹ We have found that dichlorodihydroabietic acid may be obtained directly from rosin and in apparently equal purity as when Steele's abietic acid² is used as a starting material. On splitting off of hydrogen chloride from dichlorodihydroabietic acid with sodium ethoxide, we have been able to obtain an abietic acid with the positive sign of rotation. This seemingly new abietic acid was characterized through its di-*n*-amylamine salt. According to Levy,¹ the dichlorodihydroabietic acid produces an abietic acid with the negative sign of rotation when heated at its melting point, and when treated with quinoline.

Experimental

Dichlorodihydroabietic Acid.—This compound was prepared substantially according to the directions given by Levy.¹ Ninety-five grams of "X" grade gum rosin was dissolved in 70 cc. of glacial acetic acid and boiled for four hours. The solution was then cooled to about 0°. The semi-solid mixture was saturated with dry hydrogen chloride, the bottle sealed and the mixture left standing at room temperature for two weeks. It was then filtered and the solid collected; yield 26 g. It was recrystallized from acetone and then from ethyl acetate, m. p. 190.5° dec. (corr.); (α)_D -10° (in ethanol).

Anal. Calcd. for C₂₀H₃₂O₂Cl₂: C, 63.97; H, 8.59. Found: C, 63.76; H, 8.60.

The dichlorodihydroabietic acid prepared from Steele's abietic acid,² m. p. 163.5–165° (corr.); (α)_D -76° (in ethanol) showed the following constants: m. p. 190.5° dec. (corr.); (α)_D -8.1 (in ethanol). Found: C, 63.96; H, 9.16.

***d*-Abietic Acid.**—Ten grams of dichlorodihydroabietic acid (m. p. 190.5° dec. (corr.); (α)_D -10°), was added in portions over a period of five to ten minutes to a boiling solution of sodium ethoxide (17.5 g. of sodium and 175 cc. of absolute ethanol). The mixture was refluxed for one hour. Water was then added and the excess of alcohol was distilled off. As much of the liquid as possible was poured off the remaining solid. The soapy residue was then dissolved in 3 liters of water. The solution was filtered and the filtrate acidified with dilute hydrochloric acid. The white precipitate was filtered off and crystallized from methanol, yield 3.5 g. (40%), m. p. 137–139°; (α)_D +27° (in ethanol), which on successive crystallization yielded an abietic acid melting at 142–143° (corr.); (α)_D +20° (in ethanol). Calcd. for C₂₀H₃₀O₂: C, 79.40; H, 10.05. Found: C, 79.33; H, 10.08; C, 79.57; H, 10.32.

(1) Røy and Simonsen, *Indian Forest Records*, **11**, 211 (1924); Levy, *Ber.*, **64**, 2441 (1931).

(2) Steele, *This Journal*, **44**, 1333 (1922).

The mother liquor from this separation yielded small quantities of an acid melting at 207–212° (corr.), probably identical with the dihydroabietic acid of melting point 217.5–218.5° (corr.), previously described by us.³

A solution of 6 g. of *d*-abietic acid of m. p. 139–143° (corr.) was treated with 4 g. of di-*n*-amylamine (Eastman Kodak Company) in acetone. The solution was boiled for about ten minutes and filtered. On standing 10.6 g. of crude di-*n*-amylamine salt was obtained, m. p. 117.5–119° (corr.). The compound was recrystallized four times, the melting point remaining constant after the third crystallization, being 119–119.5° (corr.), (α)_D +3.3° (in ethanol). The amounts of recovered di-*n*-amylamine salt were too small to be converted into the original acid.

Anal. Calcd. for C₃₀H₅₀O₂N: C, 78.35; H, 11.62. Found: C, 78.61; H, 11.76.

The di-*n*-amylamine salt of *l*-abietic acid shows the following constants: m. p. 141–142°; (α)_D -74.5°.⁴

This investigation is being continued.

(3) Hasselstrom and McPherson, *ibid.*, **61**, 1228 (1939).

(4) Palkin and Harris, *ibid.*, **56**, 1935 (1934).

