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Alcohol.

From "filler washings" I distilled 1250 c.c. of C_2H_5OH . To this I added 1250 c.c. from the laboratory and distilled as follows:-

Oct. 31. 165 c.c. at 78.5°

Nov. 1. 250 " 78.5°

4. 550 " 78.5°

5 650 $78.5 - 78.8$

6 560 $78.5 - 78.8$

2175 c.c.

Sp. gr. of this product at 15° was .822. To 2050 c.c., of this, in a bottle was added 550 grms. of finely powdered CaO and allowed to stand about ten days shaking once or twice each day. The calcium hydrate mixed with the alcohol making a thick liquid; this was poured into a dry flask and distilled in a water

bath. The product thus obtained had a molecular refracting power of 0.4578 corresponding to 99.2 per cent. of C_2H_5OH .

For the first two distillations a column with seven partitions was used.

Ethyl Bromide.

Used 50 grms amorphous phosphorus, 375 c.c. ethyl alcohol of about 0.8 sp. gr., and 100 c.c. bromine. The alcohol and phosphorus were placed in a litre flask connected with a separator and a ^{reversed} condenser. By means of the separator the bromine was slowly dropped into the flask. At first the flask was surrounded with ice water but when the violent action ceased it was gently warmed to complete the reaction. Allowed to stand over night.

The flask containing the product of the above reaction was connected with a flask containing dilute KOH solution by means of a tube which dipped below the surface

of the KOH solution and the later flask connected with a condenser. Both flasks heated in a water bath. The temperature of the vapor in the first was about 45° and the KOH solution was maintained at about 60° . This distillate was redistilled using a three bulb Hertz tube and gave 125 cc of a turbid liquid boiling at 36° - 37° . The liquid remaining in the flask after the first distillation was filtered to remove the phosphorus and distilled using a Hertz tube. After collecting 60 cc from 38.7° - 38.9° the boiling point rapidly increased to 81° - 82.5° and alcohol came over. The 125 cc. boiling at 36° - 37° was added to the 60 cc. boiling at

38.7° - 38.9° and mixed with an equal volume of strong sulphuric acid, allowed to stand over night and then separated by drawing off the acid. Washed well with water to remove all traces of the acid and to remove the water distilled over CaCl_2 . This process gave about 120 c.c. of colorless ethyl bromide having a sp. gr. of 1.463.

Ethyl Iodide.

Used 250 grams finely powdered iodine, 25 grams amorphous phosphorus, and 156 c.c. ethyl alcohol of 0.82 sp. gr. These materials were placed in a flask connected with a reversed condenser and boiled for six hours. Then the liquid in the flask was filtered, litharge added and distilled. The product was colored with iodine; to remove this was washed with dilute KOH solution then with water and distilled over CaCl_2 . Product only 25 c.c. A part of this loss was due to the breaking of a flask.

With the follow process I obtained 140 c.c. of colorless $\text{C}_2\text{H}_5\text{I}$ from 200 grams iodine, 20 grams. amor-

phos phosphorus and 200 g.c.
ethyl alcohol.

The phosphorus was placed in a flask and the iodine in an adaptor placed above the flask. The alcohol was poured into the flask through the iodine and then an inverted condenser was connected with the adaptor and the contents of the flask boiled for ten hours. In this way I was able to get all my iodine into solution. Then distilled off nearly all the liquid and when the residue commenced to thicken a crystalline substance about the color of bichromate of potassium was deposited on the neck of the flask. The product of this distillation contained free iodine; to remove this I washed with dilute KOH solu-

tion and then with water using a large amount of water to precipitate any iodide of ethyl that might be dissolved in the unconverted alcohol. On standing the ethyl iodide separated as a heavy white liquid this was drawn off a little litharge added and distilled over CaCl_2 .

In washing with KOH solution care should be taken not to use an excess.

Acetyl Chloride.

Used 280 grams phosphorus pentachloride and 78 c.c. glacial acetic acid. Phosphorus pentachloride was placed in a litre flask, surrounded with cold water, connected with a separator and a reversed condenser. By means of the separator the acid was slowly dropped into the phosphorus pentachloride. When the reaction was finished the separator and condenser removed and the contents of the flask distilled using a three bulb Hurty tube. A black residue was left on the bottom and sides of the flask.

Product about 100 c.c. of colorless strongly fuming acetyl chloride

Ethyl Amines.

