

GLAMORGAN  
EDUCATION COMMITTEE

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Subject *Chemistry*

E. J. Arnold & Son Ltd. Leeds

### Tests and Properties

1. It is a colourless, odourless, tasteless gas.
2. When the jar of the gas is ignited a gas which turned lime water milky, carbon dioxide, and water, which condensed on the sides of the jar was given off. Methane does not affect lime-water.

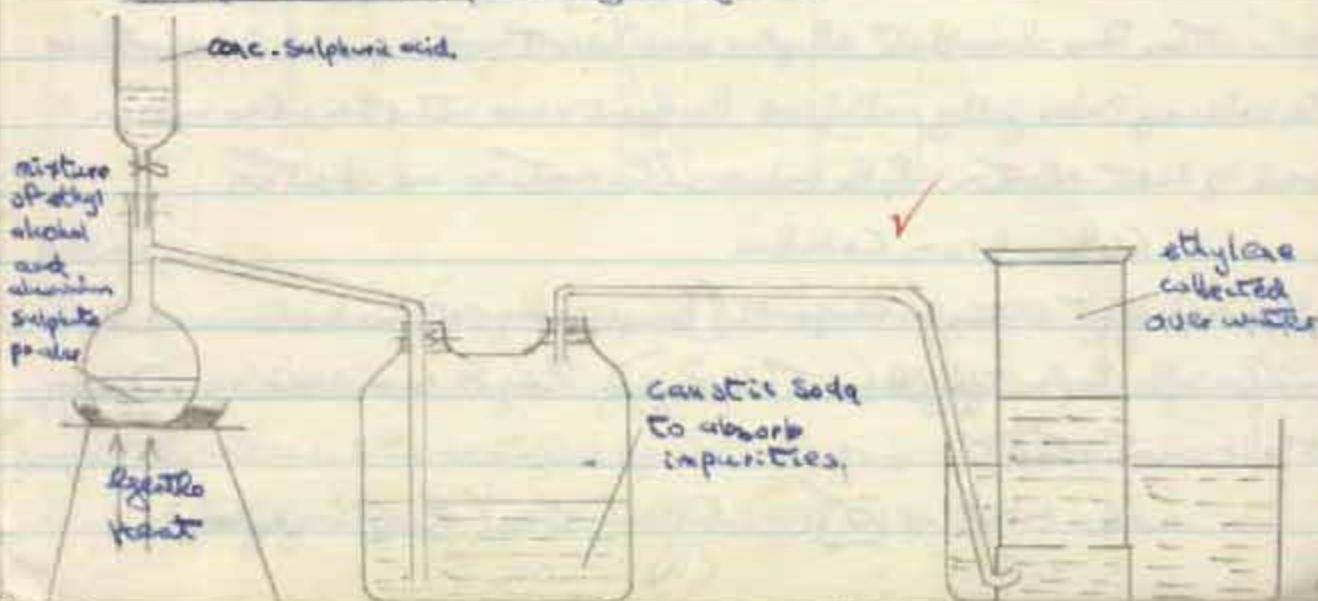


3. There is no action with brown water. But with bromine vapour a substitution reaction set up with ~~hydrogen~~ hydrogen bromide and methyl bromide is formed.



4. There is no action with alkalies, yet sodium permanganate, a strong oxidising agent, showing that methane is a saturated gas, i.e. has its full complement of hydrogen atoms. Slightly reactions in which it takes place are complete oxidation to carbon dioxide and water and substitution.

### The Preparation and Properties of Ethylene.



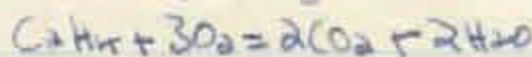


### Method.

Some aluminium sulphate powder and 30 c.c. of ethyl alcohol were put into a distilling flask and the apparatus was assembled as shown in the diagram. 80 c.c. of conc. sulphuric acid was slowly added to the mixture in the flask. The flask was slowly and gently heated and the ethylene gas evolved passed into caustic soda to absorb impurities such as sulphur dioxide gas and was then collected over water. Sulphuric acid, the dehydrating agent, may be regarded theoretically as a catalyst, because it undergoes no permanent chemical change.

### Results.

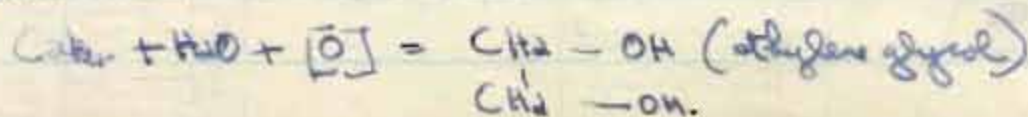
1. It is a colorless, odorless gas.
2. It has no effect on lime water, but when it is ignited carbon dioxide gas, which turns lime water milky, and steam, which condenses on the sides of the gas jar are given off. It burns explosively with a luminous flame.



3. Bromine water is decolorized by the gas forming ethylene dibromide with direct combination. This shows that ethylene is an unsaturated gas which does not have its valency links fully satisfied. Therefore it is more active than alkanes and can react by direct combination with the halogens. Its reactions are additive.



Alkaline potassium permanganate (Bayer's reagent) is decolorized by the gas forming ethylene glycol (antifreeze). The gas is oxidized again along with the unsaturation.



### Equations



### The Preparation

water.

distilling flask containing caustic soda

carbide powder

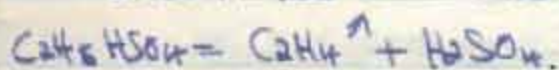
### Method.