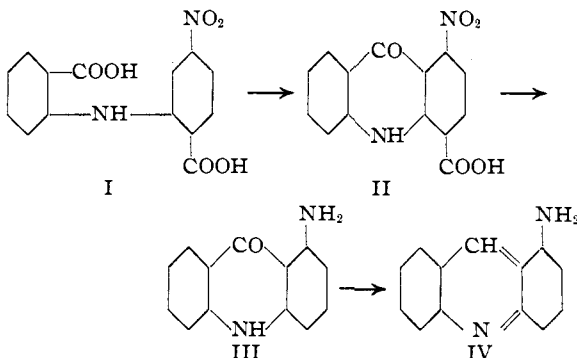
G & A LABORATORIES, INC.
SAVANNAH, GEORGIA

RECEIVED MAY 29, 1939

The Identification of 1-Nitroacridone-4-carboxylic Acid as 1-Aminoacridine

BY KONOMU MATSUMURA

In a previous paper,¹ it was stated that the nitroacridone-carboxylic acid (m. p. 333° dec.) which was obtained from 5-nitrodiphenylamine-2,2'-dicarboxylic acid (I) by ring closure should have the constitution of 1-nitroacridone-4-carboxylic acid (II) in consideration of the observations that the fluorescence and solubility of the aminoacridone (III), which was converted from the nitroacridonecarboxylic acid in question, exactly coincided with those of 1-aminoacridone but were widely different from those of the 3-isomer.



The present paper indicates the previous interpretation to be altogether correct, but not in harmony with the Lehmstedt conclusion,² on

(1) Matsumura, *This Journal*, **60**, 591 (1938).

(2) Lehmstedt and Schlader, *Ber.*, **70**, 1526 (1937); Lehmstedt, *ibid.*, **71**, 1609 (1938).

TABLE I
COMPARISON OF THE PROPERTIES OF SUBSTITUTED ACRIDINES

Substituent	1-Amino-	3-Amino-
Fluorescence in alc. soln.	None	Intense green
M. p., °C.	{ 178 (uncorr.) 182.3 (corr.) ^a	{ 218 (uncorr.) ^b 223.5 (corr.)
Hydrochloride {	Cryst. color	Blue-black
	Aq. soln.	Violet-red color with no fluores.
	M. p., °C. (uncorr.)	285 (dec.) ^c
		Orange-red
		Orange-yellow color with green fluores.
		328-329 (dec.)

^a Albert and Linnell [*J. Chem. Soc.*, 22 (1938)] give m. p. 181° (corr.), and Lehmsstedt [*Ber.*, 71, 808 (1938)] gives m. p. 165-170°. ^b Albert and Linnell [*J. Chem. Soc.*, 1618 (1936)] give m. p. 219° (corr.) and Scherlin [*Ann.*, 516, 218 (1935)] m. p. 221-222° for their products, respectively. ^c Lehmsstedt (*loc. cit.*) gives m. p. 286° (dec.) for his product.

the basis of further investigations as follows. The aminoacridine (IV) which was obtained from the aminoacridone in question (III), on admixture with synthesized 3-aminoacridine, showed a great depression of the melting point, while with synthetic 1-aminoacridine it showed no depression; moreover, its other properties were in good agreement with those of 1-aminoacridine shown in Table I.

The nitroacridone-carboxylic acid must, therefore, be 1-nitroacridone-4-carboxylic acid.

Experimental

1-Aminoacridine.—Four-tenths gram of the aminoacridone in question (m. p. 289-290°) and 5 g. of sodium amalgam (5%) in 30 cc. of dilute alcohol (33%) were heated on a water-bath for three hours with frequent shaking. The solution was decanted from mercury, acidified with hydrochloric acid and oxidized with 2 g. of ferric chloride. The base (yield, 0.25 g.) which was set free from the hydrochloride, crystallized from alcohol (33%) into gold-yellow prismatic needles, melting at 178° alone or on admixture with a sample of 1-aminoacridine (m. p. 178°), whereas a mixture (7:3) of this compound with an authentic specimen of 3-aminoacridine (m. p. 218°) melted at 135-140°.

Anal. Calcd. for C₁₃H₁₀N₂: C, 80.41; H, 5.15. Found: C, 80.22; H, 5.28.

The hydrochloride gives blue-black prismatic needles from dilute hydrochloric acid (5%), m. p. 285° (dec.). The aqueous solution assumes a violet-red color, while an alcoholic solution has a violet-blue color, both showing no fluorescence in any dilution.