Used two tubes of hard glass of about 80 c.c. capacity. 25 c.c. ethyl iodide and 43 c.c. strong NH_4OH solution was poured into each tube. Then the tubes were closed in lamp flame and laid inside iron pipes to prevent the glass from flying in case of explosion. The tubes were surrounded with boiling water for ~~three~~ hours or until nearly all the ethyl iodide had disappeared.

When the tubes were cool the ends were broken and contents, poured into a porcelain dish, evaporated to dryness and dried at 110° . This gave a brown solid mass. Treated this with 99% alcohol and

filtered off the insoluble tetra-ethyl ammonium iodide. Evaporated the filtrate to dryness dried at 110° and transferred the solid thus obtained to a flask added strong KOH solution and distilled below 90° .

Product about 20 c.c. of mixed ethyl amines.

Zinc Ethyl.

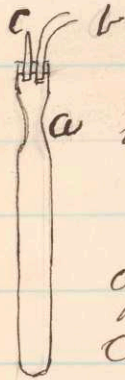
65 c.c. ethyl iodide and about 20 grams of clean bright zinc chips were placed in a 500 c.c. flask connected with a reversed condenser and the open end of the condenser was connected with a tube dipping below the surface of mercury.

The apparatus was filled with dry carbonic acid and the contents of the flask boiled for six hours. If zinc ethide free from ethyl iodide is required it is necessary to continue the boiling until the ethyl iodide ceases to flow back from the condenser. During the boiling a small quantity of gas was given off at the surface of the mercury. This

gas burnt with a pale flame and was probably butane.

Then the flask was allowed to cool and the contents became nearly solid; the position of the condenser changed and the zinc ethide distilled off in a current of dry carbonic acid.

The arrangement figured below was used to collect the zinc ethide



The tube was about 150 m.m. in diameter and was drawn out at a to about 3 m.m. The opening was closed with a cork and through this passed the end of the condenser b and also a tube c drawn out to a fine point to admit the exit of the carbonic acid. When all the zinc ethide had distilled over the tube was heated at a

and the upper part drawn off.
Also used zinc dust and an
alloy of zinc and sodium but
neither of these answered as
well as the clean bright zinc
chips.

Chloral.

About 90 c.c. ethyl alcohol were placed in a baryta tube resting in a water trough. Dry chlorine was passed through the alcohol until it assumed a permanent yellow color. Then the water was heated to about 90° by blowing in steam and the treatment with chlorine continued until a small portion of the liquid on being agitated with six times its volume of concentrated sulphuric acid gave a layer, on standing, of an oily liquid. Then the liquid in the baryta tube was emptied into a small flask and this connected with the baryta tube so that the chlorine passed through both tube and flask

and the operation continued as before except the water in the trough was heated at the beginning of the operation to about 40° and gradually increased to about 90° . I found that the conversion took place much faster in the second case.

From time to time as the liquid decreased in bulk more alcohol was added.

150 c.c. of a thick yellow liquid thus produced was mixed with four times its bulk of concentrate sulphuric acid and allowed to stand for about three days. The result of this was a cake, about 3 in. in diameter and $\frac{1}{4}$ in. thick, of a white solid. This was separated from the acid as much as

possible from the acid and divided into two equal portions. One half was mixed with CaCl_2 and distilled; this gave a dark colored liquid which when redistilled became colorless. I took this to be pure chloral and added water to form the hydrate but obtained no crystals. Then tried remove the water by distilling over CaCl_2 and found that there was so much water present that this was impracticable so mixed with six times its volume of concentrated sulphuric acid and allowed to stand over night. The result was that I obtained a layer of insoluble chloral and a few crystals of chloral hydrate were sublimed on the sides of the beaker

Trichloroacetic Acid.

Took half the first solid product obtained in making chloral placed it in a flask added fuming nitric acid and gently warmed until all the solid disappeared. Then distilled from a retort; toward the end of the operation the residue blackened probably due to sulphuric acid. The distillate was partially neutralized with sodic carbonate and again distilled. After this operation had been performed three times the distillate appeared to be free of nitric and nitrous acids and had strong penetrating odor. This product was probably a water solution of trichloroacetic acid. Owing to the want of time this product was not further

purified.

During the second distillation
a gas, which burnt with a pale
flame, was given off at the
mouth of the receiver.