

obtain and the dimer had a viscosity index (Dean and Davis system) of 123.0.

STANDARD OIL COMPANY OF NEW JERSEY
ELIZABETH, NEW JERSEY RECEIVED MARCH 3, 1944

NEW COMPOUNDS

New Phenolic Mercurials¹

In a study of the preparation of new phenols of possible pharmaceutical interest the following new derivatives, which include mercurials of *p*-*t*-octylmono-,^{2a} cyclohexylidenedi-,^{2b} and hexyl-tetraphenols,^{2c} have been prepared and characterized.

for such ions with potassium iodide solution (red precipitate of mercuric iodide soluble in excess of reagent), the solution was then chilled in the refrigerator and the crude mono-mercurial filtered off and dried. The monomercurial was recrystallized from 50% aqueous alcohol containing 5% glacial acetic acid. The yield was about 80-85%.

Chloromercurials.—These were obtained by pouring an alcoholic or a glacial acetic acid solution of the acetoxymercuri derivative into an equal volume of a 20% aqueous solution of reagent grade sodium chloride. The chloromercurial was filtered off and recrystallized from ethanol or glacial acetic acid. The yields varied from 70-90%.

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TABLE I
NEW PHENOLIC MERCURIALS

Name	Formula	M. p., °C. (uncor.)	Yield, %	Percentage Hg ^a Calcd.	Found
I. 4- $\alpha,\alpha,\gamma,\gamma$ -Tetramethylbutylphenols					
(a) 2-Acetoxymercuri-	C ₁₆ H ₂₄ O ₂ Hg	158	80	43.16	43.10
(b) 2-Chloromercuri-	C ₁₄ H ₂₁ OHgCl	161	90	45.46	45.30
(c) 2,6-Diacetoxymercuri-	C ₁₆ H ₂₀ O ₄ Hg	181	90	55.46	54.60
(d) 2,6-Dichloromercuri-	C ₁₄ H ₂₀ OHg ₂ Cl ₂	238D ^a	80	59.18	58.60
(e) 2,6-Diacetoxymercuri-3-hydroxy-	C ₁₈ H ₂₆ O ₄ Hg ₂ ^b	183D ^a	75	54.26	54.68
(f) 2-Acetoxymercuri-6-methyl-	C ₁₇ H ₂₆ O ₂ Hg	149	80	41.87	42.05
II. 1,1-Bis-(4'-hydroxyphenyl)-cyclohexane					
(a) Bis-(2'-acetoxymercuri-6'-methyl)-	C ₂₄ H ₂₈ O ₆ Hg ₂	200D	90	49.33	49.60
(b) Bis-(2'-chloromercuri-6'-methyl)-	C ₂₀ H ₂₂ O ₂ Hg ₂ Cl ₂	225D	80	53.05	52.10
(c) Bis-(2',6'-diacetoxymercuri)-	C ₂₈ H ₂₈ O ₁₀ Hg ₄	210D	82	61.60	61.50
(d) Bis-(2',6'-dichloromercuri)-	C ₁₈ H ₁₆ O ₂ Hg ₄ Cl ₄	222D	75	66.45	66.10
III. 2,2,5,5-Tetrakis-(4'-hydroxyphenyl)-hexanes					
(a) Tetrakis-(2',6'-diacetoxymercuri)-	C ₄₆ H ₄₆ O ₂₀ Hg ₈	308D	91	63.60	63.00
(b) Tetrakis-(2',6'-dichloromercuri)-	C ₃₀ H ₂₂ O ₄ Hg ₈ Cl ₈	247D	80	68.74	67.70

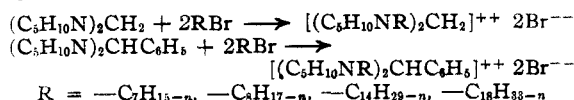
^a Decomposes. ^b The chloromercurial is an oil.

Polyacetoxymercurials.—A molar equivalent of mercuric acetate was allowed for each free position ortho-to-the-hydroxyl group per mole of phenol. Knowing the ratio of the reactants to be used, the quantities of phenol and mercuric acetate could be calculated for any size batch desired or found to be convenient. A 2-6% solution of the phenol with the calculated amount of mercuric acetate was refluxed in 95% ethyl alcohol containing 5% glacial acetic acid in an appropriate flask, fitted with a reflux condenser, for one to two hours. The flask was then chilled in the refrigerator overnight, the polymercurial filtered off and recrystallized from ethanol or glacial acetic acid. The yields were in the neighborhood of 70-90%.

Monoacetoxymercurials.—For these one mole of phenol was allowed to react with only one-half the quantity of mercuric acetate as calculated above, in a 50% aqueous ethyl alcohol solution containing 5% glacial acetic acid, at room temperature, over a period of one to two weeks. When the solution no longer contained free mercuric ions, as shown by the absence of a positive reaction when tested

Double Invert Soaps: Symmetrical Di-piperidinium Salts¹

Several long chain substituted dipiperidinium salts have been prepared in the course of investigation of various types of symmetrical double invert soaps. Methylene- and benzal-di-piperidine, prepared by condensing formaldehyde or benzaldehyde respectively with two moles of piperidine, were treated with double molar quantities of various *n*-alkyl bromides, such as *n*-heptyl, *n*-octyl, *n*-tetradecyl and *n*-hexadecyl bromides. Reaction took place rather readily in all cases and evidently proceeded as follows



Procedure.—To 0.02 mole of methylene- or benzal-di-piperidine was added 0.04 mole of the respective alkyl bromide and 20 ml. of 95% ethyl alcohol. The mixture was then refluxed gently for four hours. The alcohol was then removed by evaporation under reduced pressure. The remaining residue was repeatedly crystallized from ethyl acetate; yields, 50-70%.

(1) Abstracted from the thesis of Anthony J. Shukis presented to the Graduate School of New York University, New York, N. Y., in partial fulfillment for the degree of Master of Science, June, 1940.

(2) J. B. Niederl and co-workers, (a) THIS JOURNAL, **55**, 2571 (1933); (b) *ibid.*, **61**, 345 (1939); (c) *ibid.*, **63**, 1235 (1941).

(3) A. J. Shukis and R. C. Tallman, *Ind. Eng. Chem., Anal. Ed.*, **12**, 123 (1940).

(1) Abstracted from the thesis of Anthony E. Lanziloti presented to the Graduate School of St. Peter's College, Jersey City, N. J., in partial fulfillment for the degree of Master of Science, May, 1944.