CHEMICAL LABORATORY OF
KITASATO INSTITUTE
TOKYO, JAPAN

RECEIVED MAY 13, 1939

Synthesis of Aldehydes by Stephen's Method¹

BY JONATHAN W. WILLIAMS

The application of Stephen's² procedure to the preparation of several aldehydes has led to the conclusion that the method is not as general as

(1) Presented before the Division of Organic Chemistry at the Baltimore meeting of the American Chemical Society, April, 1939.

(2) Stephen, *J. Chem. Soc.*, 127, 1874 (1925).

claimed. Stephen states, "the method is applicable to aliphatic and aromatic nitriles, and the yields are usually almost quantitative." Exceptions are made by Stephen only in the cases of *o*-tolualdehyde and α -naphthaldehyde, where "steric hindrance" is offered as an explanation for poor yields.

The method has been tried in this Laboratory with good results in the preparation of benzaldehyde, *p*-tolualdehyde and β -naphthaldehyde, and with only fair results in the preparation of phenylacetaldehyde and isocaproaldehyde. Stephen's poor yields with α -naphthaldehyde and *o*-tolualdehyde have been verified, and the method has been found completely unsatisfactory for the preparation of β -hydroxypropionaldehyde.

Experimental

Preparation of Phenylacetaldehyde.—Eleven runs were made, each different, and the following was found to give the highest yield.

A mixture of anhydrous stannous chloride (57 g., 0.3 mole, prepared by heating the hydrated salt for one hour in a 200° bath, cooling and pulverizing the resulting solid mass) and dry ether (400 ml., twice distilled from sodium) was placed in a 2-liter three-necked round-bottomed flask, provided with a mechanical stirrer and a reflux condenser fitted with a calcium chloride drying tube. The mixture was saturated with dry hydrogen chloride,³ using slow stirring to agitate the stannous chloride. Within two hours all of the solid stannous chloride had dissolved, forming a separate lower viscous layer.

The source of hydrogen chloride was then disconnected and phenylacetonitrile (16 g., 0.136 mole, freshly distilled) was added rapidly by means of a dropping funnel. The mixture was stirred vigorously for one hour and then allowed to stand for eighteen hours. The aldimine hydrochloride-stannic chloride complex began to separate after five hours.

The ethereal hydrogen chloride was removed by decantation and the residue washed twice with 100-ml. portions of dry ether to remove hydrogen chloride. To the residue were added 800 ml. of water and sufficient solid sodium bicarbonate to render the solution neutral to congo red.

(3) "Organic Syntheses," Coll. Vol. I, p. 287 (1932).

The mixture was then brought to boiling and live steam passed through it until the total volume of distillate was 250 ml.

The steam distillate was extracted with two 75-ml. portions of ether and the ethereal extract dried over sodium sulfate. The ether was removed on the steam-bath and the residue vacuum-distilled: yield of phenylacetaldehyde, b. p. 87–88° (18 mm.), 5.4 g., 0.045 mole, 33%.

The other aldehydes studied were prepared by much the same procedure. The results are summarized in the table.

Aldehyde prepared	Time allowed for pptn. of addn. complex	Yield, %
Benzaldehyde ⁴	5 hours	97
β -Naphthaldehyde ⁵	12 hours	91
<i>p</i> -Tolualdehyde ⁴	12 hours	77
Phenylacetaldehyde ⁴	18 hours	33
Isocaproaldehyde	7 days	31
<i>o</i> -Tolualdehyde ^{7,8}	8 days	9
α -Naphthaldehyde ⁷	7 days	7
β -Hydroxypropionaldehyde ⁶	7 days	0

(4) Reported by Stephen² in almost quantitative yields.

(5) One liter of hot water was added to the residue and live steam passed through the mixture as long as any solid came over in the steam distillate. The solid was recrystallized from 50% aqueous alcohol. Fulton and Robinson, *J. Chem. Soc.*, **152**, 200 (1939), reported the preparation of β -naphthaldehyde by the Stephen technique in 75% yield. Their work was published after the preparation had been completed at the University of Maryland.

(6) Removal of the aldehyde by steam distillation was not attempted; 500 ml. of water was added to the addition complex, the mixture warmed on the steam-bath for one hour, allowed to cool and extracted with ether. The product was a thick, tarry liquid which gave negative tests with Schiff and Tollens reagents.

(7) Reported by Stephen² in poor yield.

(8) There was no precipitate after seven days so one-half of the ether was removed and the reaction mixture allowed to stand another twenty-four hours.