Some

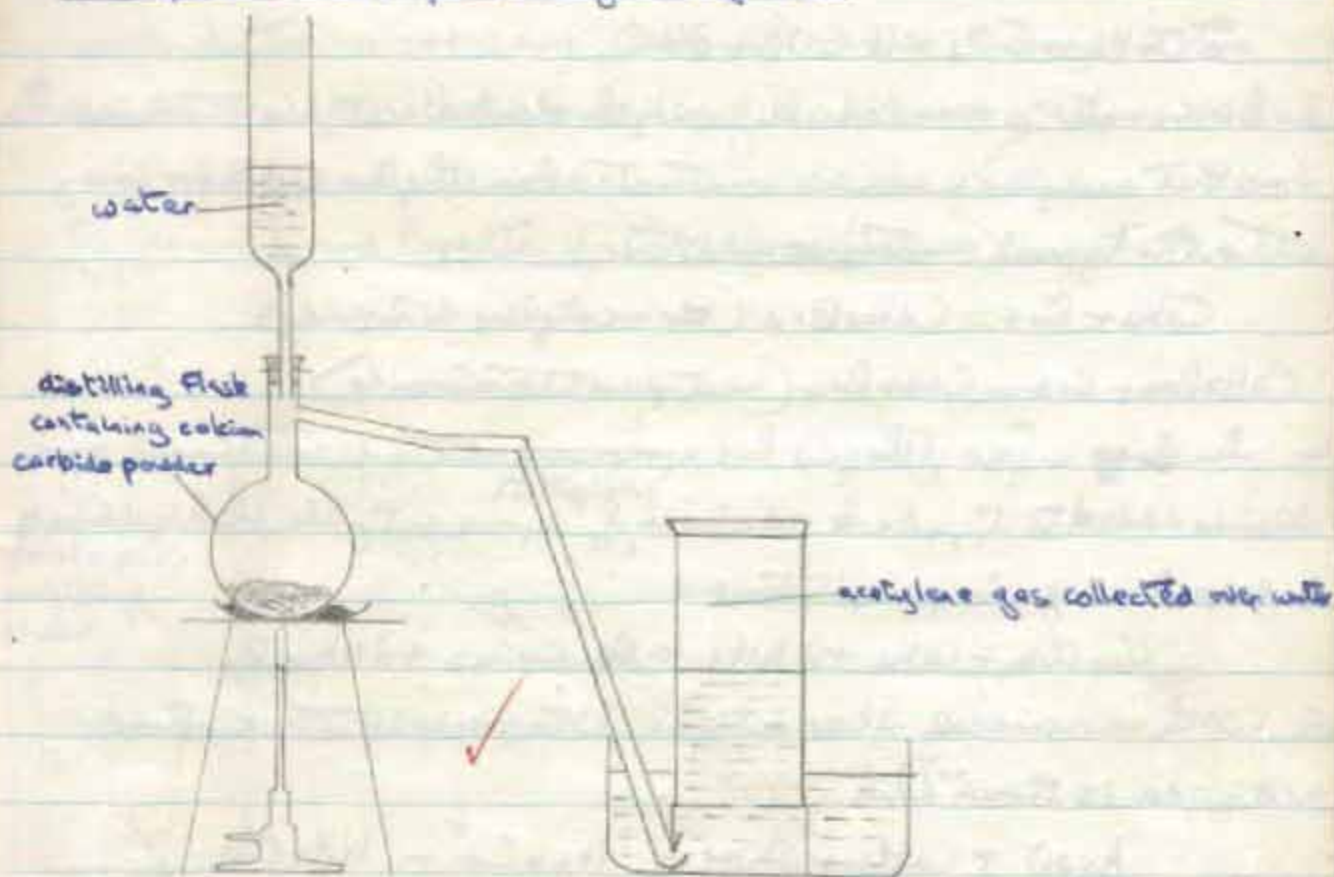
apparatus was drop into the

C

### Equations

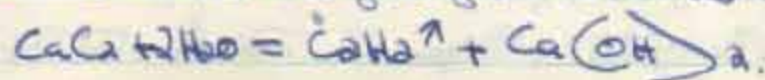


### The Preparation and Properties of Acetylene.



### Method.

Some calcium carbide powder was put into the distilling flask with apparatus inserted as in the diagram. Some ~~disposable~~ water was allowed to drop into the carbide and the acetylene gas collected over water.





## Tests and Properties

1. It is a colorless gas with a sweetish smell.
2. It has no effect on lime water but when ignited it burns explosively with a luminous flame forming carbon dioxide gas which turns lime water milky, and steam.



3. Bromine water is decolorized, forming the colorless acetylene tetrabromide. This shows that acetylene is even more unsaturated than ethylene and therefore is very active. Its typical reaction is additive.



4. When ~~the~~ a jar of the gas has a prepared solution of ammoniacal cuprous chloride added to it, a dark red-brown <sup>precipitate</sup> of cuprous acetylide, which is deeply explosive when dry, is precipitated.



5. With ammoniacal silver oxide white precipitate of silver acetylide is thrown down.



## Preparation of ammoniacal cuprous chloride.

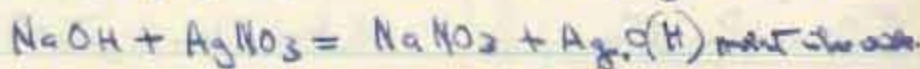
A portion of copper carbonate powder was dissolved in concentrated hydrochloric acid in a test tube. A piece of copper was added to the solution and the solution boiled for a few minutes. The mixture was then poured into another test tube half

full of water. It was decanted and the white solid, cuprous chloride, dissolved in ammonia solution.

