LXIII.—*Phenylsulphonic Anhydride.*

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IN the *Annalen* (223, 238), Hübner describes a series of endeavours to prepare the anhydride of phenylsulphonic acid. His last attempt, in which he heated silver phenylsulphonate with phenylsulphonic chloride, was partly successful, but he lost the result of his labours by extracting the product with ether, which combined with the anhydride to form ethylic phenylsulphonate.

In ignorance of Hübner's work, Professor Odling suggested the preparation of this substance by the method mentioned above, and its extraction with chloroform. About 30 grams of the chloride, which, as Mr. Gossage has found, can be easily obtained in a state of purity (m. p. $18\cdot5^{\circ}$) by distilling the washed crude product under reduced pressure (8 mm. of mercury), was mixed with a slight excess of the silver salt prepared from sodium phenylsulphonate and silver nitrate, the two substances having been previously dried with great care. The mixture was heated at $160\text{--}180^{\circ}$ for 20 hours, and the greyish-brown solid formed was extracted with chloroform. On evaporating the brown solution, it deposited fine prismatic crystals, which were

obtained almost colourless by recrystallisation. After crushing them between porous plates the sulphur was estimated by Carius' method:—

I.	0.2032	gram substance	yielded	0.3114	gram barium sulphate,
II.	0.1890	„	„	0.2761	„

corresponding with 21.07 and 20.63 per cent. sulphur, the theoretical percentage for $(C_6H_6SO_2)_2O$ being 21.48.

As the substance is extremely deliquescent, it was impossible to weigh it without some absorption of water from the air. This will probably account in part for the somewhat low percentage of sulphur.

It melts at 54° , and when strongly heated a viscid liquid distils over, which deposits crystals on cooling; the greater part, however, is decomposed with formation of a black tarry matter.

It combines immediately with water, and gradually, as Hübner has shown, with ether. With phenol it forms a viscid liquid which I have not yet further examined.

It is remarkable that this anhydride corresponds with sulphur trioxide much more closely than the sulphonic chloride does with the chlorides of sulphuric acid, phenylsulphonic chloride being inappreciably acted on by water and only slowly decomposed by dilute alkalis. In this respect it contrasts with the anhydrides of the carboxylic acids, which are more stable in presence of water than their corresponding chlorides.

I must gratefully acknowledge the help which I have received from Mr. A. M. Gossage, of Magdalen College, in the preparation of this substance.