CHEMISTRY LABORATORY
UNIVERSITY OF MARYLAND
COLLEGE PARK, MD.

RECEIVED APRIL 14, 1939

Crystalline Lead Orthophosphate

BY EDWARD J. ROEHL

In the preparation of crystalline metathetical reaction products where the solubility of the reaction product is very low, it is often difficult to obtain crystals large enough for microscopic examination, and a number of methods have been employed for maintaining the necessary slight supersaturation. The writer has successfully employed a cellophane membrane for a number of such cases.

By treating a solution of lead acetate with sodium hydrophosphate, Aiders and Stähler¹ succeeded in preparing lead orthophosphate, but without any signs of microscopically recognizable crystals. In fact, it appears from the literature that the only method of preparing this salt is the rather cumbersome one of Zambonini,² who fused

(1) Aiders and Stähler, *Ber.*, **42**, 2263 (1909).

(2) Zambonini, *Z. Kryst.*, **58**, 226 (1923).

and slowly cooled the white powder. In our experiments, a cellophane membrane was tied over the mouth of a test-tube (an open tube could of course be used) containing a 1% solution of Na_2HPO_4 and the tube inverted in a slightly acidified 0.2% solution of $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2) \cdot 3\text{H}_2\text{O}$. At once an almost amorphous precipitate of lead orthophosphate appeared on the under side of the membrane and in about a minute crystals of the white salt started to fall.

The crystals (some of which were 2 mm. in length) were uniaxial hexagonal prisms exhibiting strong birefringence and showing an optically negative character. The values of $\omega = 1.970$ and $\epsilon = 1.936$ check the data in the literature for the refractive indices of lead orthophosphate. Analysis of the crystals gave a value of 17.4% as compared with the calculated value of 17.5% for $\text{Pb}_3(\text{PO}_4)_2$.

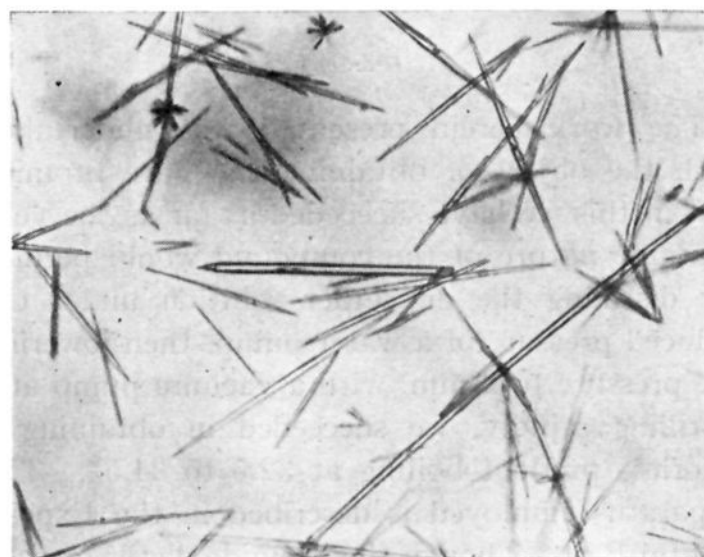


Fig. 1.

The accompanying photomicrograph at (50 X) shows some of the crystals obtained.

RESEARCH LABORATORY
INTERNATIONAL NICKEL CO.
BAYONNE, NEW JERSEY

RECEIVED APRIL 14, 1939

α -Furfuryl Bromide (2-Bromomethylfuran)

BY J. E. ZANETTI AND J. T. BASHOUR

In 1927 one of us¹ prepared an ether solution of α -furfuryl bromide by acting on furfuryl alcohol with phosphorus tribromide. The solution proved satisfactory in the synthesis of various furfuryl ethers but the pure bromide was not separated as its decomposition was rapid once the solvent was evaporated. Von Braun and Kohler² had

(1) Zanetti, *THIS JOURNAL*, **49**, 1065 (1927).

(2) Von Braun and Kohler, *Ber.*, **51B**, 86 (1918).

had a similar experience when they synthesized the bromide by acting on α -furfurylmethylethylamine with cyanogen bromide in ether solutions; they were unable to obtain the pure compound from the ether solution. Recently the ether solution of the bromide has been used by Paul³ in the synthesis of alkyl furans with the Grignard reagent and by Paul and Normant⁴ in a study of the preparation of phenyl- α -furfuryl ether and its rearrangement.