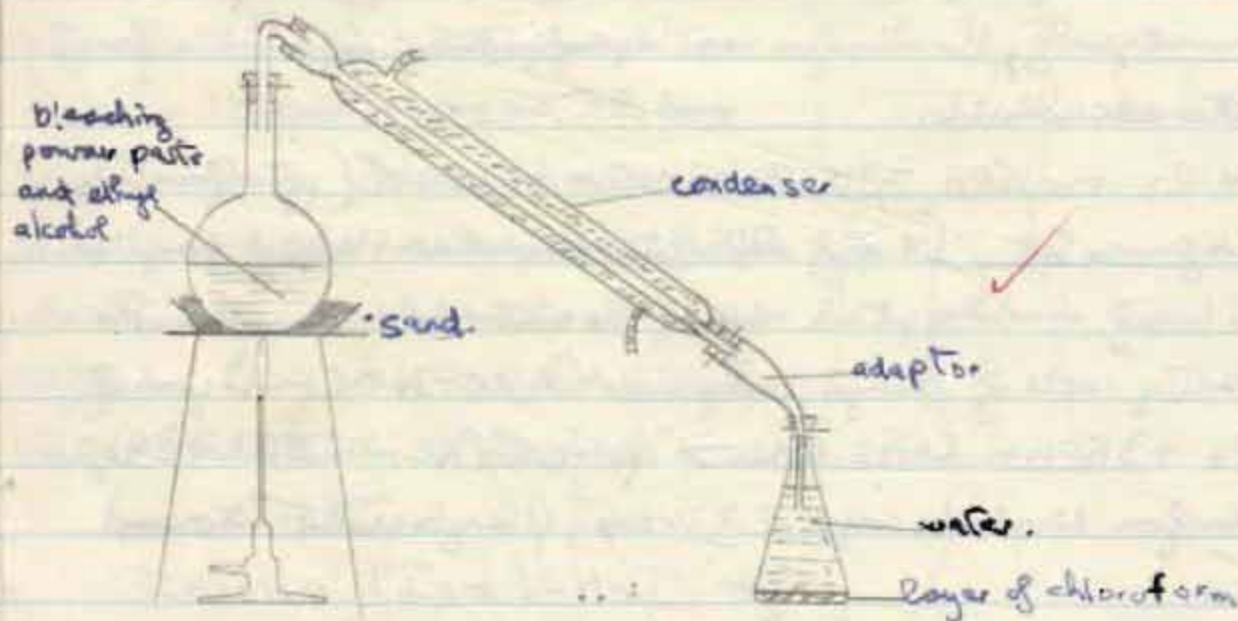


### b) The Preparation of Ammoniacal Silver Oxide.

Excess cuprous oxide solution was added to a test tube full of solution of silver nitrate to precipitate the oxide. The liquid was decanted and ammonium hydroxide solution was added drop by drop with constant shaking until the precipitate dissolved.



### The Preparation and Properties of Chloroform. ( $\text{CHCl}_3$ )



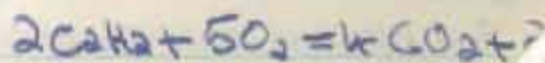
#### Method

A paste consisting of 50 gms of bleaching powder and 150 cc. of water was mixed in a pestle and mortar. The paste was transferred to a flask and 200 cc. of



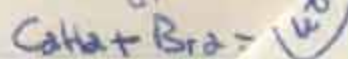
## Tests and Properties

1. It is a colourless gas with a sweet odour.
2. It has no effect on lime water but when ignited it gives a luminous flame forming carbon dioxide gas.



3. Bromine water is decolorised

Shows that acetylcholine is even  
active. It's typical range



4. Wanted  
chloride  
acid

ant 5 drops of absolute ethanol added. The mixture is warmed gently. The  
boiling point of phenyl isocyanide  $C_6H_5-N=C$  was given off.



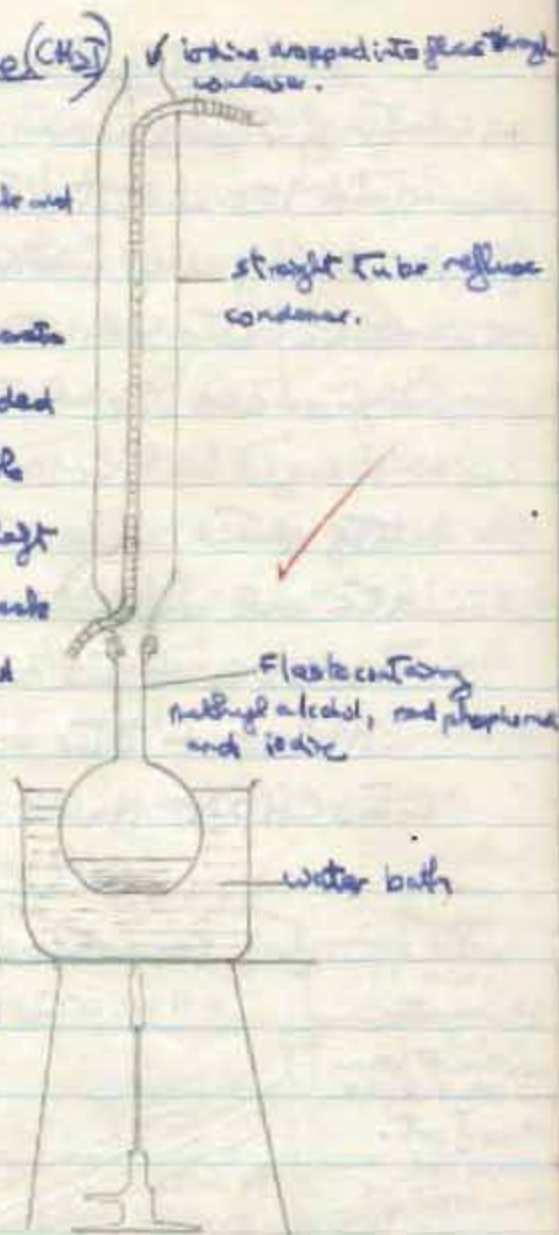
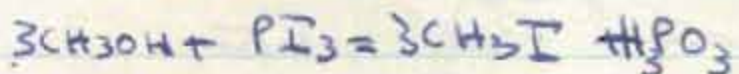
4. Chloroform dissolved a crystal of iodine, giving a violet coloured solution.



