330. *Diaryl-2 : 2'-disulphonic Acids and Related Compounds. Part I. The Diphenyl and the Ditolyl Series.*

By W. L. F. Armarego and E. E. Turner.

Syntheses of derivatives of diphenyl-2 : 2'-disulphonic acid are more surely based on Ullmann reactions with phenyl *o*-iodobenzenesulphonate than on the Barber–Smiles process of heating the alkali *o*-iodobenzenesulphonate with copper in aqueous copper sulphate. The reductive scission of 4 : 4'-dimethyldiphenyl-2 : 2'-disulphonyl dichloride into *m*-mercaptotoluene recorded by Barber and Smiles [1] is shown to be illusory.

The optical resolution of diphenyl-2 : 2'-disulphonic acid,[2] whilst contributing much to our knowledge of the relation between steric effects and optical stability, left unsolved a number of problems, to the solution of which the present investigations are addressed.

The formation of sodium diphenyl-2 : 2'-disulphonate by the action of copper powder on a boiling aqueous solution of sodium *o*-iodobenzenesulphonate and copper sulphate is not uniformly successful. Although the diphenyl derivative is formed, some of the iodosulphonate remains unchanged and some is de-iodinated. A surer way into the series is now found to be by the action of copper on phenyl *o*-iodobenzenesulphonate, which is readily obtained from *o*-iodobenzenesulphonyl chloride. The diphenyl diphenyl-2 : 2'-disulphonate formed is with difficulty hydrolysed by acid, but is converted by boiling alcoholic sodium ethoxide into the sodium disulphonate. It may be noted that Ferns and Lapworth,[3] by the action of one mol. of sodium ethoxide on phenyl toluene-*p*-sulphonate, obtained phenetole as one product, the process being expressed as follows :

$$C_7H_7{\cdot}SO_3Ph + NaOEt = C_7H_7{\cdot}SO_3Et + NaOPh$$
$$C_7H_7{\cdot}SO_3Et + NaOPh = C_7H_7{\cdot}SO_3Na + PhOEt$$

We find that if two mols. of sodium ethoxide are used no phenetole is detectable. Probably in this case the second reaction is :

$$C_7H_7{\cdot}SO_3Et + NaOEt = C_7H_7{\cdot}SO_3Na + Et_2O$$

Barber and Smiles [1] reduced diphenyl-2 : 2'-disulphonyl dichloride to 2 : 2'-dimercaptodiphenyl, which was oxidised by aqueous-alcoholic ferric chloride to the cyclic disulphide. We find that the latter is formed by the action of hydriodic acid in acetic acid on the

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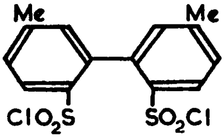
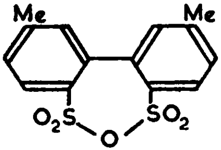
[1] Barber and Smiles, *J.*, 1928, 1141.
[2] Lesslie and Turner, *J.*, 1932, 2394.
[3] Ferns and Lapworth, *J.*, 1912, **101**, 279.

disulphonyl dichloride, and that the disulphide, and the cyclic thiolsulphonate obtained from it by oxidation,[1] both give dibenzothiophen when they are heated with copper powder.

By the deamination of *o*-tolidine-6 : 6′-disulphonic acid (cf. Limpricht[4] and Helle[5]) 5 : 5′-dimethyldiphenyl-2 : 2′-disulphonic acid is obtainable. The sodium salt of this acid is now found to react with a mixture of phosphorus pentachloride and phosphoryl chloride to give approximately equivalent quantities of the disulphonyl dichloride (I) and the disulphonic anhydride (II). The disulphonyl dichloride is reduced by zinc and acid to the 2 : 2′-dithiol, an alkaline solution of which reacts with 1-chloro-2 : 4-dinitrobenzene to give the di-(2 : 4-dinitrophenyl sulphide). The dithiol is readily oxidised to the cyclic disulphide, which is also formed from (I) and (II) by reduction with hydriodic–acetic acid. Both (I) and (II) are converted by aqueous alkaline alkali sulphite, followed by acidification, into the cyclic thiolsulphonate (III) which is also obtainable by oxidation of the cyclic disulphide. This disulphide, when heated with copper, passes into a dimethyldibenzothiophen which could be either (IV) or (V). The product is identical with that formed through the diazo-derivative of 2-amino-4 : 4′-dimethyldiphenyl sulphide and is therefore (IV). The two specimens of cyclic sulphide (IV) of different origin were oxidised to identical sulphones. 2 : 2′-Dihydroxy-5 : 5′-dimethyldiphenyl was obtained by demethylating its dimethyl ether, but it could not be converted into a dibenzothiophen by the action of phosphorus pentasulphide (cf. Gilman and Jacoby[6]).



