

NOTES.

Note on the Drying of Gas Streams. By LOUIS LEIGHTON BIRCUMSHAW.

FOR certain experiments it was necessary to have a constant stream of hydrogen in which the water content of the gas was reduced to such an extent that the finest film of oxide was not formed on a number of metals, *e.g.*, lead and tin, during prolonged passage of the gas through these metals at a temperature just above their melting points. The hydrogen was generated electrolytically from 5% sodium hydroxide solution, freed from traces of oxygen by passage over a long roll of hot copper gauze and then a roll of iron gauze at 600°, and dried by passage through tubes containing calcium chloride. Before entering the gas-tight furnace containing the metal it was passed through the usual form of trap immersed to the junction of the outlet tube in a Thermos flask containing acetone and solid carbon dioxide (− 78.0°). The pressure of water vapour in the gas should then be 0.0004 mm. After several hours a thin film of ice was found on the inner surface of the inner tube at the level of the freezing mixture. This film extended down the tube for about 5 mm., its lower boundary being quite sharp, and no ice was visible on the rest of the apparatus. In order to test the efficiency of this method, the gas after passing through this trap was conducted through an exactly similar one, the two being connected by 50 cm. of glass tubing. A similar thin film of ice was formed in the second as well as in the first trap, in spite of the fact that no ice could be seen on the inner wall of the outer tube and the outer wall of the inner tube of the first trap. When three traps were similarly connected in series, however, no ice was found in the third.

One possible method for the passage of water vapour through the first trap might be that owing to the intense “chilling” of the gas when it enters the trap a certain amount of ice fog is formed. This would probably take place in the gas phase away from the sides,

i.e., by "volume" cooling rather than "surface" cooling. The particles of this "ice aerosol" may fail to condense on the sides of the trap owing to an adsorbed film of gas. With a view to trapping these particles, if they are formed, and so throwing some light on this phenomenon, the following experiments were carried out.

(1) Two traps were used, the first being filled with small pieces of dried coco-nut charcoal. A good film of ice was still formed in the second trap.

(2) The first trap was immersed in a mixture of ice and water, and the second and third in acetone-carbon dioxide snow. A good film of ice was formed in the second and a fine film in the third.

(3) The first trap, containing a roll of copper gauze, was placed in the first Thermos flask, and empty traps in the second and third flasks. A ring of ice was formed in the second trap but none in the third.

(4) Experiment (3) was repeated with glass wool instead of copper gauze, with the same result. When the trap was packed with thoria a visible ring of ice was formed in the third trap as well as the second. This may have been due to an increase in the formation of ice fog owing to the α -particles and ionic nuclei present.

(5) It was thought that, although the particles of an ice aerosol might be totally reflected from a cold glass wall, if the latter were covered with a film of ice this reflexion would not take place; therefore the first trap, covered inside with a thin layer of ice, was placed in the first Thermos flask, and empty traps in the second and third. A good ring of ice was seen in the second trap and a faint ring in the third.

(6) Since it was possible that the above hypothesis was incorrect, it was decided to use even greater chilling. Acetone was rapidly evaporated from a mixture of acetone and solid carbon dioxide contained in a Thermos flask and the temperature of the mixture was thus reduced to -95° . The first trap was placed in this, and the second in ordinary acetone-carbon dioxide mixture (-78°). A distinct ring of ice was seen in the second tube, and the same result was obtained when the first trap was filled with (*a*) copper gauze and (*b*) glass wool.

(7) Three traps connected by pieces of glass tubing 75 cm. long were each immersed in a Thermos flask containing acetone-carbon dioxide: a good film of ice was seen in the second and a faint trace in the third.

(8) Experiment (7) was repeated, the long connecting tube between the first trap and the second being replaced by a short lagged connexion. A trace of ice was then seen in the second trap and a heavy film in the third.

Experiments (7) and (8) were repeated, with the same results, and hence it is concluded that the sudden cooling in the first trap causes the formation of an ice fog, and if the gas is then passed through a length of glass tubing exposed to room temperature this fog is destroyed and the remaining trace of water deposited in the next trap.