## The Laboratory Preparation of Methyl Iodide ( $\text{CH}_3\text{I}$ )

### Procedure

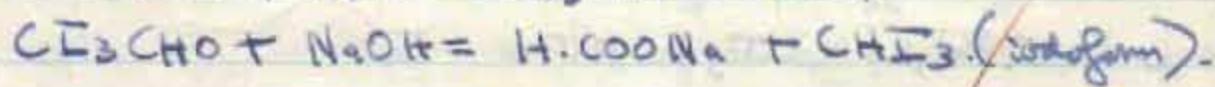
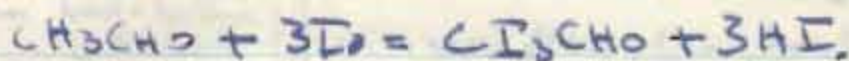
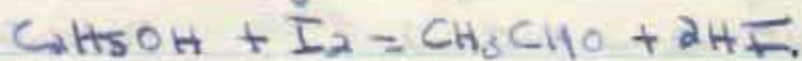
25 cc of methyl alcohol was put into a dry flask and the flask clamped into a water bath. 3 gms of red phosphorus was added to the alcohol and the apparatus assembled as the diagram. 30 gms of iodine was added over a period of 1/2 hr while heating the water bath. The water bath was then heated for further 1/2 hr and then left overnight. It was then transferred to a distilling flask and distilled. The only distillate was collected and placed in a separating funnel, run off, and the lower layer kept. Then, in a separating funnel it was shaken up with carbon tetrachloride and the lower layer - no longer colored with excess iodine. It was run off and the lower layer kept. Red phosphorus and calcium chloride was added and the methyl iodide left to dry. It was redistilled, the distillate being collected between  $43^\circ\text{C}$  and  $46^\circ\text{C}$ .





### The Laboratory Preparation of Iodoform ( $\text{CHI}_3$ )

a solution of 5 gms of sodium carbonate in 50 c.c. of distilled water was prepared and 10 c.c. of it put aside. To the 40 c.c. of solution the flask 7 c.c. of ethyl alcohol was added. The flask containing the solution was immersed in a water bath at  $60^\circ\text{C}$  and 2.5 gms of iodine were gradually added. The solution is turned into a brown. The colour was discharged with the 10 c.c. of sodium carbonate kept back. The resulting solution was heated for a few minutes at  $70^\circ\text{C}$ . Then allowed to cool and filtered.



### The Laboratory Preparation of Ethyl Bromide.



Method.

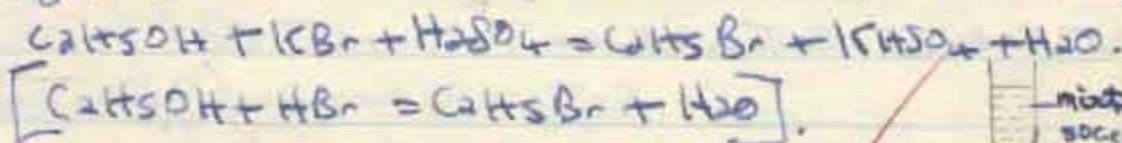
The mixture is placed in a round-bottom flask and a layer of sodium carbonate is added and the mixture is separated and the lower solution is the resulting for heating.

The preparation of Ethyl Bromide.



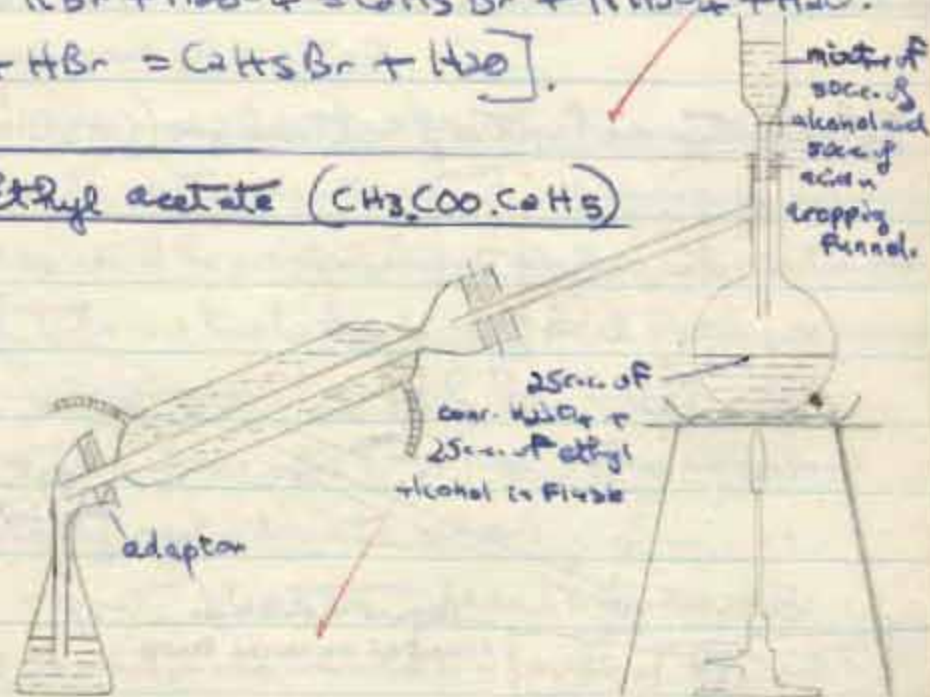
### Method.