(I)



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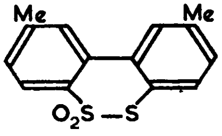

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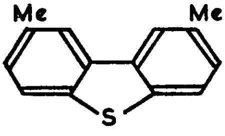

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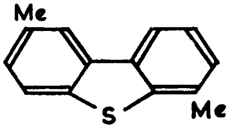

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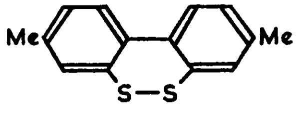

(VI)

By heating phenyl 4-iodotoluene-3-sulphonate with copper, phenyl 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonate is obtained. This ester is converted by boiling *n*-butanolic sodium *n*-butoxide into sodium 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonate, and the latter, with phosphorus pentachloride, gives 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonyl dichloride, m. p. 183—184°, not 117—118° as stated by Barber and Smiles. According to these authors, the disulphonyl dichloride was reduced by zinc and acid to *m*-mercaptotoluene. This seemed to us to be totally at variance with our knowledge of the 1 : 1′-diaryl linking, and we have re-examined the compounds concerned. The action of copper on a boiling aqueous solution containing copper sulphate and sodium 4-iodotoluene-3-sulphonate does not give appreciable quantities of sodium 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonate. The bulk of the material is unchanged and the rest is convertible by phosphorus pentachloride into 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonyl dichloride and toluene-*m*-sulphonyl chloride. The latter is evidently the source of the *m*-mercaptotoluene which Barber and Smiles isolated. We find that the dimethyldisulphonyl dichloride can in fact be reduced to a dithiol, which however readily undergoes oxidation to the cyclic disulphide (VI). An alkaline solution of the dithiol reacts with 1-chloro-2 : 4-dinitrobenzene to give the di-(2 : 4-dinitrophenyl sulphide). When heated with copper bronze, the cyclic disulphide (VI) gives 2 : 7-dimethyldibenzothiophen.

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[4] Limpricht, *Annalen*, 1891, **261**, 310.
[5] Helle, *ibid.*, 1892, **270**, 359.
[6] Gilman and Jacoby, *J. Org. Chem.*, 1938, **3**, 109.

EXPERIMENTAL

Diphenyl-2 : 2′-disulphonyl Dichloride.—The procedure of Barber and Smiles [1] was modified to the following.

To a boiling, stirred solution of sodium *o*-iodobenzenesulphonate (20 g.) in water (100 ml.) containing 1 ml. of 5% copper sulphate solution copper bronze (12 g.) was added. After 2½ hr. the mixture was filtered. The filtrate was evaporated to dryness and the white residue was dried at 120—130° and treated with an equal weight of phosphorus pentachloride. Benzenesulphonyl chloride was identified by conversion into benzenesulphonamide, m. p. 153°. The diphenyl-2 : 2′-disulphonyl dichloride, crystallised from glacial acetic acid, had m. p. 142—144° (Limpricht gives m. p. 138°) (yield, 2·9 g., 12% calc. on the iodo-salt).

Diphenyl Diphenyl-2 : 2′-disulphonate.—(*a*) The sulphonyl chloride (1·1 g., 1 mol.) in dry pyridine (10 ml.) was heated with phenol (1·2 g., 4 mols.). The ester obtained in the usual manner crystallised from ethanol in needles, m. p. 127—128° (0·4 g., 28%).

(*b*) *o*-Iodobenzenesulphonyl chloride (132 g., 1 mol.) was heated with phenol (2 mols.) and sodium carbonate (1½ mols.) on a boiling-water bath for 1 hr. *Phenyl* o-*iodobenzenesulphonate* crystallised from ethanol in needles, m. p. 93—94° (142 g., 92%) (Found: C, 40·3; H, 2·4; I, 35·4; S, 8·8. $C_{12}H_9O_3IS$ requires C, 40·0; H, 2·5; I, 35·2; S, 8·9%).

