washed in dilute, aqueous sodium carbonate solution and recrystallized twice from cold 30% ethanol and twice from cold 95% ethanol to give a white crystalline product.

In this and in the following syntheses in which the preliminary purifications are described as recrystallizations from solvents diluted with water, the product was first dissolved in a minimum of hot, concentrated solvent and then precipitated by addition of water after the solution had cooled.

Anal. Calcd. for $C_{15}H_{20}O_2$: sapn. equiv., 232.2. Found: sapn. equiv., 232.2.

***p*-Cyclohexylphenyl *n*-butyrate** (m. p. 30–31°) was prepared in 78% yield by the reaction of 17.6 g. of *p*-cyclohexylphenol with 10.3 ml. of *n*-butyryl chloride at 100° for fifteen minutes. The crude product was washed like the propionate and recrystallized twice from slightly diluted propanol. The crystals were handled in chilled apparatus to prevent fusion. To dry the product, it was dissolved in chloroform and the solvent evaporated. The colorless, odorless oil that remained solidified to white crystals in the refrigerator.

Anal. Calcd. for $C_{16}H_{22}O_2$: sapn. equiv., 246.2. Found: sapn. equiv., 245.7.

***p*-Cyclohexylphenyl *n*-valerate** (m. p. 19–20°) was prepared in 84% yield as follows: 5 ml. of valeric acid and 5 ml. of thionyl chloride were warmed together, and 8 g. of *p*-cyclohexylphenol was added. The mixture was warmed for fifteen minutes, 5 ml. more of thionyl chloride was added and the heating continued another thirty minutes. The product was washed like the propionate, cooled to a semisolid state, and recrystallized once from 18% ethanol and twice from ligroin, the crystals being handled in chilled apparatus. The excess ligroin was evaporated, and the product was distilled at 185–189° at 9 mm. to give an oil that yielded waxy crystals when cooled.

Anal. Calcd. for $C_{17}H_{24}O_2$: sapn. equiv., 260.2. Found: sapn. equiv., 267.5.

***p*-Cyclohexylphenyl *p*-methoxybenzoate** (m. p. 97–98°) was obtained in 86% yield as follows: 10 g. of *p*-cyclohexylphenol and 14 ml. of *p*-methoxybenzoyl chloride were warmed together for an hour. The product was stirred into hot, dilute, aqueous sodium carbonate solution and cooled overnight. The white crystals of crude product were recrystallized from propanol and washed with cold ethanol.

Anal. Calcd. for $C_{20}H_{22}O_3$: sapn. equiv., 310.2. Found: sapn. equiv., 303.7.

***p*-Cyclohexylphenyl *o*-bromobenzoate** (m. p. 90.5–92°) was prepared in 48% yield as follows: 16 g. of *o*-bromobenzoic acid was kept at 100°, and 20 ml. of thionyl chloride was added in small portions. A clear melt was obtained, and to it was added 10 g. of *p*-cyclohexylphenol. The mixture was warmed for twenty minutes and washed like the *p*-methoxybenzoate. The white crystals of crude product were recrystallized once from 15% propanol and once from 95% ethanol.

Anal. Calcd. for $C_{19}H_{19}O_2Br$: sapn. equiv., 359.2. Found: sapn. equiv., 357.9.

***p*-Cyclohexyl *m*-bromobenzoate** (m. p. 89–90°) was obtained in 74% yield by the reaction of 10 g. of *p*-cyclohexylphenol with 15 ml. of *m*-bromobenzoyl bromide at 100° for thirty minutes. The product was washed like the *p*-methoxybenzoate and recrystallized from propanol to yield white crystals.

Anal. Calcd. for $C_{19}H_{19}O_2Br$: sapn. equiv., 359.2. Found: sapn. equiv., 350.9.

***p*-Cyclohexyl *p*-bromobenzoate** (m. p. 118–119°) was prepared in 20% yield as follows: To 18.5 g. of *p*-bromobenzoyl chloride that had evidently become partially hydrolyzed was added 20 ml. of thionyl chloride in small portions. The mixture was warmed for twenty minutes, 10 g. of *p*-cyclohexylphenol was added and the heating continued another twenty minutes. The product was washed like the *p*-methoxybenzoate, recrystallized once from 30% propanol and twice from pure propanol and then washed with cold 95% ethanol to give white crystals.