The oily distillate was collected after distillation (as in diagram) and placed in separating funnel. It was run off and the lower layer kept, the upper layer discarded. The lower layer was returned to the funnel and shaken up with sodium carbonate solution to neutralize the excess acid. The solution was again run off and the lower layer kept. It was returned a third time to the separating funnel and shaken up with water. The solution was run off and the lower layer kept. Anhydrous calcium chloride was added and the solution left overnight to get rid of excess alcohol as  $C_2H_5O_2$  and  $CaH_2SO_4$ . The resulting liquid was redistilled using a thermometer and a water bath for heating purposes and the distillate collected between  $35^\circ C$  and  $40^\circ C$ .

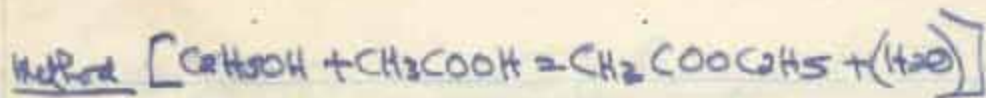


### The preparation of Ethyl acetate ( $CH_3COO.C_2H_5$ )

#### Reaction.

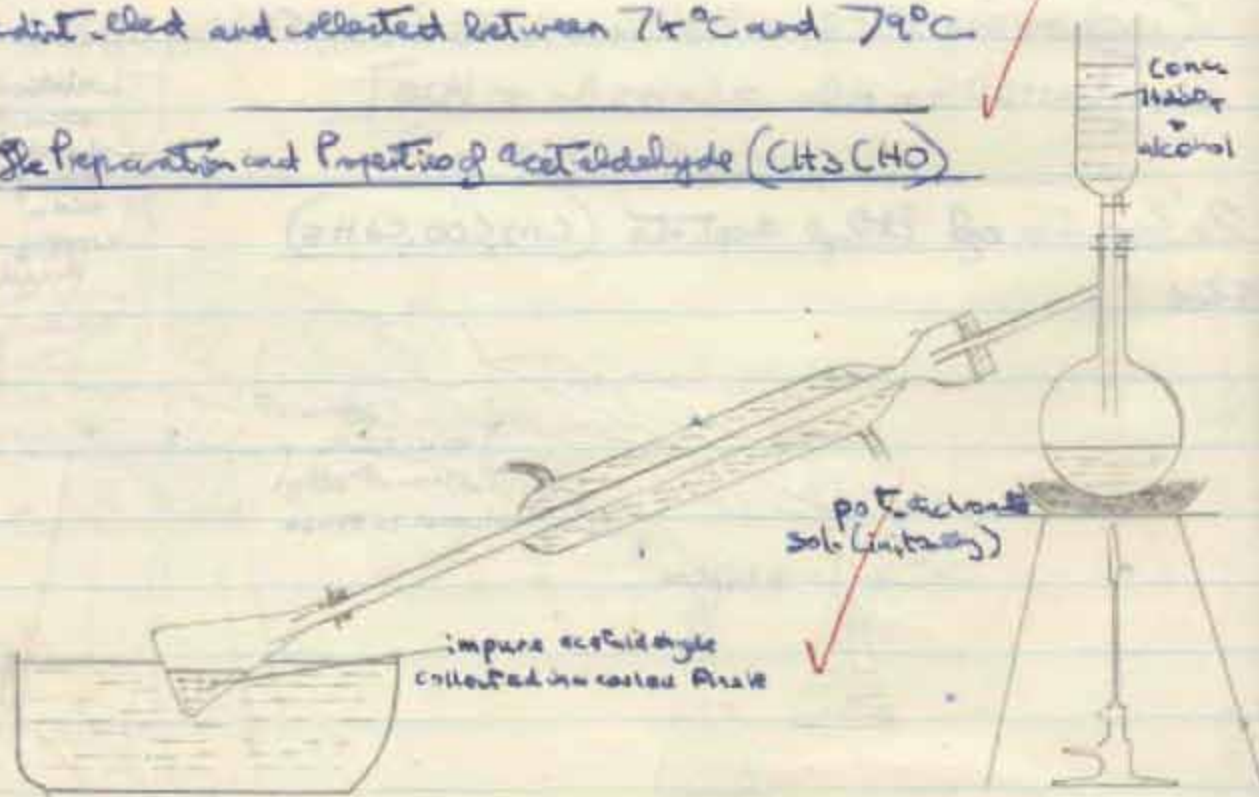






a 25c.c. of conc. sulphuric acid was added to 25c.c. of ethyl alcohol and the mixture cooled and poured into the distilling flask. 50c.c. of alcohol and 50c.c. of glacial acetic acid were mixed together and put into the dropping funnel. The distilling flask is heated to about  $140^\circ C$ , and, keeping the temperature constant, at the same time the mixture in the funnel was dropped at the same rate so the final product distills over. The distillate is transferred to a separating funnel and shaken with strong sodium carbonate solution until all further action. The upper layer is kept and anhydrous calcium chloride added to remove excess alcohol as  $CaCl_2$  and  $C_2H_5OH$ . The upper layer was kept and anhydrous calcium chloride added. It was left to dry and redistilled and collected between  $74^\circ C$  and  $79^\circ C$ .

### The Preparation and Properties of Acetaldehyde ( $CH_3CHO$ )



Method.

