decarboxylation of 13 g. of this material was carried out by heating at 330° until the evolution of carbon dioxide ceased. The resulting crude 4-hydroxy-7-iodoquinoline was recrystallized from 50% ethanol; 6 g. (54%) of light yellow powder, m. p. 306–308° (lit.[6] 346–348°).

Anal. Calcd. for C_9H_6ONI: C, 39.85; H, 2.22. Found: C, 40.31; H, 2.55.

4-Chloro-7-bromoquinoline.—Three grams of the 4-hydroxy-7-bromoquinoline was converted to 4-chloro-7-bromoquinoline by treatment with phosphorus oxychloride essentially as described by Surrey and Hammer.[6] After recrystallization from 95% ethanol there was obtained a 61% yield (2.0 g.) of the 4-chloro-7-bromoquinoline as white crystals, m. p. 105–106° (lit.[6] 100.5–101.5°).

Anal. Calcd. for C_9H_5NClBr: C, 44.50; H, 2.06. Found: C, 44.76; H, 2.35.

4-Chloro-7-iodoquinoline.—This was obtained in an analogous manner from 4 g. of 4-hydroxy-7-iodoquinoline. Recrystallization from 75% ethanol gave a 35% yield (1.5 g.) of light yellow crystals, m. p. 101–102° (lit.[5] 101°; lit.[6] 95.5–97°).

4-(7-Bromo-4-quinolylamino)-2-diethylaminomethylphenol.—The hydrolysis of 35.4 g. of 2-diethylaminomethyl-4-acetylaminophenol[14] was accomplished by refluxing for two hours with 300 ml. of 6 *N* hydrochloric acid. The *p*H of the solution was adjusted to approximately 3 with 105 ml. of a 40% sodium hydroxide solution. To 250 ml. of this solution was added 22 g. of 4-chloro-7-bromoquinoline and the reaction mixture was refluxed for three and one-half hours according to the method of Burckhalter and co-workers.[8,15] The viscous oil which separated was removed, dissolved in methanol, and reprecipitated by dilution with dilute ammonia solution. The product, after twice recrystallizing from a 1:1 solution of 95% ethanol and acetone, was obtained in 55% yield (20 g.) as a light yellow powder, m. p. 206–208° dec.

Anal. Calcd. for $C_{20}H_{22}ON_3Br$: C, 60.00; H, 5.50. Found: C, 59.96; H, 5.73.

4-(7-Iodo-4-quinolylamino)-2-diethylaminomethylphenol.—This was prepared and purified in a similar manner from 16 g. of 4-chloro-7-iodoquinoline; 49% yield (12 g.), light yellow powder, m. p. 196–198° dec.

Anal. Calcd. for $C_{20}H_{22}ON_3I$: C, 53.70; H, 4.92. Found: C, 54.18; H, 5.16.

Acknowledgment.—The authors are indebted to Parke, Davis and Company, Detroit, Michigan, for financial assistance.

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(14) Supplied by Parke, Davis and Company.

(15) Burckhalter, *et al.*, THIS JOURNAL, **70**, 1363 (1948).

DEPARTMENT OF CHEMISTRY
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STATE COLLEGE, PENNSYLVANIA
RECEIVED MAY 5, 1949

N-(Hydroxylethylmethylaminoethyl)-phenothiazine SC 1923: a New Antihistaminic

BY JOHN W. CUSIC

Many derivatives of phenothiazine have recently been made and studied for their anti-histaminic properties.

The 8-chlorotheophyllin salt of N-(dimethylaminoethyl)-phenothiazine was made by the author and tried clinically by Gay and Carliner.[1] Halpern[2] has reported extensively on several phenothiazines and recently N-pyrrolidylethylphenothiazine has been reported by Hunter, *et al.*[3]

N-(Hydroxylethylmethylaminoethyl)-phenothiazine (SC 1923) has been prepared by the reaction of N-methylethanolamine with N-(β-chloroethylphenothiazine).[4] Its hydrochloride melted at 185–186°. *Anal.* Calcd. for $C_{17}H_{21}N_2SOCl$; S, 9.52. Found: S, 9.62. The methobromide melted at 154–155°. *Anal.* Calcd. for $C_{18}H_{23}N_2SOBr$; Br, 20.21; S, 8.11. Found: Br, 20.24; S, 8.02.

When tested by Dr. Homer Freese of our Pharmacology Department according to the histamine spray technic of Loew[5] SC 1923 had an $ED_{50} = 0.43 \pm 0.15$ mg./kg. as compared to an ED_{50} of 0.66 ± 0.13 mg./kg. for β-dimethylaminoethylbenzhydryl ether.

Its effect on the mammalian capillary bed has been studied by Haley.[6]

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(1) Gay, *etc.*, *Bull. Johns Hopkins Hosp.*, **83**, 356 (1948).

(2) Halpern, *Compt. rend. soc. biol.*, **140**, 361, 363 (1946).

(3) Hunter, *et al.*, THIS JOURNAL, **70**, 3100 (1948).

(4) Gilman, THIS JOURNAL, **66**, 888 (1944).

(5) Loew, *etc.*, *J. Pharm. and Exper. Therap.*, **83**, 120 (1945).

(6) Haley and Harris, *ibid.*, **95**, 293 (1949).

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Non-exchange of Sulfur between Carbon Disulfide and Hydrogen Sulfide in Benzene Solution

BY DAVID L. DOUGLAS, ROBERT A. COOLEY AND DON M. YOST

The recent communication of Edwards, *et al.*,[1] in which they mention a study of the exchange of $S^{=35}$ in aqueous solution with carbon disulfide as a separate phase, prompts us to report some work done in this laboratory in 1941. We undertook the investigation of the exchange of S^{35} between H_2S^{35} and carbon disulfide in benzene solution. Our experiments, detailed in Table I, showed that no exchange greater than the experimental error (1%) occurs between carbon disulfide and hydrogen sulfide in benzene solution after ninety-five hours at 120°.

TABLE I

THE NON-EXCHANGE BETWEEN CARBON DISULFIDE AND HYDROGEN SULFIDE IN BENZENE SOLUTION[2]

Temp., °C.	Time of exchange, hr.	Concns. of reactants, moles/liter × 10^3		Observed activity, counts/min.		% exchange, max.
		H_2S	CS_2	H_2S	CS_2	
97	1	4.2	108	411 ± 3	0 ± 1	0.3
120	95	4.2	108	178 ± 1	0 ± 2	.8
120	95	4.2	108	181 ± 1	0 ± 1	.3

Experimental.—The source of the active sulfur and the counting technique are described in a previous paper.[3] A CS_2–C_6H_6 solution was made up by weighing out reagent grade carbon disulfide and mixing it with reagent benzene in a volumetric flask. The H_2S^{35}–C_6H_6 solution was prepared and analyzed by standard methods.

In a typical experiment 1 ml. of each of the two solutions were pipetted into a glass bulb of 5–10 ml. capacity. This was immediately immersed in liquid air and sealed off. The bulb was then placed in boiling water or a thermostated oven for a measured period of time. On completion of the run the hydrogen sulfide was trapped in 1 *N* sodium hydroxide and precipitated as silver sulfide. The carbon disulfide in the benzene was separated as potassium xanthate and precipitated as copper xanthate. The

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(1) R. R. Edwards, F. Nesbitt and A. K. Solomon, THIS JOURNAL, **70**, 1670 (1948).

(2) Ph.D. Thesis, R. A. Cooley, 1941, Cal. Tech.

(3) R. A. Cooley and D. M. Yost, THIS JOURNAL, **62**, 2474 (1940).