tion was filtered off, and the filtrate lyophilized. Approximately 20 g. of an amorphous hygroscopic product was obtained. The product was added to 100 ml. of glacial acetic acid, heated to 95° and kept warm until crystalline. After cooling to room temperature, the product (18 g.) was filtered off and was washed several times with ether. The compound was recrystallized as bundles of microscopic needles from approximately 50 times its weight of hot glacial acetic acid. The compound is assumed to be the acetic acid salt of S-cysteinosuccinic acid.

Anal. Calcd. for $C_9H_{15}NO_8S$: C, 36.47; H, 5.05; N, 4.88; S, 10.78. Found: C, 35.90; H, 5.10; N, 4.85; S, 10.77.

After drying over $CaCl_2$ and KOH for two weeks, the total acid equivalency of a sample based on a mol. wt. of 297 was 2.88 (theory 3.0), equivalents volatile acid 0.85 (theory 1.0), and equivalents non-volatile acid (calculated) was 2.03 (theory, 2.00). Upon heating the crystals became moist at 118 to 120°; they contract from 120 to 124°, and then swell until they decompose with bubbling at 132 to 134°. The compound is very soluble in ethanol and water but only slightly soluble in ether. It gives a negative —SH or —S—S— spot test but a positive test for thioether[5] and a positive ninhydrin test for amino acids.

Although the addition of sterile fumaric acid to agar media containing cysteine stimulates growth of small inocula of the yeast phase of *Histoplasma capsulatum* on agar media,[6] the addition of the S-cysteinosuccinic acid salt instead of fumaric acid has no effect. In liquid media the compound does not substitute for the cysteine requirement.

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3,4,5-Triiodobenzoyl Chloride as a Reagent for Identifying Mercaptans

BY DAVID C. O'DONNELL, HENRY A. MARIANI AND DENIS J. DOWNING[1]

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We have reported previously[2,3] the use of 3,4,5-triiodobenzoyl chloride for the identification of cellosolves, carbitols and alcohols. The increasing use of mercaptans has prompted us to extend the use of this acid chloride to the identification of these compounds. The acid chloride was prepared by the method previously described.[4] All of the mercaptans were obtained commercially.

Experimental

With all but two of the mercaptans, 1 ml. of the mercaptan was added to 1 g. of the acid chloride in a 15-cm. test-tube and gently heated with a micro-burner for 10 minutes. Methyl and isopropyl mercaptans, because of their low boiling points, were treated differently. In these two cases 1 g. of the acid chloride was dissolved in 50 ml. of ether, 1 g. of the mercaptan added and the solution was allowed to stand for 10 minutes. It was then heated for 5 minutes, the ether evaporated and the residue crystallized from a solvent. The lower molecular weight thioesters were crystallized from 40 ml. of either methyl or ethyl alcohol. The higher molecular weight aliphatic and the aromatic thioesters were crystallized from 20 ml. of *n*-butyl alcohol and after filtration the crystals were washed with a small amount of ether. All of the derivatives crystallized in the form of fine white needles with the exception of the two noted in Table I. All melting points are corrected.

TABLE I

THIOESTERS OF 3,4,5-TRIIODOBENZOIC ACID

Mercaptan used	M.p., °C.	Yield, %	Formula	Iodine, % Calcd.	Found
Methyl	153.6–154.6[a]	43	$C_8H_5OSI_3$	71.85	71.56
n-Propyl	97.8–98.8[a]	37	$C_{10}H_9OSI_3$	68.24	68.38
Isopropyl	153.4–155[a]	28	$C_{10}H_9OSI_3$	68.24	68.40
n-Butyl	90–91.4[a]	48	$C_{10}H_{11}OSI_3$	66.57	66.96
Isobutyl	89.8–90.8[a]	37	$C_{11}H_{11}OSI_3$	66.57	66.39
n-Amyl	83.2–84.4[b,d]	34	$C_{12}H_{13}OSI_3$	64.97	64.63
n-Hexyl	64.3–65.2[b]	49	$C_{13}H_{15}OSI_3$	63.63	63.63
n-Heptyl	70–70.8[b]	41	$C_{14}H_{17}OSI_3$	62.00	62.13
n-Octyl	67–68.2[b]	46	$C_{15}H_{19}OSI_3$	60.61	60.50
n-Nonyl	70–70.8[c]	64	$C_{16}H_{21}OSI_3$	59.29	59.03
n-Decyl	76–77.2[b]	55	$C_{17}H_{23}OSI_3$	58.04	58.09
n-Undecyl	78.6–79.8[c]	63	$C_{18}H_{25}OSI_3$	56.81	57.08
n-Dodecyl	78.4–78.8[c]	53	$C_{19}H_{27}OSI_3$	55.65	55.59
n-Tetradecyl	86.4–87.4[c,e]	39	$C_{21}H_{31}OSI_3$	53.46	53.76
Hexadecyl	91.0–91.8[c]	62	$C_{23}H_{35}OSI_3$	51.48	51.65
o-Thiocresol	98.4–99.0[c]	30	$C_{14}H_9OSI_3$	62.83	62.44
Benzyl	116.2–117.0[c]	60	$C_{14}H_9OSI_3$	62.83	62.88
β-Phenylethyl	99.8–100.6[c]	63	$C_{15}H_{11}OSI_3$	61.41	61.62
α-Phenylpropyl	133.4–134.2[c]	59	$C_{16}H_{13}OSI_3$	60.05	59.83

[a] Methyl alcohol as solvent. [b] Ethyl alcohol as solvent. [c] *n*-Butyl alcohol as solvent. [d] White plates. [e] White granules.

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Preparation of Pure 2-Aminonitropyridines and 2-Aminonitropicolines. Rapid Separations by Sublimation

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Considerable information exists on the nitration of 2-aminopyridine and the 2-aminopicolines. However, there is reported no simple, rapid method of separating the isomeric nitroamines of the pyridine series. This paper gives the results of a study of sublimation as a means of rapid, clean separation.

Separation of aminonitropyridine has been accomplished by steam distillation and fractional crystallization.[2a,b,3] In some instances, no attempt at separation was made until the mixture of isomers had completed a series of additional reactions after which one modified isomer was recovered and the other lost.[2,3]

Separation by sublimation seemed feasible since a vicinal nitroamine is capable of chelation with a resulting increase in molecular symmetry and vapor

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