50gms of  
flasks - a  
was transfe  
temperature  
The mixture  
dist. water  
alcohol, and  
of acetic  
 $CH_3CHO$   
 $Na_2Cr_2O_7$   
Properties  
1) Reducing  
Excess  
precipitate  
was added  
put into a  
The result  
Reaction  
also with  
2) Soluble  
acetalde  
after heat

The  
mixture  
of a  
50g  
brown  
solid  
sand



### Method.

50gms of pot. perm. dichromate were dissolved in 75c.c. of water in a distilling flask. A mixture of 20c.c. of concentrated sulphuric acid and 10c.c. of ethyl alcohol was transferred to a dropping funnel. The flask was heated to a moderate temperature and ~~the~~ the mixture was allowed to run in from the funnel slowly. The action was allowed to moderate then the flask was heated gradually. The distillate was collected in a cooled flask. The product is a mixture of acetaldehyde, alcohol, and water. [Pure acetaldehyde may be prepared by forming crystals of acetaldehyde-ammonia and regenerating pure acetaldehyde from the]  
$$\text{CH}_3\text{CHO} + \text{H}_2\text{O} = \text{CH}_3\text{CH}_2\text{OH} \quad (0) = \text{CH}_3\text{CHO} (\text{acetaldehyde}) + \text{H}_2\text{O}.$$
$$\text{Na}_2\text{Cr}_2\text{O}_7 + 4\text{H}_2\text{SO}_4 + 3\text{C}_2\text{H}_5\text{OH} = 2\text{Na}_2\text{SO}_4 + \text{Cr}_2(\text{SO}_4)_3 + 7\text{H}_2\text{O} + 3\text{CH}_3\text{CHO}$$

### Properties and Tests.

#### 1) Reducing action of Ammoniacal silver nitrate

Excess of caustic soda was added to silver nitrate in a test tube to precipitate silver oxide. The mixture was decanted and ammonium hydroxide was added drop by drop until the precipitate just dissolves. The mixture was put into a clean test tube and two to three drops of acetaldehyde added. The resulting solution was warmed in a beaker of hot water. After heating traces of metallic silver were to be seen. Thus ammoniacal silver nitrate was reduced by acetaldehyde to silver.

2) Fehling's solution was put into a test tube and a few drops of acetaldehyde added. The solution was warmed in a beaker of hot water. After heating a red precipitate of cuprous oxide was to be seen at the bottom.

Conn  
Haber  
alcohol

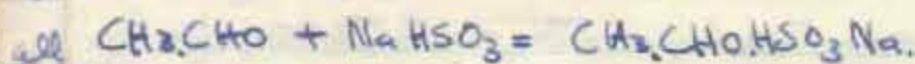
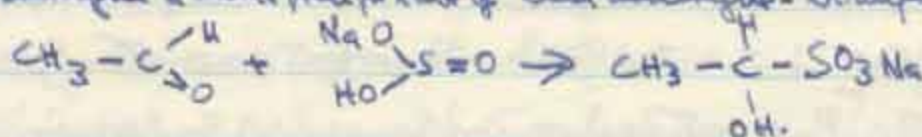


of the test tube. Thus acetaldehyde reduces Fehling's solution to give a precipitate of cuprous oxide.

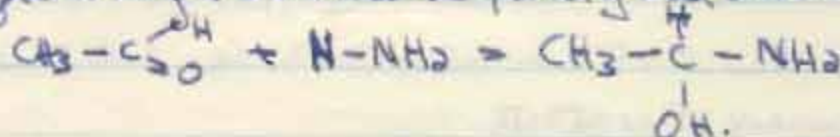
In the above reaction acetaldehyde is oxidised to acetic acid.



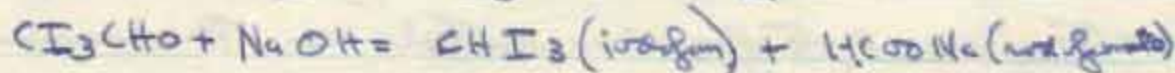
3) When a strong solution of sodium bisulphite was shaken up with acetaldehyde a white precipitate of acetaldehyde-bisulphite was formed.



4) With strong ammonia solution in a test tube acetaldehyde gave a white precipitate after cooling a colourless solution of acetaldehyde-ammonia was formed.



5) Iodoform Test. When acetaldehyde was added to iodine solution, alkali added, and the solution warmed, yellow traces of iodoform could be seen around the test tube.



The above is a substitution reaction of acetaldehyde.

6) Schiff's reagent was turned violet. Acetaldehyde restores a colour to magenta which has been bleached with sulphur dioxide.



Reaction To give

acid.

up with.

to be formed.

to give a  
reaction is formed.

is a solution,  
iodoform

(red fumes)

