Anal. Calcd. for $C_{14}H_{13}Cl_3N_2$: C, 53.5; H, 4.1; N, 8.9. Found: C, 54.0; H, 4.3; N, 9.1.

The diamine is very soluble in acetone and warm ethanol; soluble in benzene, ether and chloroform; slightly soluble in petrol-ether (80°), very slightly soluble in water; soluble in dilute hydrochloric and sulfuric acids. It darkens on exposure to air or when its solutions are heated.

1,1,1-Trichloro-2,2-bis-(4′-acetaminophenyl)-ethane.—When a benzene solution of the diamine (I) was added to acetic anhydride at room temperature a colorless precipitate of the diacetyl derivative was immediately formed in quantitative yield, m.p. 268°.

Anal. Calcd. for $C_{18}H_{17}O_2N_2Cl_3$: C, 54.1; H, 4.3. Found: C, 54.6; H, 4.4.

Diazotization of (I).[3]—A solution of 0.47 g. sodium nitrite in 1 cc. of water was added dropwise with shaking to a solution of 1.052 g. (0.003 mole) of (I) in 6 cc. of water and 1 cc. of hydrochloric acid (25%) at 0–5°. After the diazonium solution had been carefully neutralized with sodium bicarbonate, a solution of α-naphthol was added, and a dark cherry-red precipitate was obtained.

When a diazonium solution of (I), prepared in a similar way, was warmed on a water-bath during two hours, the known bis-phenol precipitated. Recrystallization from benzene–ethanol gave a product of m.p. 202° (dec.).[2]

The nitrilation of the diazonium solution, prepared as above, gave a coke-like brown mass, from which we were unable to isolate a pure product either by crystallization or sublimation in high vacuum.

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[3] "Organic Syntheses," Coll. Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1946, p. 514.

[4] An earlier version of this manuscript was received on February 27, 1947.—*The Editor.*

2-Chloro-4-aminoquinazoline

BY F. J. WOLF, R. H. BEUTEL AND J. R. STEVENS[1]

In the course of an investigation of quinazoline derivatives, 2-chloro-4-methoxyquinazoline was treated with ammonia in an effort to prepare 2-amino-4-methoxyquinazoline. However, none of the desired product was isolated from the reaction mixture. Instead, 2-chloro-4-aminoquinazoline was obtained in good yield. The ester-like properties of alkoxy groups in the 4-position of 2,4-disubstituted quinazolines has been noted,[2,3] although treatment of 2-chloro-4-methoxyquinazoline with aniline at moderate temperature yields the expected 2-anilino-4-methoxyquinazoline.[3]

2-Chloro-4-aminoquinazoline.—A solution of 2.5 g. of 2-chloro-4-methoxyquinazoline[4] in 50 ml. of methanol saturated with anhydrous ammonia at 5° was heated at 100° for eighteen hours. The mixture was concentrated to dryness *in vacuo* and the residue dissolved in hot 2.5 *N* hydrochloric acid. The product, 1.7 g. (74% yield), was obtained by neutralization with dilute ammonium hydroxide. The product melted at 239° and was analytically pure.

Anal.[5] Calcd. for $C_8H_6N_3Cl$: C, 53.50; H, 3.36; N, 23.4. Found: C, 53.76; N, 3.20; N, 23.7.

The compound was more readily obtained by treating 2,4-dichloroquinazoline[6] with aqueous ammonia.

A mixture of 300 ml. of 28% ammonium hydroxide and 30 g. of 2,4-dichloroquinazoline was allowed to stand three hours at room temperature and then heated on a steam-bath for one hour. After cooling, the precipitate was filtered and dissolved in 500 ml. of boiling 0.5 *N* hydrochloric acid. The crude product, m. p. 234–235°, obtained on neutralization of the solution, was recrystallized from 350 ml. of 95% ethanol, yielding light yellow product, m. p. 237°, 13 g. (48% yield). When mixed with the product from 2-chloro-4-methoxyquinazoline, no depression in the melting point was observed.

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[1] Present address: J. T. Baker Co., Phillipsburg, New Jersey.

[2] Lange and Sheibley, THIS JOURNAL, **54**, 4305 (1932); **55**, 1188 (1933).

[3] Lange and Sheibley, *ibid.*, **54**, 1994 (1932).

[4] Lange, Roush and Asbeck, *ibid.*, **52**, 3699 (1930).

[5] Microanalyses were kindly carried out by R. N. Boos and W. K. Humphrey.

[6] Gabriel and Colman, *Ber.*, **38**, 3561 (1905); Bogert and Scatchard, THIS JOURNAL, **41**, 2061 (1919).

NEW COMPOUNDS

Kojic Acid Derivatives

2-(s-Butylthiomethyl)-5-hydroxy-4-pyrone.—Two and eight-tenths grams (0.12 mole) of sodium in 100 ml. of anhydrous toluene was powdered in the usual manner in a 250-ml. interjoint flask fitted with a sealed Hershberg stirrer, reflux condenser (soda lime tube), dropping funnel and thermometer. After cooling to about 70°, a solution of 10.8 g. (0.12 mole) of *s*-butyl mercaptan in 50 ml. of toluene was added during fifteen minutes with vigorous stirring. The mixture was stirred and refluxed for two hours, at the end of which time it was cooled to 10°, 18.5 g. (0.115 mole) of chlorokojic acid[1] was added and after remaining at that temperature for one hour, refluxing was initiated and maintained for three hours. After cooling, the mixture was transferred to a separatory funnel, cautiously diluted with 100 ml. of ice-water, shaken thoroughly and separated. The toluene layer was dried with sodium sulfate, filtered, and the solvent removed by distillation at reduced pressure, leaving a crystalline solid (12.1 g., 49% yield). This was recrystallized three times from hexane, giving a product melting at 93°.

Anal.[2] Calcd. for $C_{10}H_{14}O_3S$: C, 56.05; H, 6.58. Found: C, 56.32; H, 6.52.

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[1] Chlorokojic acid (m. p. 166°) has been prepared by Yobuta, *J. Chem. Soc.*, **125**, 575 (1924), from kojic acid and thionyl chloride in the absence of a solvent. It has been found more convenient to prepare this compound by the interaction of one mole of kojic acid (supplied by the Northern Regional Research Laboratory, Peoria, Illinois) with two moles of purified thionyl chloride in anhydrous chloroform under gentle reflux, followed by cooling, filtration and recrystallization of the compound from water. The yield of product melting at 166° was 78%.

[2] Analyses by Oakwold Laboratories, Alexandria, Virginia.