procedure developed by Jones and Swift,[3] and also by a combination acidimetric–iodometric method in which total acid was determined by titration with standard NaOH to the phenolphthalein end-point, and phosphorous acid was determined by the method of Jones and Swift. It should be pointed out here that no reliable method for the analysis of mixtures of hypophosphorous and phosphorous acids, involving only volumetric procedures, was found in the literature. Confirmatory data, demonstrating the accuracy of the method of Jones and Swift, will appear in a subsequent publication.[3]

The results of the analyses can be briefly summarized as follows: The mole percentage of phosphorous acid in solid hypophosphorous acid which had not been subjected to the liquefaction–filtration process was about 2.6%. Two filtration–liquefaction cycles reduced the mole percentage of phosphorous acid to 0.15%. Three such cycles reduced it to 0.05%.

Qualitative tests for phosphate were run on aqueous solutions of some batches of solid hypophosphorous acid prepared by this method, using the ammonium molybdate procedure recommended by Swift,[4] modified by the substitution of perchloric acid for nitric acid. At no time was any yellow coloration or yellow precipitate noted. Comparison tests run on mixtures of hypophosphorous acid and NaH_2PO_4 showed this test to be sensitive to about 0.1 mole per cent. phosphate in the presence of hypophosphorous acid.

Stability of Hypophosphorous Acid and Sodium Hypophosphite Solutions.—In acid solution, the hypophosphite ion is slowly air-oxidized. In neutral and basic solutions, the mechanism of decomposition is more complex.[1] Since very few data are available on this point, we conducted some experiments to ascertain the extent of decomposition of H_3PO_2 under various conditions. The results were as follows: (1) Solid acid, stored in a desiccator at 5°, underwent no appreciable decomposition in three months. (2) The phosphorous acid content of 0.1 *f* solutions of solid acid, stored at 5°, increased about 0.1% in three months. (3) The rate of decomposition of 0.05 *f*. solutions of C.P. sodium hypophosphite, stored at room temperature, increased with *p*H,[5] the percentage decomposition in a year being 85% for a solution of *p*H 5 and 0.8% for a solution of *p*H 1.5.

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(3) R. T. Jones and E. H. Swift, to be submitted to *Anal. Chem.*

(4) E. H. Swift, "A System of Chemical Analysis," Prentice-Hall, Inc., New York, N. Y., 1946, p. 550.

(5) *p*H varied by adding HCl.

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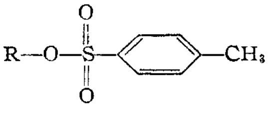
Infrared Absorption Spectra of *p*-Toluenesulfonic Acid and of Some of Its Esters

BY R. STUART TIPSON

In a study of certain *p*-toluenesulfonic esters of sugars and sugar derivatives, their infrared absorption spectra have been recorded. For interpretation of these spectra, it became desirable to identify the frequencies characteristic of molecules of the type



For (a) a para-disubstituted benzene ring, the Colthup chart[1] predicts a weak band in the 1590–1650 cm.$^{-1}$ region and a strong band in the 800–855 cm.$^{-1}$ region; for (b) $ROSO_2R'$, it predicts[1] strong bands in the 1330–1420 cm.$^{-1}$ and 1150–1200 cm.$^{-1}$ regions, respectively.

The infrared absorption spectra of *p*-toluenesulfonic acid (monohydrate) and of five of its esters have now been recorded;[2] the observed bands presumed due to the $-OSO_2$-*p*-C_7H_7 group are given in Table I.

TABLE I

INFRARED ABSORPTION OF *p*-TOLUENESULFONIC ACID AND ESTERS

Compound	Observed bands at (cm.$^{-1}$)
p-Toluenesulfonic acid monohydrate[a]	1605, 815; 1240, 1065, 1030
Phenyl *p*-toluenesulfonate[a]	1600, 816; 1375, 1192, 1170
2,4-Dinitrophenyl *p*-toluenesulfonate[b]	1610, 822; 1355, 1190, 1175
β-Phenoxyethyl *p*-toluenesulfonate[b]	1600, 815; 1350, 1185, 1168
Tetra-O-*p*-toluenesulfonylerythritol[c]	1600, 815; 1365, 1190, 1180
1-O-*p*-Toluenesulfonylglycceritol[d]	1605, 827, 810; 1360, 1180, 1170

[a] Eastman Kodak Co. [b] R. S. Tipson, *J. Org. Chem.*, 9, 235 (1944). [c] R. S. Tipson and L. H. Cretcher, *ibid.*, 8, 95 (1943). [d] R. S. Tipson, M. A. Clapp and L. H. Cretcher, THIS JOURNAL, 65, 1092 (1943).

Experimental

The source of each compound studied is indicated in Table I.

The infrared absorption spectra were recorded[2] on a Baird spectrophotometer; the wave length accuracy of this instrument is better than ±0.05 micron. All spectra were obtained for suspensions of the solid samples in Nujol. The relevant bands observed are given in Table I.

The bands exhibited by *p*-toluenesulfonic acid monohydrate are compatible with the frequencies predicted by the Colthup chart[1] for molecules of the type RSO_3H.

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(1) N. B. Colthup, *J. Optical Soc. Am.*, 40, 397 (1950).

(2) The author is indebted to Dr. Foil A. Miller and R. B. Hannan, Jr., of the Department of Research in Chemical Physics, Mellon Institute, for recording the infrared spectra.

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The Synthesis of δ-(*p*-Chlorophenyl)-hydantoic Acid-Cl^{36}

BY HOWARD H. WOEBER[1]

Many derivatives, formed by the reaction of phenyl isocyanate with amino acids, are described in the literature. These hydantoic acids are usually crystalline compounds soluble in organic solvents, and when labeled with a radioactive isotope, should be useful in amino acid determinations. δ-*p*-Chlorophenyl isocyanate-Cl^{36} was selected for study and the glycine derivative prepared. Although the steps in the synthesis are not new, considerable modification was necessary for the use of radioactive chlorine.[2]

***p*-Chloroacetanilide-Cl^{36}.**—Chlorine (35 mg., 5 microcuries) was evolved by the addition of cold, fuming sulfuric acid (3 ml., 15% SO_3) to $NaCl^{36}$ dissolved in superoxol (1 ml.) cooled in ice (chlorine yield, 95–100%). The chlorine was passed into 30 ml. of a saturated solution of acet-

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(1) 1325 N. W. 6th Ave., Gainesville, Florida.

(2) For material supplementary to this article order Document 3408 from American Documentation Institute, 1719 N Street, N.W., Washington 6, D. C., remitting $1.00 for microfilm (images 1 inch high on standard 35 mm. motion picture film), or $1.00 for photocopies (6 × 8 inches) readable without optical aid.