Anal. Calcd. for $C_{19}H_{19}O_2Br$: sapn. equiv., 359.2. Found: sapn. equiv., 350.2.

Saponification Procedure.—In the determination of the saponification equivalents, the esters were hydrolyzed under reflux in hot ethanol with excess standard aqueous sodium hydroxide solution. The excess alkali was then neutralized with standard acid, and the solution was back-titrated with alkali and phenolphthalein indicator, the final volume being controlled to give a maximum ethanol content of 25% and the temperature of the mixture being maintained below 5°. Under these conditions, satisfactory blanks and end-points were obtained in the presence of free *p*-cyclohexylphenol. These were not satisfactory with higher temperatures or greater ethanol concentrations or with propanol as solvent or when the saponification was carried out with potassium hydroxide in diethylene glycol.[2] Under those unsatisfactory conditions, the ionization of the phenol was probably encouraged.

After the titrations, each mixture contained a voluminous precipitate that was identified by its melting point in each case as *p*-cyclohexylphenol. This identification established that the hydrolyzed compound was the ester and not a product of a side reaction.

***p*-Cyclohexylphenyl acetate** had been previously reported,[3,4] but it had been purified only by vacuum distillation to give a product melting at 35°. This report was checked by the preparation of the ester from *p*-cyclohexylphenol and acetyl chloride with subsequent recrystallizations from ethanol. The white crystals obtained melted at 43–45°.

***p*-Cyclohexylphenyl benzoate** had been previously reported[5] as melting at 114–114.5°. To check this report *p*-cyclohexylphenol and benzoic anhydride were refluxed in anhydrous pyridine. The crude product was precipitated by addition of water to the pyridine and was recrystallized twice from propanol to give white crystals melting at 118.5°.

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(2) Ernst C. Redeman and Howard J. Lucas, *Ind. Eng. Chem., Anal. Ed.*, **9**, 521–522 (1937).

(3) J. V. Braun, E. Anton, W. Haensel and G. Werner, *Ann.*, **472**, 1–89 (1929).

(4) "Phenol, *p*-cyclohexyl-, acetate" is listed in *C. A.*, **33**, 10872 (1929); but the name should be *p*-cyclohexylphenoxyacetic acid. The mistake springs from an ambiguous listing of the unnamed compound VI Aa in the table by Martin E. McGreal, Victor Niederl and Joseph B. Niederl, THIS JOURNAL, **61**, 345 (1929). Their other unnamed aryloxyacetic acids—VII Aa, VIII Aa, IX Aa and X Aa—were similarly listed in the table and are also carried in *C. A.* as "acetates." Suitable names for these would be, respectively, *p*-cyclohexyl-*o*-methylphenoxyacetic acid, *p*-(3-methylcyclohexyl)-phenoxyacetic acid, *p*-(4-methylcyclohexyl)-phenoxyacetic acid and *p*-cyclopentylphenoxyacetic acid. Their unnamed compound VIIa is doubly ambiguous in that it is shown in the text both as a true "diacetate" and as an "aryloxyacetic acid," with two methods of preparation that could not give the same product. It would seem to be *p,p'*-(1,1-cyclohexyl)-bis-(3-methylphenoxyacetic acid).

(5) J. F. Bartlett with C. E. Garland, THIS JOURNAL, **49**, 2098–2101 (1927).

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Hexadecyl Trisulfide and Hexadecyl Tetrasulfide

Several low molecular weight tetrasulfides were prepared by Chakravarti[1] by the action of sulfur monochloride on mercaptans. This method has now been applied successfully to the preparation of hexadecyl tetrasulfide, and by an obvious extension of the method, hexadecyl trisulfide has been prepared by the reaction of hexadecyl mercaptan with sulfur dichloride.

(1) Chakravarti, *J. Chem. Soc.*, **123**, 964 (1923).

TABLE I

HEXADECYL SULFIDES

Sulfides		Tri-	Tetra-
M. p., °C.		41.2–41.9	35.9–36.5
Yield, %		62	67
Formula		$C_{32}H_{65}S_3$	$C_{32}H_{65}S_4$
Sulfur, %	Calcd.	17.58	22.15
	Found	17.10 17.35	21.78 21.71
X-Ray diffraction, Å.		13.0	11.9
		4.39[b]	5.14
		4.26	4.66[a]
		4.03	3.82
		3.82[a]	3.70[b]
		3.70	3.58
			3.32

[a] Most intense line. [b] Second most intense line.