to give a colour to

### Hydrolysis or Saponification of Ethyl acetate

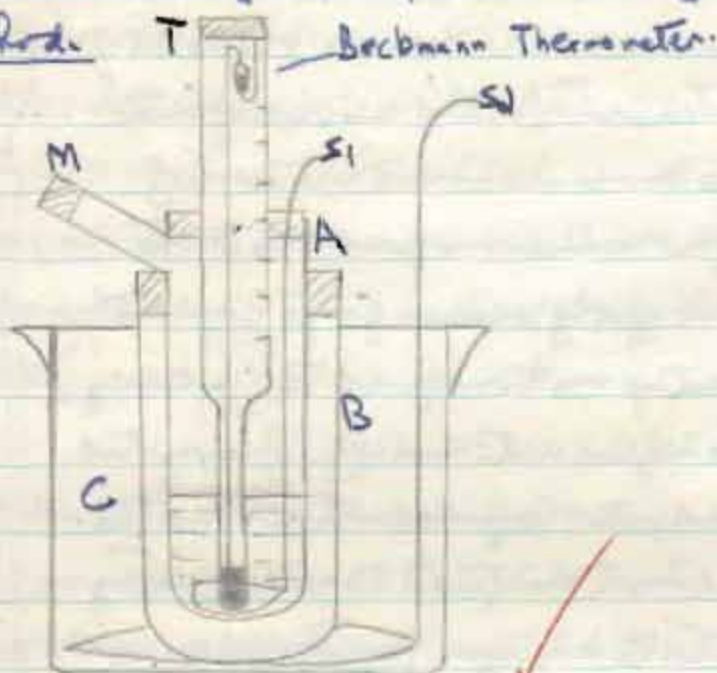


15 gm of caustic soda was dissolved in 40 cc. of water in a flask. Some glass beads were added and 20 cc. of ethyl acetate. A reflux condenser was fixed to the flask and the mixture boiled gently on a gauze for about 30-40 mins. The mixture was transferred to a distilling flask and half the liquid distilled off. This consisted of ethyl alcohol and water. Confirmed with iodoform test. The remaining liquid was transferred to an evaporating dish and evaporated to dryness. The solid left was powdered and distilled with concentrated sulphuric acid.





To Find the molecular weight of Naphthalene in Benzene by the Freezing Point Method.



### Method

A contains the weighed solvent with the bulb of the thermometer and a stirrer and has a side-arm M through which the solvent is introduced. A is surrounded by a water tube B which provides air insulation to ensure uniform cooling. The freezing agent in C was a mixture of ice and salt stirred by S2.

The F.P. of the pure solvent was first measured. A weighed amount of solvent is put in A and both A and C well stirred. Supercooling often occurs but when the solvent began to separate out the temperature rose to the F.P. A temperature-time graph was plotted and the level portion revealed the freezing point.

The tube A was now removed and the solvent allowed to melt. A

weighed in  
in pellet for  
results.

Volume

Density

Weight

F.P. of

F.P. of

S.P. of

Depression

Molecular

To find

part

Sealing

weighed amount of substance under investigation was introduced through M in pellet form. The experiment was now repeated.

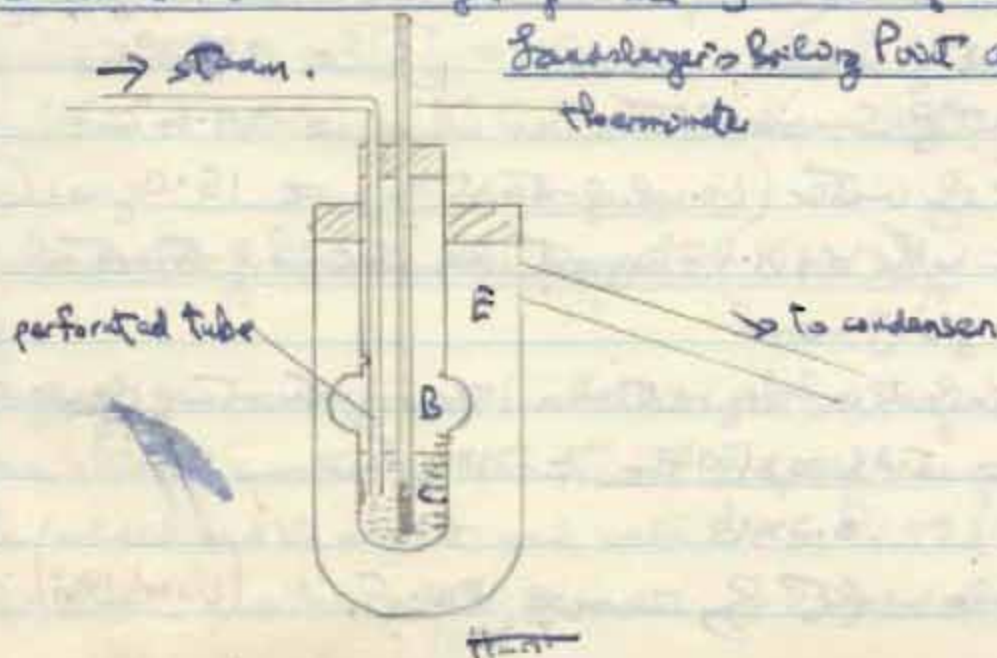
Results.

Volume of benzene = 18.5 cc.  
 Density = 0.88 g/cc  
 Weight of benzene =  $18.5 \times 0.88$   
 F.P. of pure benzene =  $5.0^{\circ}\text{C}$   
 F.P. of constant of benzene =  $51.0 \text{ per } 100^{\circ}\text{C}$   
 F.P. of benzene and naphthalene =  $1.8^{\circ}\text{C}$   
 $\therefore$  Depression =  $3.2^{\circ}$

Molecular weight of naphthalene =  $\frac{51 \times 100 \times 1.139}{18.5 \times 3.2 \times 0.88}$  = 111.5 (Correct 120.0)

To find the Molecular Weight of Sugar by the Boiling Point Method.

Sandberger's Boiling Point apparatus





### Method

The steam was passed into the tube C, which is graduated, and contained a little water. The water was raised to its boiling point by the latent heat of condensation of the steam, and the boiling point of the pure distilled water found with the accurate thermometer. When the water boils the steam passes through it without condensing and passes out through a small hole in the inner tube to jacket the latter with the vapour of the water to provide excellent insulation to prevent loss of heat due to radiation. The bulb stops water being splashed through the hole. A known weight of the mercuric chloride was introduced and the boiling point of the sugar solution found accurately as before. The volume of solution in the graduated tube was read.