This ester (133 g.) was heated in a metal bath at 180—190°, and an equal weight of copper bronze was added at such a rate as to keep the temperature below 210°. This reaction was vigorous. The *diphenyl ester* was extracted with *o*-dichlorobenzene, and the product obtained by concentration. It crystallised from ethanol in needles, m. p. 127—128° (70 g., 81%) (Found: C, 61·6; H, 3·8; S, 13·6. $C_{24}H_{18}O_6S_2$ requires C, 61·8; H, 3·9; S, 13·7%).

Hydrolysis.—Diphenyl diphenyl-2 : 2′-disulphonate (9·4 g., 1 mol.), in absolute ethanol (200 ml.), was treated with a solution of sodium (1·84 g., 4 atoms) in ethanol (150 ml.). The mixture was boiled under reflux for 2 hr.; separation of solid was then complete. This was filtered off and dissolved in water (100 ml.), the solution was acidified with dilute hydrochloric acid, and phenol extracted with ether. The aqueous layer, after careful neutralisation with 10% aqueous sodium hydroxide, was evaporated to dryness. The residue (4·8 g.) with phosphorus pentachloride gave a disulphonyl dichloride (43%), m. p. 143—144°, mixed m. p. 142—143°.

2 : 2′-Diphenylene Disulphide (*Dibenzo-*o-*dithiin*).—Diphenyl-2 : 2′-disulphonyl dichloride (0·9 g.) in hot glacial acetic acid (10 ml.) was added to 55% w/w hydriodic acid (20 ml.), and the solution left at room temperature for 24 hr. The solid that separated was washed with aqueous sulphur dioxide; it crystallised from glacial acetic acid as yellow needles (0·4 g., 55%), m. p. 113—114° alone or mixed with the disulphide obtained by oxidation of the dithiol with ferric chloride.

Dibenzothiophen from "Diphenyl-2 : 2′-thiolsulphonate" (*Dibenzo-*o-*dithiin 5 : 5-Dioxide*; Ring Index No. 1936).—The thiolsulphonate (1 g.) was heated with copper bronze (2 g.) at 250° for 2 hr. Extraction with ethanol gave dibenzothiophen (0·5 g., 63%), m. p. 100°. The mixed m. p. with dibenzothiophen obtained by similar treatment of 2 : 2′-diphenylene disulphide was 98—100°. The thiolsulphonate was obtained from the disulphide in 60% yield by oxidation with concentrated nitric acid in acetic acid.

Disodium 5 : 5′-Dimethyldiphenyl-2 : 2′-disulphonate.—This was prepared by a modification of the method used by Limpricht [4] for diphenyl-2 : 2′-disulphonic acid. *o*-Tolidine-6 : 6′-disulphonic acid (111·6 g., 1 mol.) was suspended in water (600 ml.) and neutralised with a solution of sodium hydroxide (16 g. in 20 ml. of water). Diazotisation was carried out between −5° and −2° since the tetrazo-salt decomposed rapidly above −2°. Sodium nitrite (60 g., 1 mol.) in water (150 ml.) was added to the cold solution, and was followed slowly by dilute sulphuric acid (concentrated acid, 120 ml., and ice 600 g.). The tetrazo-salt that separated was kept at −5° for 1 hr., then rapidly filtered off, dried between filter paper, and added to absolute ethanol (600 ml.). On addition of copper bronze (1 g.) the tetrazo-salt decomposed. The alcohol was then distilled off and the residue dissolved in water (800 ml.), neutralised with 10% aqueous sodium hydroxide, boiled with charcoal, filtered, and concentrated. On cooling, the sodium salt crystallised in plates (yield of anhydrous salt, 88 g., 68%).

5 : 5′-Dimethyldiphenyl-2 : 2′-disulphonyl Dichloride and -2 : 2′-disulphonic Anhydride.—Sodium 5 : 5′-dimethyldiphenyl-2 : 2′-disulphonate (88 g., 1 mol.) was ground with phosphorus pentachloride (94 g., 2 mols.). The mixture did not melt when heated on a boiling-water bath, and phosphoryl chloride (25 ml.) was added and the mixture heated in a metal-bath at 130° under a condenser for 1 hr. The yellow pasty product was decomposed with ice-water, filtered off, dried, and dissolved in hot benzene. On cooling, the *anhydride* crystallised in rhombic crystals, m. p. 234—235° (18 g., 27%) (Found: S, 19·5. $C_{14}H_{12}O_5S_2$ requires S, 19·8%). The mother-liquor was concentrated to a small volume by distilling off the benzene at a low temperature in a vacuum. The *disulphonyl dichloride* crystallised in needles (24 g., 28%), m. p. 227—228° (mixed m. p. with anhydride, 195—199°) (Found: S, 17·1. $C_{14}H_{12}O_4Cl_2S_2$ requires S, 16·9%). Both substances were recrystallised from acetic anhydride.

