

the free amino acid in pure form has not been recorded in the literature.¹ In connection with studies in which S³⁵ was employed we had occasion to synthesize pure *d,l*-cysteine hydrochloride and wish to report our data at this time.

An optically inactive mixture of *d,l*-cystine and *meso*-cystine² was prepared from elementary sulfur by way of benzylmercaptan, benzylthiomethyl chloride, benzylthiomethylphthalimidomalonic ester and S-benzylcysteine by Seligman's³ modification of the procedure of Wood and du Vigneaud.⁴ Reduction of the mixed product with tin and hydrochloric acid afforded *d,l*-cysteine hydrochloride, m. p. 140–141.5° (dec.) in 62% yield. The sulfhydryl content of the product as determined by iodine titration was 98.7% of the theoretical value.

Experimental⁵

d,l-Cysteine Hydrochloride.—Optically inactive cystine (418 mg.) was dissolved in 5 ml. of 4.0 *N* hydrochloric acid; 400 mg. of granulated tin was added, and the reaction mixture was allowed to stand at room temperature in an atmosphere of nitrogen. At the end of four hours the tin had dissolved, and the solution was evaporated to dryness under reduced pressure to remove excess hydrochloric acid. The residue was dissolved in water, and the tin removed by saturation with hydrogen sulfide followed by filtration. The filtrate was then concentrated to dryness in vacuum. The crude product was finally washed with ethyl acetate, which removed a small amount of oil, and crystallized from absolute ethanol-ether; yield 340 mg. (62%), m. p. 140–141.5° (dec.). An additional 60 mg. of less pure material, m. p. 137–

140° (dec.), was obtained from the mother liquor. All operations were conducted under nitrogen.

Anal. Calcd. for C₃H₇O₂NS·HCl: C, 22.86; H, 5.11; N, 8.89; S, 20.34; Cl, 22.50. Found: C, 23.00; H, 5.20; N, 8.88; S, 20.66; Cl, 22.56.

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NEW COMPOUNDS

N,N'-Bis-(trichloromethylmethylol)-melamine

12.6 g. (0.1 mole) of pure melamine¹ was added to a stirred solution of 132 g. (0.8 mole) of chloral hydrate in 125 cc. of water at 70–80°. The melamine was completely dissolved within two minutes and a granular solid began to precipitate. After five minutes heating the suspension was cooled, the product filtered off and washed well with water. It weighed 35 g. (83% yield). On larger scale runs yields were usually 90% of theoretical. *Anal.* Calcd. for C₂H₈N₆·2C₂HOC₂Cl₃: N, 19.95; Cl, 50.6. Found: N, 19.9, 20.2; Cl, 50.5, 50.7.

When heated on a spatula the substance did not melt but evolved chloral. It did not react with boiling formalin. It was stable to dilute sodium hydroxide at room temperature but hydrolyzed rapidly on the steam-bath, liberating chloroform; pure melamine crystallized as the solution cooled.

We have observed that the reaction of chloral with melamine-formaldehyde mixtures or with water soluble methylolmelamines results in the formation of gels; this is attributed to the hydrolysis of chloral which lowers the reaction mixture to pH 4.

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(1) Salley and Gray, *This Journal*, **70**, 2650 (1948).

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COMMUNICATIONS TO THE EDITOR

SIMULTANEOUS VICINAL DICHLORINATION

Sir:

Iodine trichloride, postulated as the reagent when iodine is a carrier in direct chlorination,¹ has been neglected as a chlorinating agent. Crepaz² obtained *p*-chloroacetanilide when acetanilide was treated with KICl₄. Spring and Winsinger³ heated sulfonic acids with iodine trichloride and obtained various products. Thus ethanesulfonic acid with excess reagent heated to 150°

for seven hours produced hexachloroethane and chlorosulfonic acid, but with a smaller amount of reagent apparently 1,2-dichloroethanesulfonic acid was produced. On the other hand, 1-propanesulfonic acid yielded 1,1,1-trichloropropane and chlorosulfonic acid with excess iodine trichloride. The latter case suggests a normal ionic substitution mechanism, but the former is more difficult to interpret. Ingold and Ingold⁴ suggest that iodine trichloride acts as a chlorinating agent by dissociating to iodine monochloride and two active chlorine atoms



(4) Ingold and Ingold, *J. Chem. Soc.*, 1314 (1926).

(1) Fieser and Fieser, "Organic Chemistry," D. C. Heath and Co., Boston, Mass., 1944, p. 174.

(2) Crepaz, *Atti ist. veneto sci.*, **94**, 555 (1934–1935); *C. A.*, **31**, 6209 (1937).

(3) Spring and Winsinger, *Ber.*, **15**, 445 (1882); **16**, 326 (1883).