Hexadecyl Trisulfide.—A solution of 0.5 mole of sulfur dichloride in 150 ml. of petroleum ether was added slowly to a stirred solution of 1 mole of hexadecyl mercaptan in 500 ml. of petroleum ether, the temperature rising from 17 to 27°. After all of the sulfur dichloride had been added, the reaction mixture was refluxed for one hour to drive off hydrogen chloride. The solution was then diluted with an equal volume of dry acetone, chilled and the resulting white crystals recrystallized from a mixture of equal volumes of petroleum ether and acetone.

Hexadecyl Tetrasulfide.—The same procedure as above was followed, except that 0.25 mole of sulfur monochloride and 0.5 mole of hexadecyl mercaptan in 250 ml. of petroleum ether was used. Also, the final crystals were further purified by twice dissolving in petroleum ether and acetone and cooling.

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COMMUNICATIONS TO THE EDITOR

HIGHLY POLYMERIZED DESOXYPENTOSE NUCLEIC ACID FROM YEAST

Sirs:

The isolation of highly polymerized desoxypentose nucleic acid (DNA) from yeast is described below.

A chilled suspension of fresh washed baker's yeast (800 g.) in 0.1 *M* sodium citrate (180 cc.) was passed through a bacterial mill[1] (placed at our disposal through the courtesy of Dr. D. E. Green). On centrifugation at 4000 r. p. m. for ninety minutes two solid layers sedimented, the lower one consisting of intact cells. The upper layer (290 g.), containing, among other cellular fragments, material stainable by the Feulgen technique, was treated with one volume of 1 *M* sodium chloride solution.[2] The addition of 2 volumes of alcohol to the supernatant resulting from the centrifugation of the very viscous mixture for 120 minutes at 4000 r. p. m. produced threads which were easily separated from a granular precipitate. (All operations were performed in the cold.)

The threads were dissolved in 1 *M* sodium chloride solution (300 cc.) and freed of protein by repeated shaking with chloroform–octanol.[3] The amount of extracted DNA, estimated by means of the diphenylamine reaction[4] (standardized against DNA from thymus), corresponded to about 0.04% of the cell debris.

The preparations still contained considerable amounts of ribonucleic acid and of a hexose-containing polysaccharide which could be removed by electrophoretic fractionation or, more economically, by the following method. The threads produced from the deproteinized solution by 2 volumes of alcohol were dissolved in a 10% calcium chloride solution to give an approximately 0.2% solution. The cloudy viscous solution was clarified by centrifugation at 20,000 r. p. m. and the nucleic acid precipitated as threads with 0.3 volume of alcohol. (The yields were increased by reworking the granular sediments produced in these operations.) To the solution of the combined threads in 0.1 *M* borate buffer of *p*H 7.8 a small amount (1% of the nucleic acid weight) of crystalline ribonuclease, obtained through the kindness of Dr. M. Kunitz, was added and the mixture subjected to dialysis against the same buffer for fourteen hours at room temperature and then against ice-cold distilled water for one day. The solution was deproteinized once more and evaporated in the frozen state in a vacuum. In this manner around 70% of the DNA present in the original extract was recovered.

The purified preparations were readily soluble in water giving clear highly viscous solutions. Different samples were found to contain 87 to 91% of DNA, 6 to 8% of ribonucleic acid and 3 to 5% of a polysaccharide. A representative specimen contained N 13.5, P 8.3% (N:P ratio 3.6).

The DNA of yeast exhibited an absorption maximum at 2605 Å., a minimum at 2320 Å. Its electrophoretic mobility (phosphate buffer, *p*H 7.4) was -15.7×10^{-5}. The specific viscosity of a 0.115% solution in water at 30.3° was 5.9,

(1) V. H. Booth and D. E. Green, *Biochem. J.*, **32**, 855 (1938).

(2) F. Miescher in F. Hoppe-Seyler, "Medicinisch-chemische Untersuchungen," Berlin, 441 (1871).

(3) M. G. Sevag, *Biochem. Z.*, **273**, 419 (1934).

(4) Z. Dische, *Mikrochemie*, **8**, 4 (1930).