Diphenyl 5 : 5′-*Dimethyldiphenyl*-2 : 2′-*disulphonate*.—This was prepared in the usual manner from the anhydride or disulphonyl dichloride but with dry pyridine instead of sodium carbonate; with the latter the original material was always hydrolysed to the disulphonic acid. The *ester* crystallised from *n*-butanol in needles, m. p. 189—191° (Found: S, 13·2. $C_{26}H_{22}O_6S_2$ requires S, 12·9%).

5 : 5′-*Dimethyldiphenyl*-2 : 2′-*disulphonanilide*.—This *dianilide* was obtained in the usual manner from the dichloride, and crystallised from *n*-butanol in needles (27%), m. p. 234—236° (decomp.) (Found: S, 12·9. $C_{26}H_{24}O_4N_2S_2$ requires S, 13·0%).

" 5 : 5′-*Dimethyldiphenyl*-2 : 2′-*thiolsulphonate* " (2 : 9-*dimethyldibenzo*-o-*dithiin* 5 : 5-*dioxide*) (III) was obtained by shaking the anhydride with alkaline sulphite for 6 hr. at 80—100°, and then strongly acidifying the solution with dilute mineral acid. It crystallised from *n*-butanol in needles, m. p. 146—147° (45%) (Found: S, 23·2. $C_{14}H_{12}O_2S_2$ requires S, 23·2%). Similar results were obtained with the disulphonyl dichloride.

5 : 5′-*Dimethyl*-2 : 2′-*diphenylene Disulphide* (2 : 9-*Dimethyldibenzo*-o-*dithiin*).—5 : 5′-Dimethyldiphenyl-2 : 2′-disulphonyl dichloride (7·6 g.) in boiling glacial acetic acid (120 ml.) was treated with 55% (w/w) hydriodic acid (80 ml.). An oil separated which on cooling solidified. It crystallised from glacial acetic acid in golden-yellow plates, m. p. 88—89° (3·3 g., 69%). Under similar conditions the anhydride gave the disulphide, m. p. and mixed m. p. 89—90°, in 53% yield.

Oxidation. The disulphide (1 g.) in boiling glacial acetic acid (15 ml.) was slowly treated with concentrated nitric acid (20 ml.). The solution was kept for 3 hr. but no solid separated. The mixture was poured into water (100 ml.) and the white thiolsulphonate that separated was filtered off and dried; it crystallised from *n*-butanol as needles, m. p. and mixed m. p. 144—146° (0·2 g., 42%).

5 : 5′-*Dimethyldiphenyl*-2 : 2′-*dithiol and the* 2 : 2′-*Di*-(2 : 4-*dinitrophenyl Sulphide*).—A solution of 5 : 5′-dimethyldiphenyl-2 : 2′-disulphonic anhydride (7·6 g.) in boiling glacial acetic acid (300 ml.) was rapidly cooled to 15° in order to avoid hydrolysis. Zinc dust (12 g.) was added, followed by concentrated hydrochloric acid (20 ml.). The acid was added at such a rate as to keep the temperature below 20°. Then the mixture was heated on a boiling-water bath until all the metal had dissolved. The solution was concentrated to 150 ml. *in vacuo*, poured into water (400 ml.), and acidified with 2N-hydrochloric acid (400 ml.). The oil which separated was steam-distilled, and the distillate extracted with ether, dried ($CaCl_2$), and freed from ether. The oily residue had an unpleasant odour and did not solidify. This was the dithiol as it was freely soluble in alkali. It was dissolved in ethanol (15 ml.), made alkaline with 10% aqueous sodium hydroxide (2 ml.), and mixed with a solution of 1-chloro-2 : 4-dinitrobenzene (2 g.) in ethanol (10 ml.). The yellow solid *disulphide* suddenly separated. It crystallised from large volumes of *n*-butanol in golden-yellow needles, m. p. 230—231° (0·9 g., 20%) (Found: S, 11·3. $C_{26}H_{18}O_8N_4S_2$ requires S, 11·1%).

