XIX.—*Note on the Preparation of Glycol.*

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IN preparing glycol by the usual method, namely, digesting ethylene dibromide with potassium carbonate solution, evaporating the product nearly to dryness, and extracting the glycol by means of a mixture of ether and alcohol, a very small yield only is obtained, due principally to the volatility of the glycol with steam, and consequent loss during the evaporation.

As we required a considerable quantity of glycol for some synthetical work, we made many experiments with the object of

improving the method of preparation, and found that by the following simple modifications of the usual process, the yield may be greatly increased.

In the first place, potassium carbonate (138 grams) is dissolved in water (1 litre), and the solution boiled in a stout, round-bottomed flask with ethylene dibromide (188 grams) in a reflux apparatus, from the top of which a glass tube leads to a couple of wash bottles containing bromine. When almost all the oily drops have disappeared (which is usually the case after 8—10 hours), the same quantities of potassium carbonate and ethylene dibromide are again added to the solution, and the boiling continued as before; the operation being repeated until 1128 grams of ethylene dibromide have been decomposed.

After the third addition of ethylene dibromide, crystals of potassium bromide separate on standing over night. These (and those which separate after each succeeding operation) are removed in the morning, by filtration on a vacuum pump, before the action is again started. The crystals are then washed with absolute methylated spirit, the washings being subsequently used for the isolation of the glycol, as explained below. After the decomposition of the ethylene dibromide is complete, the solution of glycol is heated in an oil bath to slowly distil off the water, using a colonna to prevent, as far as possible, loss of glycol by evaporation.

When the distillation has continued some time, the liquid begins to bump violently, owing to the separation of potassium bromide. The solution is now cooled, the crystals of potassium bromide removed, as before, and the distillation then continued. All the water which distils over should be carefully preserved for use in a subsequent preparation. When the solution becomes very viscid, and the temperature of the vapour passing over begins to rise, the distillation is stopped, and the residue is mixed with the methylated spirit employed in washing the potassium bromide crystals, as explained above. After standing for some time, the crystals of potassium bromide, which separate in quantity, are removed by filtration on the pump, washed with absolute methylated spirit, and the combined alcoholic extracts concentrated by slow distillation, as before, from a flask fitted with a colonna.

The residue is then again treated with absolute methylated spirit, which separates more potassium bromide; this treatment is repeated, now using a mixture of alcohol and ether, until almost the whole of the potassium bromide has been removed.

The solvent is then removed by distillation, and the residual glycol fractionated, first under reduced pressure, and finally at the ordinary pressure.

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The yield of glycol obtained in this way is about 50—60 per cent. of the theoretical, and we have found it practicable in one apparatus to prepare 1 kilo. of glycol in about 10—14 days.

During the action, considerable quantities of vinylic bromide are formed; this is absorbed by the bromine in the wash bottles, and by subsequently treating the product with dilute potash, to remove the excess of bromine, and fractionation, tribromethylene, $\text{CH}_2\text{Br}\cdot\text{CHBr}_2$, is readily obtained in a pure state.

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