duction of a small amount of impurity, such as water vapor, with each new sample of acrolein. If the impurity would act as a remover of radicals, the polymerization process as outlined above would be retarded until such time as the inhibitor was eliminated.

Summary

1. A definite correlation between photochemical polymerization and the absorption spectrum of acrolein has been found. In the region of sharp-banded absorption, λ 3660 to λ 3130, the quantum yield, Φ_P, is less than 0.5; at λ 3020, where the absorption is very diffuse, Φ_P is 1.0; at λ 2804, which lies on the border line between banded and the continuous region of absorption, Φ_P is 10; and at both λ 2654 and λ 2537, which presumably are definitely in the continuum, Φ_P is 19.

2. A mechanism involving free radicals has been proposed to account for the large quantum yields. The experimental results found by the authors and others are in good agreement with this mechanism.

3. An induction period has been found in the polymerization experiments. A possible cause of this induction has been suggested but further experiments will have to be done before the cause of induction can be established.

LOS ANGELES, CALIF. RECEIVED AUGUST 23, 1937

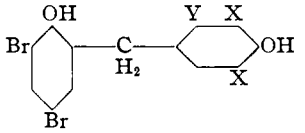
[CONTRIBUTION FROM THE CHEMICAL AND BACTERIOLOGICAL RESEARCH LABORATORIES OF HYNSON, WESTCOTT & DUNNING, INC.]

Condensation Products of 2-Hydroxy-3,5-dibromobenzyl Bromide with Phenols and their Germicidal Power

BY WILTON C. HARDEN AND JOHN H. BREWER

Auwers and Rietz,[1] Kohn and Jawetz[2] and others have described the condensation of various pseudo phenols with phenols. The purpose of the present paper is to report the preparation and some of the properties of a new series of benzyl phenols prepared by such condensations.

The dibromohydroxybenzyl bromide (2-hydroxy-3,5-dibromobenzyl bromide) used was prepared from *o*-cresol by the method of Auwers and Schröter.[3] This compound was condensed with the following phenols: phenol, resorcin, *o*-cresol and *m*-cresol, to give products having the general formula



where Y represents H, OH, or CH_3 and X represents H, CH_3, Br, or I.

The dibromo and in one case the diiodo derivatives of these substituted benzyl phenols were prepared. The properties of these compounds are summarized in Table I.

Similar condensations with *p*-cresol, naphthol, thymol and guaiacol have been made. Tribromo, tetrabromo, and diiodo-*o*-hydroxybenzyl bromides also have been prepared and condensed with various phenols. The resulting compounds will be described subsequently.

(1) Auwers and Rietz, *Ber.*, **38**, 3302 (1905).

(2) Kohn and Jawetz, (a) *Monatsh.*, **44**, 198 (1923); (b) *ibid.*, **45**, 251 (1924).

(3) Auwers and Schröter, *Ann.*, **344**, 142 (1906).

Experimental Part

The condensations were carried out by several different methods: (1) in boiling toluene in the presence of a piece of zinc; (2) in boiling toluene in the presence of anhydrous sodium carbonate; and (3) using aqueous alkali as described by Kohn and Jawetz.[2a] In general, the latter method gave the best yields, although in some cases even by this method the yields were poor. The following will serve as typical examples.

Preparation of 2-Hydroxy-3,5-dibromobenzyl-*o*-cresol.—34.5 grams of 2-hydroxy-3,5-dibromobenzyl bromide, 60 g. of *o*-cresol, 10 g. of sodium hydroxide and 10 g. of water were mixed and boiled gently with stirring under a reflux condenser for two hours. The mixture was then allowed to stand overnight, made acid, and steam distilled to remove unreacted *o*-cresol. The residue was dissolved in ether, dried with anhydrous sodium sulfate and crystallized twice from toluene; m. p. 154–155°. *Anal.* Calcd. for $C_{14}H_{12}O_2Br_2$: Br, 42.89. Found: Br, 43.04.

