

Test.⁴ Treatment of I with acetic anhydride and sulfuric acid² yields scyllitol hexaacetate, m.p. 286°,⁵ in 60–70% yield (based on the cyclitol content of I). *Anal.* Calcd. for C₁₈H₂₄O₁₂: C 49.95, H 5.60. Found C 49.93, H 5.67. Myo-inositol hexaacetate was not detected as a product. After acidification of I with sulfuric acid, the resultant solution was subjected to repeated evaporation to dryness *in vacuo* at 50° with methanol. The final residue was taken up in water, deionized with Amberlite MB-3, and the eluate again taken to dryness *in vacuo*. The deionized residue, obtained in 90% yield from I (based on cyclitol content of I), was shown to give only one spot with the same *R_f* as scyllitol in paper chromatography with phenol-water or acetone water.

From microanalysis⁶ data, and with the assumption that it is monomolecular, compound I appears to be a monohydrate of scyllitol diborate. Calcd. for C₆H₈O₈B₂Na₂·9H₂O; C 16.45, H 5.99, B 4.94, water content 37.02. Found C 16.52, H 6.07, B 4.87. Weight loss on drying at 100° for 18 hr., 37.27.

If scyllitol is heated at 100° with 0.125*M* borate, a compound migrating at the same rate as I in borate ionophoresis is obtained. Presumably this is also scyllitol diborate. The stereochemistry of the borate complex is still unproved and is being further investigated.

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(4) J. T. Park and M. J. Johnson, *J. Biol. Chem.*, **181**, 149 (1949).

(5) Melting point determined on a Fisher-Johns melting point block.

(6) I am indebted to Dr. W. C. Alford of the National Institute of Arthritis and Metabolic Diseases, National Institutes of Health for the microanalyses.

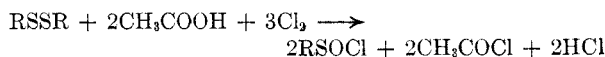
An Improved Method for Preparing Sulfinyl Chlorides

Sir:

The new method for the preparation of sulfinyl chlorides recently reported¹ represented a great improvement over previously available methods. The procedure has certain disadvantages, however, in that one has difficulty in determining when the stoichiometric quantity of chlorine has been added, a relatively large volume of inert solvent must be used to impart fluidity to the two-phase system, and the reaction must be carried out at

low temperatures to avoid the premature decomposition of the organosulfur trichloride.

We have recently found that if one mole of alkyl disulfide is mixed with exactly two moles of glacial acetic acid in the absence of solvent and chlorinated at 0° the reaction proceeds smoothly in a one-phase system and produces the desired sulfinyl chloride in high yield. On the first addition of chlorine the disulfide is transformed to the reddish orange sulfenyl chloride, RSCl. Additional chlorine apparently changes the sulfenyl chloride to the organosulfur trichloride, RSCl₃, which rapidly reacts with acetic acid to form acetyl chloride and the desired sulfinyl chloride. Since acetyl chloride is colorless and the sulfinyl chlorides are only faintly yellow, the disappearance of the reddish orange sulfenyl chloride color constitutes a good endpoint to indicate completion of the reaction. The products may be separated by fractionation of the reaction mixture without further treatment.



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Base-Catalyzed Rearrangement of α -Haloacetanilide into α -Anilinoacid Derivatives

Sir:

It has already been demonstrated that the action of sodamide in liquid ammonia upon β -chloropropionanilide and acrylanilide produces *N*-phenyl- β -lactam in fair yields.¹ In this communication we wish to report our results obtained in a study of the reaction of sodamide in liquid ammonia, and of a suspension of sodium hydride in dry benzene, upon α -chloro- α -phenylacetanilide (I). We have found that (I) gives by the action of sodamide in liquid ammonia a mixture of three compounds of which two have been identified as α -anilinophenylacetamide (III) and 2,5-diketo-1,3,4,6-tetrahydropiperazine (IV). Reaction of (I) with sodium hydride yielded IV as a major product.

To a solution of 5.1 g. (0.13 mole) of freshly prepared sodamide in 250 ml. of liquid ammonia was added 30 g. (0.12 mole) of solid (I)² with stirring during 1 hr. Within about 5 min. an orange-red solution was obtained which became brown-red at the end of the addition. The ammonia

(1) S. Sarel and R. Ben-Shoshan, *Bull. Res. Council of Israel*, **6A**, 298 (1957).

(2) C. A. Bischoff and P. Walden, *Ann.*, **279**, 124 (1894).

(1) I. B. Douglass and D. R. Poole, *J. Org. Chem.*, **22**, 536 (1957).