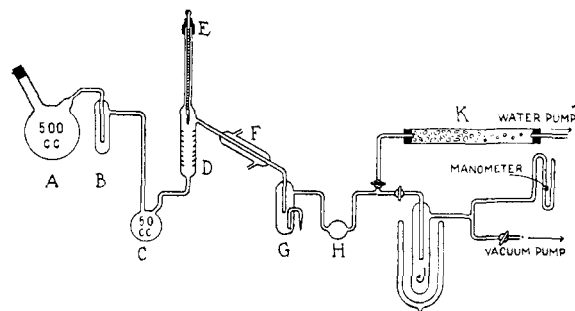


Fig. 1.

The work herein presented was undertaken with the object of obtaining the pure bromide and in this we have succeeded as far as the very unstable nature of the compound would permit. By distilling the dry ether solution under the reduced pressure of a water pump, then lowering the pressure to 2 mm. with a vacuum pump and distilling rapidly, we succeeded in obtaining a colorless product boiling at 32.5 to 34.5°. The apparatus employed is described in the Experimental Part. Though the bromide distils over as a colorless liquid, in a few minutes it begins to color yellow and then brown. Its rate of decomposition seems to be a function of the concentration of hydrobromic acid, which is one of the decomposition products. Hence it must never be kept long in a confined space as the rapidity of the decomposition reaches explosive violence. During a specific gravity determination a pycnometer containing 3 cc. of the bromide exploded a few minutes after being taken out of the balance case. The operator fortunately had moved far enough away so that he was not injured, but no trace of the pycnometer could be found except one of the caps closing the capillaries. Even when kept in loosely stoppered containers the decomposition is rapid the vessels becoming coated with tough graphitic films

(3) Paul, *Bull. soc. chim.*, [5] 2, 2227 (1935).

(4) Paul and Normant, *ibid.*, [5] 5, 1148 (1938).

similar to those described in the preparation of the α -furfuryl iodide.⁵

The bromide is slightly lachrymatory and a distinct lung irritant, the irritation not appearing for some hours after inhaling the vapor.

Density determinations gave d^{20}_{20} 1.560 and the index of refraction with the Abbe refractometer was n^{20} 1.5380. Chemically the compound was identified by condensing it with furfuryl alcohol to di- α -furfuryl ether, b. p. 100–102° at 2 mm.

Because of the short life and dangerous nature of this compound and the fact that the ether solution in which it is prepared is satisfactory for synthetic work, it did not appear of importance to carry this investigation any further.

Experimental

Preparation of the α -Furfuryl Bromide.—This was carried out as described by Zanetti¹ using smaller quantities (20 g. of furfuryl alcohol and 20 g. of phosphorus tribromide). The solutions stood from twenty to forty hours over granulated sodium hydroxide. The solutions were then rapidly filtered and distilled in the apparatus shown in Fig. 1.

Apparatus.—The apparatus consisted of a 500-cc. bulb (A) connected through a trap (B) with a smaller bulb (50 cc.) C. This was connected through a 5-cm. reflux column (D) with thermometer attachment (E) and a condenser (F) with another trap (G) provided with a siphon side-tube ending in a capillary. A small bulb (H) of 50-cc. capacity was inserted before trap (J) which was immersed in "dry-ice"–ethylene trichloride mixture and used to protect the vacuum pump. (K) was a drying tube to prevent water vapor from entering the apparatus. The entire apparatus was made of Pyrex glass. The only points where the compound might come in contact with organic material were at the side tube of (A) and at the thermometer holder (E) where a piece of rubber vacuum tube was used to hold the thermometer in place. The side-tube of (A) and the thermometer holder were made of 8-mm. tubing, the stoppered ends far removed from the warm vapors so that contamination from these sources was unappreciable.