Preparation of 2-Hydroxy-3,5-dibromobenzyl-monobromo-*o*-cresol.—7.4 grams of dibromohydroxybenzyl-*o*-cresol was suspended in 50 cc. of glacial acetic acid and 7.0 g. of bromine in 25 cc. of glacial acetic acid was added drop by drop to the warm solution. The solution was warmed on the water-bath and stirred mechanically for one-half hour. It was then cooled, the crystals filtered, washed with cold water and recrystallized from dilute alcohol;

TABLE I

Dibromohydroxybenzyl-	Approx. yield, %	M. p., °C.	Formula		Halogen, % Found	Halogen, % Calcd.	Max. killing dilution at 37°[a] *S. aureus*	Max. killing dilution at 37°[a] *E. typh.*
Phenol	30	172–174	$C_{13}H_{10}O_2Br_2$		44.54	44.56	1:1200	1:500
Dibromophenol	90	195–197	$C_{13}H_8O_2Br_4$		61.92	61.89	1:3000	1:400
Diiodophenol	40	199–200	$C_{13}H_8O_2Br_2I_2$	I	41.22	41.62	1:5000	1:600
				Br	25.51	26.40		
Resorcin	91	198–199	$C_{13}H_{10}O_3Br_2$		42.59	42.79	1:250	1:200
Dibromoresorcin	90	213–214	$C_{13}H_8O_3Br_4$		60.03	60.15	1:300	Less than 1:100
m-Cresol	40	135–137	$C_{14}H_{12}O_2Br_2$		43.26	42.89	1:2000	1:600
Dibromo-*m*-cresol	90	157–158	$C_{14}H_{10}O_2Br_4$		60.31	60.37	1:8000	1:200
o-Cresol	24	154–155	$C_{14}H_{12}O_2Br_2$		43.04	42.89	1:1000	1:200
Monobromo-*o*-cresol	60	190–192	$C_{14}H_{11}O_2Br_3$		53.82	53.21	1:5000	1:500

[a] The dilution which gives a positive five-minute reading and negative ten- and fifteen-minute readings is taken as the "maximum killing dilution."

m. p. 190–192°. *Anal.* Calcd. for $C_{14}H_{11}O_2Br_3$: Br, 53.21. Found: Br, 53.82.

Bacteriological

The maximum killing dilutions shown in Table I were obtained using the F. D. A. Special Method.[4] The *Eberthella typhi* (Hopkins strain) was a twenty-four hour culture, the tenth in a series of transplants from agar. The *Staphylococcus aureus* (209) was the fourteenth in a series of transplants. Both strains show the proper resistance to phenol.

These tests were made at 37° and although no provision is made in the F. D. A. method for using *E. typhi* at this temperature, in order to get a more accurate comparison of these compounds, both organisms were used at 37°.

In regard to the dilutions used in these bacteriological tests, several of the compounds gave heavy precipitates with the organisms suspensions, so that the results may or may not be due to the actual dilution used. In any case, the bactericidal action is due to the sodium salt of the phenol.

Discussion

It is quite generally recognized that the activity of phenolic germicides is influenced greatly by the hydrogen ion concentration and that in general the sodium salts of such compounds are much less active than the free phenols. This has been demonstrated by Hailer,[5] Kojima,[6] and many others. The present compounds are of special interest because they constitute the first series of phenolic germicides, in the authors' experience, which are active at high dilution in the form of their *sodium salts*. They all form soluble monosodium salts when warmed with a slight excess over the calculated amount of standard alkali solution. All bacteriological tests were made using either 0.25, 0.5, or 1% solutions of these monosodium salts. These solutions all oxidize slowly, as is evidenced by a gradual darkening in color and eventual precipitation. The rate of this decomposition varies with different members of the series.

It will be noted that the compounds are much more active against the gram positive *Staph. aureus* than against the gram negative *E. typhi*.

(4) Ruehle and Brewer, U. S. Department of Agriculture, Circular No. 198 (1931).

(5) Hailer, *Arb. Reichsgesundh.*, **51**, 556 (1919).

(6) Kojima, *J. Biochem.*, **14**, 95 (1931).

Conclusion

1. The following compounds have been prepared and characterized: dibromohydroxybenzyl-phenol, resorcin, *o*-cresol, and *m*-cresol; dibromohydroxybenzyldibromo-phenol, resorcin, and *m*-cresol; dibromohydroxybenzylmonobromo-*o*-cresol; dibromohydroxybenzyldiiodophenol.

2. The bacteriological properties of these compounds have been investigated.

BALTIMORE, MARYLAND RECEIVED SEPTEMBER 3, 1937