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Organic Chemistry
Laboratory

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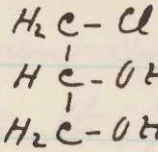
Jan 1881

Monochlorhydrine

$C_3H_5Cl(OH)_2$



References. Berthelot Chimie Organ. 272 p.
Schorlemmer. Chem. of Carbn Compds. 254 p.
Wurtz Dictionnaire de Chimie Vol I P2. p 15-80
Hellot's Chimie Vol II p 128 §1243
Watt's Dictionary of Chemistry Vol I. p 893.
Gmelin's Chemistry Vol IX p 498.

The method employed is that given by Berthelot -
Hydrochloric acid with addition of some sulphuric - was placed in a flask with a safety tube - in order to dry the HCl gas. Before passing into the glycerine - it was passed through a drying jar filled with pumice soaked with H_2SO_4(conc). A small, empty jar was also placed between the drying jar and the flask containing HCl to prevent any large amount of water passing into the drying jar. The flask containg the glycerine was warmed so that the excess of HCl would pass out of the laboratory. The glycerine was gently heated during the operation.

Dry HCl was passed through the glycerin until it was completely saturated - (about 6-8 hours) The glycerin turned brown during the process - After complete saturation the flask containing the glycerin was placed in a steam bath and kept at a temperature of 100° C for between 36 and 40 hours. The glycerin became darker and had a peculiar odor resulting like Liquorice -

In order to remove the excess of HCl in the liquid Soda crystals (carbonate of Soda) was added to it until it gave a neutral reaction with most litmus (It was then agitated with ether and after standing for some time - the ethereal solution of monochlorhydrin - separated in a separating funnel - and distilled to remove the ether: which was used a number of times. The process of Etherification was very slow and only a small quantity went into Solution -

The residue after complete of ether was then distilled in a retort with a very low heat - a thermometer being inserted in the retort.

The first products of the distillation was apparently some water. ether and a light yellow oily substance. When a temperature of about 200° was reached a brownish substance passed over and fumes having the odor acrolein. At high temperature more passed over - and finally at 235° a dense smoke filled the body of the retort. After awhile more came over a carbonaceous mass remained in the retort - and in the neck a dark brown oily liquid - with an intensely pungent odor - like a mixture of pyroligneous acid and acrolein - probably not monochlorhydrin -

The reaction by Ebel was not complete as also the ether dissolved some glycerin which was decomposed at the high temperature necessary.

Monochlorhydrin boils at 225-230° and has a fresh ethereal odor which this entirely did not have.

Dichlorhydrine

$C_3H_6Cl_2(HO)$. B.P. 178° C.

References

Wurtz Dictionnaire de Chimie Vol I Part-2 p 15-80.
Watts Dictionary of Chemistry Vol 1. p 893.
Schorlemmer Chem of Carbon Compds. p. 25-4.
Berthelot's Chimie Organ. p 273.
Kekulé's Chemie Vol II p 128 § 1243.
Gmelin's Chemistry Vol IX p 498.

The method employed is that given by Berthelot. p 273. § 14.

The same apparatus was employed as in the synthesis of monochlorhydrine - for drying dried HCl gas.

Equal parts of Glycerine and of glacial acetic acid were placed in a flask. through which dry HCl gas was passed until completely saturated.

Not completed when laboratory closed.

Formic Acid

CH_2O_2 $CHO.OH$ $O=\underset{H}{C}-OH$

References. Berthellot's Chimie Organ. 62. 439-443 p.
Schorlemmer Chem. of Carbon Cmpds. 104 p.
Wurtz. Dictionnaire de Chimie Vol I Part 2. p. 1490.
Kekule's Chemie Vol I p. 540

The method employed is that given by Wurtz to obtain 56% acid - using a mixture of oxalic acid and glycerine without the addition of water

One half a kilo of glycerine and an equal amount of oxalic acid were placed in a flask holding about two litres - connected with a condenser. In the flask a thermometer was placed: the bulb being in the liquid - When on heating gently the temperature

60° C. CO_2 was evolved the action gradually increasing as the temperature rose to 98°. At this temperature a turbid liquid was condensed - having a pungent and peculiarly disagreeable odor & giving acid reaction with litmus paper. At 105° the distillate was clearer with a strong acid reaction. The temperature was gradually increased to 120°. and when no more distilled at this temperature and the evolution of gas had apparently ceased - another half kilo of oxalic acid was added to the glycerin remaining in the flask - The temperature reached as high as 135° this time. The best results were obtained at about 120°. The solution gas ran steadily but very slowly at this temperature

The acid obtained was not a clear liquid but contained white flakes ~~as~~ of something had been carried over mechanically. It was accordingly filtered and redistilled when a clear liquid was obtained - The glycerine which remained in the flask was not pure but of a brown color ~~and~~ contained solid impurities

A portion of the acid obtained in this manner was heated to boiling and neutralized with Carbonate of lead. (which should be freshly precipitated) The solution was filtred to remove any excess of lead salt and after evaporation for a short time allowed to cool. An curling formate of lead crystallized out in slender prismatic white crystals.
The operation must be carried on when there is no trace of sulphuretted hydrogen. as this formate will be decomposed and blacken.
This formate of lead is soluble in hot but not in cold water.. The theoretical composition of the salt is as follows- formic acid dry monobasic

$C_2H_2O_4Pb$.

C. 8.08%

H. .67

O. 21.55

Pb. 69.70

100

An analysis of the salt was made and the amount of lead found was. 69.57%.

Analysis of Formate of Lead - Determination of Pb.

About 2 grms of the salt was placed in a number five crucible and a very little more than the amount of sulphuric acid found by calculation to be necessary to convert it to $PbSO_4$ added. This was carefully evaporated to dryness and finally strongly heated to expel any excess of H_2SO_4. weighed and calculated Pb as $PbSO_4$.

The amount of acid necessary is for each gramme of formate of lead - .20 + cc of acid of a Sp. G of 1.82

The sulphate of lead obtained was perfectly white