The above reduction was repeated but with the disulphonyl dichloride, and the oily dithiol on oxidation with alcoholic ferric chloride gave 5 : 5′-dimethyl-2 : 2′-diphenylene disulphide in 20% yield.

2 : 8-*Dimethyldibenzothiophen* (Ring Index No. 1743).—(*a*) 5 : 5′-Dimethyl-2 : 2′-diphenylene disulphide (2 g.) was thoroughly mixed with copper bronze (2 g.) and heated at 250—260° for 1½ hr. The residue was distilled in a vacuum and the distillate crystallised, on cooling, to needles of the *dibenzothiophen* which, recrystallised from light petroleum (b. p. 60—80°), had m. p. 121—122° (1·4 g., 80%) (Found: S, 15·3. $C_{14}H_{12}S$ requires S, 15·1%).

(*b*) (cf. Cullinane, C. Davies, and G. Davies [7] for the similar synthesis of 2-nitrodibenzothiophen.) 4 : 4′-Dimethyl-2-nitrodiphenyl sulphide was prepared in the usual manner from 4-bromo-3-nitrotoluene, 4-mercaptotoluene and sodium ethoxide. The base obtained by reduction of the nitro-sulphide with tin and hydrogen chloride was an oil and was converted directly into the thiophen (yield, 0·5 g., 11%), m. p. 120—121°; mixed m. p. with above thiophen 119—120°.

2 : 8-*Dimethyldibenzothiophen* 1 : 1-*Dioxide*.—2 : 8-Dimethyldibenzothiophen (0·25 g., from

[7] Cullinane, Davies, and Davies, *J.*, 1936, 1435.

the disulphide) in boiling glacial acetic acid (20 ml.) was oxidised with excess of 3% aqueous potassium permanganate. The product was poured into water and decolorised with sodium metabisulphite, and the white precipitate filtered off. It crystallised in needles (from glacial acetic acid), m. p. 297—298° (0·2 g., 71%). The *dioxide* obtained in a similar manner from the thiophen derived from the nitro-sulphide melted at 298—299° (mixed m. p. 297—298°) (Found : C, 69·0; H, 5·1; S, 12·9. $C_{14}H_{12}O_2S$ requires H, 69·0; S, 4·9; 13·1%).

Phenyl 2-Iodo-5-methylbenzenesulphonate.—2-Iodo-5-methylbenzenesulphonyl chloride (148 g., 1 mol.) was heated as usual with phenol (2 mols.) and sodium carbonate (1½ mols.). The *ester* crystallised from ethanol in prisms, m. p. 87—88° (164 g., 94%) (Found : S, 8·9. $C_{13}H_{11}O_3IS$ requires S, 8·6%).

Diphenyl 4 : 4′-Dimethyldiphenyl-2 : 2′-disulphonate.—Phenyl 2-iodo-5-methylbenzenesulphonate (122 g.) was heated in a bath at 180—190° and copper bronze (100 g.) added slowly, the temperature being kept below 210°. The reaction was vigorous. By extraction with chlorobenzene and concentration, the *ester* was obtained crystalline. It crystallised from *n*-butanol in flakes, m. p. 146—147° (97 g., 92%) (Found : S, 13·2. $C_{26}H_{22}O_6S_2$ requires S, 12·9%).

Disodium 4 : 4′-Dimethyldiphenyl-2 : 2′-disulphonate.—The phenyl ester (1 mol.) was hydrolysed with sodium *n*-butoxide (4 mol.) in boiling *n*-butanol in 3 hr. The product was isolated in 96% yield by distilling off the alcohol, as in the previous hydrolysis.

4 : 4′-Dimethyldiphenyl-2 : 2′-disulphonyl Dichloride.—This was obtained from the above salt with the calculated quantity of phosphorus pentachloride. It crystallised from glacial acetic acid in needles (52%), m. p. 183—184° (Barber and Smiles gave m. p. 118°) (Found : S, 17·3. $C_{14}H_{12}O_4S_2Cl_2$ requires S, 16·9%). The phenyl ester obtained by heating this substance with phenol in pyridine was identical with diphenyl 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonate. The *disulphonanilide* crystallised from *n*-butanol in needles, m. p. 205—206° (Found : S, 13·1. $C_{26}H_{24}O_4N_2S_2$ requires S, 13·0%).