(9) The first trap was immersed in a Thermos flask containing a mixture of ice and salt connected with a short length of lagged tubing to the second trap in acetone-carbon dioxide. This was followed by a third trap connected with the second by a long piece of tubing. In numerous runs a heavy ring of ice was observed in the second trap and either no ice or only the faintest trace in the third. This method was ultimately adopted as being efficient and at the same time effecting a saving in the more expensive solid carbon dioxide.

In the above experiments the gas was passed through the system at a rate of about 15 c.c./min. and the duration of a run was 6 hrs. In cases where the trap was filled with some material, *e.g.*, charcoal, the gas was passed through in the reverse direction.

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Action of Monochlorodimethyl Ether on Magnesium Benzyl Chloride.
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A PATENT taken out by the Baeyer Co. (Kl.120, N.154658 vom 2/8/1903, 17/10/1904. Compare also *Centralblatt*, 1904, ii, 1355) states that when magnesium benzyl chloride acts on monochlorodimethyl ether, a colourless oil, b. p. 187—188°, is obtained, and that investigations have shown this to be the methyl ether of *o*-tolylcarbinol, $\text{CH}_3 \cdot \text{C}_6\text{H}_4 \cdot \text{CH}_2 \cdot \text{O} \cdot \text{CH}_3$.

In view of the possible theoretical significance of such a reaction, the authors carried out the following experiments.

Magnesium benzyl chloride prepared from benzyl chloride (63.2 g.) was added to chlorodimethyl ether (40 g.) in ice-cold, dry ether (400 c.c.). The ether-soluble portion of the acidified product, distilled at atmospheric pressure, gave a main fraction (27 g.), b. p. 175—205°, another (12 g.) with b. p. 205—270°, and a residue of fluorescent oil (17 g.). The main fraction was freed from a small quantity of unchanged benzyl chloride by prolonged treatment with

zinc dust and alcoholic potassium hydroxide, recovered, and re-fractionated. The portion distilling at 175—205° (16 g.), which should have comprised the bulk of the product which the Baeyer patent describes as the methyl ether of *o*-tolylcarbinol, b. p. 187—188°, was examined (Found: C, 79.6, 79.7, 79.4; H, 9.5, 9.3, 9.2. Calc. for $C_9H_{12}O$: C, 79.4; H, 8.9%).

A sample (12 g.) was boiled with dilute aqueous permanganate and a little alkali for 48 hours; the whole was then cooled, and the manganese dioxide dissolved by leading in sulphur dioxide. A white powdery material (4.8 g.) remained undissolved: this evidently contained much benzoic acid, though it yielded some phthalic anhydride on treatment with acetyl chloride and gave the fluorescein reaction. The aqueous mother-liquor on extraction with ether yielded 4.2 g. of nearly pure benzoic acid (equiv. found, 122.9; calc., 122).

In subsequent experiments more elaborate fractionation yielded a small quantity of liquid boiling at 186—189°, and 4.6 g. of this were submitted to oxidation as before. From the product there was isolated some pure terephthalic acid, the diethyl ester of which had m. p. 138—139°, both alone and mixed with authentic diethyl terephthalate. Based on the different solubilities of the acids in water, ether, and chloroform respectively, an estimate of the amounts of oxidation products gave: benzoic acid, 3.46 g.; phthalic acid, rather less than 0.18 g.; terephthalic acid, at least 0.18 g.

The authors infer that when magnesium benzyl chloride acts on monochlorodimethyl ether, a little ω -methoxy-*o*-xylene is undoubtedly formed, but this is accompanied by ω -methoxy-*p*-xylene and a very much larger quantity of β -phenylethyl methyl ether, $C_6H_5 \cdot CH_2 \cdot CH_2 \cdot O \cdot CH_3$, as well as other products of higher boiling point.

Magnesium benzyl chloride, when condensed with acetone and acetaldehyde respectively, gives products which when oxidised by permanganate yield no traces of phthalic acid or of any aromatic acid other than benzoic acid. The behaviour of monochlorodimethyl ether is therefore, in one respect at least, different from that of an acyl chloride which here corresponds more closely in compartment with a true carbonyl compound.—THE UNIVERSITY, MANCHESTER. [*Received, July 11th, 1930.*]