Operation of the Apparatus.—The apparatus was first exhausted to 2 mm. and warmed to take out all moisture. Air was slowly let in through (J) till atmospheric pressure was reached. The filtered ether solution was then placed in (A) and the side-tube closed with a small cork thoroughly covered with collodion. The connection leading to the vacuum pump was closed and that leading to the water pump opened. When the water pump was started the boiling of the ether solution was rapid and the temperature dropped fast. In order to keep the ether boiling it was necessary to immerse (A) in water kept at about 30°. When no more ether came over, the vacuum pump was started and the pressure in the apparatus brought down to 2 mm. A small amount of ether col-

(5) Zanetti, *THIS JOURNAL*, 49, 1061 (1927).

lected in (J). Ice water-baths were then placed around (B) and (C) and a hot water-bath around (A). Distillation of the bromide began accompanied by darkening and film formation in (A). The distillate collected mostly in (B). When distillation was finished, the bath around (B) was heated almost to boiling and an ice water-bath placed around (G). When this distillation was over, the bath around (C) was raised to boiling and an ice-bath placed under (H). This was the final distillation, the colorless bromide distilling into (G) while the temperature remained at 32.5 to 34.5°. A very small amount of distillate passed into the bulb (H). When the distillation was complete, the apparatus was brought back to atmospheric pressure by slowly letting air through (J). The side-tube capillary was then broken and the liquid siphoned off into a small flask by gently sucking out the air from the flask.

The residues in (A), (B) and (C) were black and tarry but the distillate in (G) was colorless throughout the distillation, though shortly after it began to color. Five runs were made, each with a new apparatus as it was found easier to discard the one used than to attempt to remove the graphitic films formed in it from the bromide decomposition.

The yields were about 50% of the theoretical.

Properties of the Compound.—As mentioned, the compound is a colorless liquid which decomposes rapidly, turning yellow then brown and ending in a tarry mass which adheres tightly to the container. Attempts to keep the compound for more than a few hours proved futile. Moisture doubtless plays an important role in initiating the decomposition. Phosphorus pentoxide or anhydrous sodium sulfate, however, proved useless in stabilizing the compound. Solid potassium hydroxide, though unable to prevent blackening, delays total decomposition for a much longer time. This points to the possibility that the hydrobromic acid formed by the decomposition may act as an accelerator. The compound thus prepared and dissolved in ether remains undecomposed for days.

Identification.—Analyses gave for bromine 50.3 and 49.0%, calculated, 49.66%.

Four and one-half grams of the bromide was condensed with excess of furfuryl alcohol and potassium hydroxide and the product distilled at 2 mm. The di- α -furfuryl ether came over at 100–102°; yield 85%.

HAVEMEYER CHEMICAL LABORATORY
COLUMBIA UNIVERSITY
NEW YORK, N. Y.

RECEIVED MAY 5, 1939

COMMUNICATIONS TO THE EDITOR

HOMOGENEITY OF GONADOTROPIC HORMONE PREPARATIONS ISOLATED FROM PREGNANCY URINE

Sir:

We have reported the preparation from pregnancy urine of gonadotropic fractions containing 4000 minimal ovulating doses per milligram when assayed in the post-partum rabbit [*J. Biol. Chem.*, **128**, 525 (1939)]. Two such samples have recently been examined on the ultracentrifuge,¹ and appear to be homogeneous with respect to sedimentation. In each case, in the concentrations employed, a single sharply defined band was observed at approximately 250,000 times gravity.

In addition, electrochemical homogeneity of one of these preparations was demonstrated as a result of studies with the electrophoresis apparatus of Tiselius.² Evidence for the presence of only a single component (which previous analysis had shown to be a polypeptide-poly-

saccharide complex), was indicated by the appearance of a single migrating band having sharp boundaries in a phosphate buffer of ionic strength 0.1 (pH 7.0). The mobility was 4.85×10^{-5} cm.², sec.⁻¹, volt⁻¹.

Isoelectric point determinations, made by adsorbing the hormone upon collodion particles, and determining migration in a microelectrophoresis cell, showed an isoelectric point of pH 3.2–3.3.

Since such preparations appear to be identical with regard to biological activity and chemical composition, we feel that the evidence obtained with the ultracentrifuge and Tiselius apparatus demonstrates the homogeneity of our preparations.

Further detailed data bearing upon the physical and chemical properties of these and similar preparations of the hormone will be reported shortly.

DEPARTMENTS OF PHYSIOLOGICAL CHEMISTRY
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PHILADELPHIA, PA.

SAMUEL GURIN
CARL BACHMAN
D. WRIGHT WILSON

RECEIVED JULY 20, 1939

(1) Through the courtesy of Drs. A. E. Severinghaus and J. A. Chiles, Jr.

(2) Through the courtesy of Dr. Florence B. Seibert.