When sodium 4-iodo-3-methylbenzenesulphonate (20 g.) in water (100 ml.) containing 5% copper sulphate solution (1 ml.) was boiled under reflux and stirred mechanically for 3 hr., three substances were isolated : (i) sodium 4-iodo-3-methylbenzenesulphonate (13 g. anhyd., 65%), (ii) sodium 4 : 4′-dimethyldiphenyl-2 : 2′-disulphonate, and (iii) sodium toluene-*m*-sulphonate. The last two substances were converted into the corresponding sulphonyl chlorides. 4 : 4′-Dimethyldiphenyl-2 : 2′-disulphonyl chloride (2·2 g., 19% calc. on original iodo-salt) melted at 183—185° alone or mixed with the sulphonyl chloride obtained *via* the Ullmann reaction.

4 : 4′-Dimethyl-2 : 2′-diphenylene disulphide (*3 : 8-dimethyldibenzo-*o*-dithiin*) was obtained by the action of hydriodic acid (*d* 1·7) on the sulphonyl chloride in boiling glacial acetic acid. It crystallised from ethanol in yellow needles (81%), m. p. 114—115° (Found : C, 68·6; H, 4·9. $C_{14}H_{12}S_2$ requires C, 69·0; H, 4·9%).

‘ *4 : 4′-Dimethyldiphenyl-2 : 2′-thiolsulphonate* ” (*3 : 8-dimethyldibenzo-*o*-dithiin 5 : 5-dioxide*) was prepared in 75% yield in a way similar to that used for its isomer. It crystallised from *n*-butanol in needles, m. p. 170—171° (Found : C, 60·1; H, 4·7; S, 23·5. $C_{14}H_{12}O_2S_2$ requires C, 60·8; H, 4·4; S, 23·2%).

4 : 4′-Dimethyldiphenyl-2 : 2′-dithiol.—This was obtained by reducing the corresponding disulphonyl dichloride with zinc and hydrochloric acid in ethanol as in the case of the 3 : 3′-dimethyl compound. The dithiol (an oil) was not steam-distilled as it was oxidised readily to the cyclic disulphide in 63% yield. The *di-*(*2 : 4-dinitrophenyl sulphide*) crystallised from a large volume of *n*-butanol in canary-yellow needles, m. p. 236—237° (yield, 63%) (Found : N, 9·6. $C_{26}H_{18}O_8N_4S_2$ requires N, 9·7%).

3 : 7-Dimethyldibenzothiophen, obtained by the method used for its isomer, had b. p. 186°/10 mm. (yield 92%). The distillate crystallised from light petroleum (b. p. 60—80°) in needles, m. p. 151—152° (Found : C, 79·2; H, 5·9; S, 14·8. $C_{14}H_{12}S$ requires C, 79·9; H, 5·7; S, 15·1%).

*Di-*o*-iodophenyl Disulphoxide* (o*-Iodophenyl* o*-Iodobenzenethiolsulphonate*).—*o*-Iodobenzenesulphonyl chloride (6 g.) was reduced with sodium sulphite solution (15 g. of hydrated salt in 50 ml. of water) made alkaline with 10% sodium hydroxide solution (15 ml.). After being heated on a boiling-water bath for 15 min. the sulphonyl chloride dissolved. The resulting yellow solution gave *o*-iodobenzenesulphinic acid, on acidification, as needles, m. p. 112° (Barber and Smiles[1] gave m. p. 108°). The *thiolsulphonate* was isolated after the sulphinic acid had been heated for 2 hr. on a boiling-water bath in excess of dilute sulphuric acid; it crystallised from ethanol in needles (3·2 g.), m. p. 158—159° (Found : I, 51·0. $C_{12}H_8O_2S_2I_2$ requires I, 50·6%).

*Di-*o*-iodophenyl Disulphide.*—*o*-Iodobenzenesulphonyl chloride (3 g., 1 mol.) in hot glacial acetic acid (20 ml.) was treated with hydriodic acid (20 ml., 10 mols.) and left at room temperature for 24 hr. The disulphide separated and crystallised from a large quantity of glacial acetic acid in prisms (1·4 g., 83%), m. p. 135—136° (Barber and Smiles [1] give m. p. 133°).

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