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XLV.—o-Chlorobenzyl Bromide and its Products of Hydrolysis.

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THE author has prepared o-chlorobenzyl bromide by brominating o-chlorotoluene at its boiling point. The product, on treatment with a solution of potassium hydroxide in water and alcohol, gave in varying quantities o-chlorobenzyl alcohol, di-o-chlorobenzyl ether, and o-chlorobenzyl ethyl ether.

EXPERIMENTAL.

o-Chlorobenzyl Bromide, $C_6H_4Cl\cdot CH_2Br.$ —o-Chlorotoluene was heated to boiling in a flask fitted with a reflux condenser, and the calculated quantity of bromine added in drops by means of a funnel drawn out to a capillary tube. It is advisable to add a few pieces of fireclay to prevent bumping, and to dispense with all cork connexions.

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The product was washed with sodium carbonate, dissolved in ether, the solution dried, the ether evaporated, and the residue distilled in a vacuum. It is a colourless liquid boiling at 120°/ 10 mm., and possesses a peculiar, pungent odour. Its vapour irritates the eyes most powerfully:

0.2414 gave 0.3616 CO₂ and 0.0644 H₂O. C = 40.85; H = 2.96. 0.1666 AgCl , 0.2221 AgBr. Cl = 17.14; Br = 39.31. 0.2404,, C_7H_6ClBr requires C = 40.89; H = 2.94; Cl = 17.26;

Br = 38.90 per cent.

Action of Potassium Hydroxide in Aqueous Alcohol on o-Chlorobenzyl Bromide.

o-Chlorobenzyl bromide was heated under reflux with an excess of potassium hydroxide dissolved in water mixed with a small quantity of ethyl alcohol. After six or seven hours the liquid was found to be free from o-chlorobenzyl bromide by the absence of any irritating effect on the eyes. The product was then distilled in a current of steam with the following result.

The first product of the steam distillation consisted of oily drops which did not crystallise. The oil was extracted with ether and distilled in a vacuum. It is colourless, and possesses a faint but not Analysis showed it to consist of unpleasant aromatic odour. o-chlorobenzyl ethyl ether, C₉H₁₁OCl. It distils at 216°/761 mm. or at 103°/8 mm. (Mettler, Ber., 1904, 37, 3693, gives 212°).

After the distillation in steam had continued for some time the distillate commenced to crystallise in the condenser, when the receiver was changed and the distillation continued. The crystals were collected and were proved by analysis to consist of o-chlorobenzyl alcohol, C7H7OCl, melting at 70-71° (Mettler, loc. cit., gives 72°).

Di-o-chlorobenzyl Ether, $C_6H_4Cl\cdot CH_2\cdot O\cdot CH_2\cdot C_6H_4Cl$.—The contents of the flask on cooling after the steam distillation showed the presence of a crystalline residue. This was dissolved in ether, the ether evaporated, and the product twice crystallised from alcohol, from which it separates in colourless, hexagonal prisms melting at 48-49°:

0.3430 gave 0.8302 CO₂ and 0.1432 H₂O. C = 63.03; H = 4.63. 0.1244 , 0.1333 AgCl. Cl = 26.5.

 $C_{14}H_{12}OCl_2$ requires C = 62.92; H = 4.53; Cl = 26.56 per cent.

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