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PHENYL ETHER AND SOME OF ITS DERIVATIVES (ADDENDUM).[2]

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Bromination of p-Tolyl Ether.—The tetrabromo derivative was prepared by cautiously adding an excess of bromine in direct sunlight to

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[1] These references were obtained from the English translation of Pashutin's book, prepared under the direction of Francis G. Benedict of the Carnegie Institution.

[2] Omitted by mistake from the article in the October number of THIS JOURNAL.

the ether dissolved in carbon disulphide, while warming on the water bath. The resulting red viscous substance obtained was treated as in case of tetrabromophenyl ether. On distilling, a large amount of char was formed and the yield of tetrabromo-*p*-tolyl ether was small. It is a light yellow, viscous substance which crystallizes in radial aggregations on cooling *per se* and is quite identical in appearance with tetrabromo-*m*-tolyl ether. It boils at 310–330° under 40 mm. pressure. It is soluble in carbon disulphide and petroleum ether, but insoluble in alcohol and most ordinary organic solvents. On analysis it yielded 62.64 per cent. of bromine. Calculated for $(CH_3.C_6H_2Br_2)_2O$, 62.69 per cent. No compound with a higher per cent. of bromine could be prepared even with the aid of iodine as a carrier.

Bromine was gradually added to some *p*-tolyl ether heated, *per se*, to 150° in quantity to form the dibromo compound. On distillation a large amount of char resulted and there was obtained a small yield of dibromo-*p*-tolyl ether.

These two substances were boiled with water, caustic alkalies, water and calcium carbonate and sodium carbonate but no reaction could be obtained, which would indicate that the bromine had entered the nucleus.

Bromination of o-Tolyl Ether.—Since only a few grams of *o*-tolyl ether were obtained from the distillation of a kilogram of *o*-cresolate of aluminium, there was not sufficient material available for repeated attempts to prepare the bromine derivatives. The *o*-tolyl ether was undoubtedly much less pure than in case of the other ethers and the reactions were much less clear cut. Considerable char resulted on distilling the bromine derivatives and in one or two cases nothing but char was obtained. However, substances were obtained which contained bromine in amounts which corresponded more nearly to the dibromo and tetrabromo compounds than anything else, *e. g.*, in brominating the ether with a large excess of bromine in the presence of iodine there was obtained a substance which boiled at about 300° and which yielded results on analysis nearly 8 per cent. too low in bromine or the tetrabromo compound. However, while the reactions were not clear cut and the results unsatisfactory so far as obtaining pure compounds was concerned, it is evident that the bromine enters the nucleus and not the side chain, since on boiling for a long time with water, caustic alkalies, and water and calcium carbonate, the filtrate yielded no precipitate with silver nitrate.

VERMILION. S. D.

NOTE.

Note: Paraffin Hydrocarbons in Soils.—Through the kindness of Dr. F. B. Power, Director of the Wellcome Chemical Research Laboratories, of