4,4′-Dinitrodiphenyl Ether.—Nine grams (0.05 mole) of the dry potassium salt of *p*-nitrophenol was heated with 20 g. (13-g. excess) of *p*-nitrofluorobenzene and 0.2 g. of copper powder at 200° for one hour. The excess of *p*-nitrofluorobenzene was recovered by distillation in steam and the crude 4,4′-dinitrodiphenyl ether crystallized from ligroin; yield 11 g. or 85%.

The dry potassium salts of *m*-nitrophenol, salicylic aldehyde, *p*-hydroxybenzaldehyde and *p*-hydroxybenzoic acid, when substituted for the dry potassium salt of *p*-nitrophenol in the above procedure, produce approximately the same yields of the corresponding ethers.

The dry potassium salt of *o*-nitrophenol, however, gives only 30% of the theoretical amount.

Analyses.—Since some of the ethers prepared above are not recorded in the literature, their melting points and analyses are here reported.

ANALYSES

Ether	M. p., °C.	Nitrogen, % Calcd.	Found	
p-Nitrophenyl isopropyl ($C_9H_{11}O_3N$)	B. p. 276–277 at 738 mm.	7.73	7.70	
p-Nitrophenyl phenylethyl ($C_{14}H_{13}O_3N$)	56–57	5.76	5.80	
p-Nitrophenyl *m*-tolyl ($C_{13}H_{11}O_3N$)	63	6.11	6.12	
p-Nitrophenyl *o*-iodophenyl ($C_{12}H_8O_3NI$)	105	4.11	4.20	
p-Nitrophenyl *p*-phenylphenyl ($C_{18}H_{13}O_3N$)	120	4.81	4.85	4.89
p-Nitrophenyl *p*-*tert*-amylphenyl ($C_{17}H_{19}O_3N$)	60	4.92	4.95	4.99
p-Nitrophenyl *p*-acetaminophenyl ($C_{14}H_{12}O_4N_2$)	153	10.30	10.30	
p-Nitrophenyl *m*-nitrophenyl ($C_{12}H_8O_5N_2$)	123	10.77	10.84	10.87
2-(*p*-Nitrophenoxy)-benzaldehyde ($C_{13}H_9O_4N$)	112	5.76	5.83	5.96

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The Preparation of Vinyl Iodide

BY JOHN SPENCE

Vinyl iodide is the one vinyl halide suitable for study because of its comparatively high boiling point and the absence of any marked polymerizing tendency. An improved method for the preparation of vinyl iodide was used whereby the time of preparation was reduced and the yield increased from 18 to 35%.[1]

Ethylene diiodide was prepared by passing a rapid current of ethylene through a number of flasks in series containing iodine dissolved in 80% alcohol in presence of a large excess of solid iodine. It was found advantageous to carry out this operation in the illumination supplied by a 500-watt lamp; 500 g. of iodine gave approximately 300 g. of product after the customary purification.

Vinyl Iodide.—To 282 g. (1 mole) of ethylene diiodide was added 334 cc. of 3 *N* sodium ethoxide. The distillate was collected at a temperature of

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[1] Baumann, *Ann.*, **163**, 319 (1872).

−30 to −50°. Distillation was continued with heating until no further separation of halide was observed on addition of a saturated solution of sodium chloride. Crude vinyl iodide was thus separated, washed with dilute sodium bisulfite solution, with water and finally dried over calcium chloride. On redistillation practically all the product passed over at a temperature of 56–56.5°. Approximately 50 g. was obtained. The refractivity was determined. Owing to the ease of oxidation of vinyl iodide the observations were made in an atmosphere of nitrogen.

SPECIFIC GRAVITY 2.037 (20°)

Line	Index of refraction	Molecular refraction (found)	Molecular refraction (calcd.)
Hydrogen (α)	1.53232	23.365	23.546
Sodium (D)	1.53845	23.651	23.769
Hydrogen (β)	1.55186	24.153	24.209
Hydrogen (γ)	1.56468	24.495	24.706

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The Mechanism of the Reduction of Sulfonyl Halides by Phosphorus Tribromide

BY A. H. KOHLHASE[1]

In a recent paper, Hunter and Sorenson[2] have stated that "previous work[3] has shown that sulfonyl chlorides are not reduced by phosphorus tribromide." This is in error as Kohlhase showed (Table I and Experiment XI) that 3-nitro-6-methylbenzenesulfonyl chloride was reduced readily and in good yield to the corresponding disulfide. It is true that sulfonyl chlorides were reduced far less *readily* than the corresponding bromides.

Kohlhase had concluded that the thiols formed in two instances of the reduction of sulfonyl derivatives by phosphorus tribromide, resulted as follows

$$\mathrm{RSSR} + \mathrm{PBr_3} \rightleftharpoons \mathrm{(RS)_2{\cdot}PBr_3}$$
$$\mathrm{(RS)_2{\cdot}PBr_3} + 4\mathrm{H_2O} \longrightarrow 2\mathrm{RSH} + 3\mathrm{HBr} + \mathrm{H_3PO_4}$$

Hunter and Sorenson concluded that the mechanism is

$$\mathrm{RSO_2Br} \xrightarrow{\mathrm{PBr_3}} \mathrm{RSBr} \xrightarrow{\mathrm{PBr_3}} \mathrm{RSPBr_4} \xrightarrow{4\mathrm{H_2O}} 2\mathrm{RSH} + 4\mathrm{HBr} + 2\mathrm{H_3PO_4}$$

This interpretation seems rather convincing but Kohlhase[3] showed that phenyl 3,5-dinitrobenzenethiosulfonate, treated with phosphorus tribromide and then with ether and water, gave a very good yield of 3,5-di-

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(1) Present address, Whipple, Arizona.

(2) Hunter and Sorenson, THIS JOURNAL, **54**, 3368 (1932).

(3) Kohlhase, *ibid.*, **54**, 2441 (1932).