### Results

Boiling point constant for water =  $5.2^{\circ}\text{C}/100\text{ gms}$   
Weight of mercuric chloride =  $1.0414\text{ gms}$   
Boiling point of water =  $99.2^{\circ}\text{C}$   
Boiling point of Sucrose solution =  $99.4^{\circ}\text{C}$   
Weight of water (i.e. vol. of water in tube) =  $15.0\text{ gms. (cc)}$   
 $\therefore 0.20^{\circ}\text{C}$  is the rise in boiling point when  $15.0\text{ gms}$  of water contain  
 $1.0414\text{ gms}$  of mercuric chloride  
 $\therefore 5.2^{\circ}\text{C}$  is the rise in boiling point when  $100\text{ gms}$  of water contain the same amount  
of mercuric chloride =  $\frac{5.2 \times 100 \times 1.0414}{0.2 \times 15} = 180.5$   
 $\therefore$  the molecular weight of mercuric chloride =  $180.5$  (correct 180.0)

### The Preparation

#### Method.

10 gms  
mercuric chloride  
potassium  
placed in  
furnace &  
to cool and  
was filtered  
to crystallize  
and dried  
mercuric chloride  
crystallized  
(NH<sub>4</sub>)  
Cr<sub>2</sub>O<sub>7</sub>  
2/52 Cr<sub>2</sub>O<sub>7</sub>  
Residue  
The Preparation

#### Method.

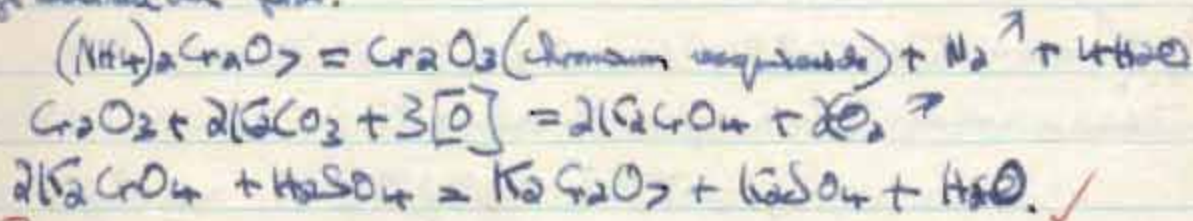
10 gms  
potassium  
mercuric chloride  
red for 15



## The Preparation and Properties of Potassium Dichromate ( $K_2Cr_2O_7$ )

### Method.

10 gms of chromium sesquioxide (obtained by heating ammonium sesquioxide), 10 gms of potassium nitrate (oxidizing agent) and 7 gms of potassium carbonate were mixed together well in a mortar. The mixture was placed in an earthenware crucible and strongly heated in a muffle furnace for a few hours. After heating the mixture in the crucible was allowed to cool and the yellow mass extracted with boiling water. The solution was filtered and divided into two portions. The second portion was exposed to crystallize out the potassium chromate solution. The first portion was acidified with dilute sulphuric acid until it became orange in color. The resulting solution was crystallized, and the potassium dichromate crystals crystallized out first.



### Result

## The Preparation of Potassium Permanganate ( $KMnO_4$ )

### Method.

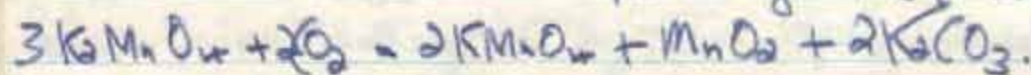
10 gms of potassium hydroxide were melted in an iron tray. 1 gm of potassium chlorate was stirred into it. 7.5 gms of manganese dioxide were then added and the whole stirred well. It was heated to bright red for 15 mins, and then cooled. The melt was broken down and



ground to a fine powder. This was transferred to a flask containing glass beads and 200 c.c. of water was added and the mixture boiled, and at the same time carbon dioxide was passed through it from a supply of dilute acid and marble.



pot. manganate (green)



pot. permanganate.

Boiled for 10 mins., cooled and filtered, the filtrate returned to the flask and carbon dioxide passed through it and boiled again for 10 mins. It was filtered and evaporated to crystallization point.

Remet

10/13/67

Summary  
Reactions

1. Potassium

1. With

orange pot.

H<sub>2</sub>SO<sub>4</sub>

2. K<sub>2</sub>CO<sub>3</sub>

2. With

a dark green

an addition

pieces of

BaCl<sub>2</sub>

Ba<sup>++</sup>

3. With

acidified

Reagents:

2 Ag<sup>+</sup>

4. With

former

(CH<sub>3</sub>COO)

5. With

iron

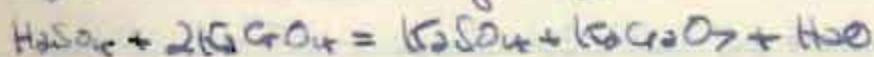
the liquid

Summer Term, 1967.

Reactions of Chromates, Dichromates, and Permanganates

1. Potassium Chromate ( $K_2CrO_4$ )

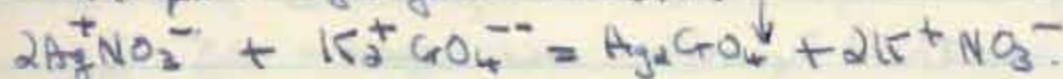
1. With dilute sulphuric acid the yellow potassium chromate was oxidized to orange potassium dichromate forming potassium sulphate.



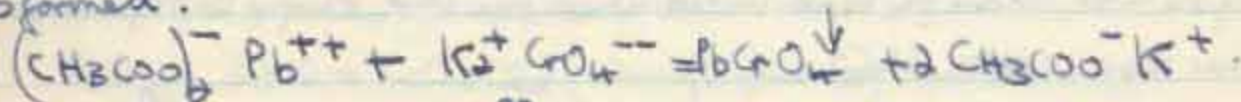
2. With barium chloride solution followed by dilute hydrochloric acid a dark yellow precipitate of barium chromate was formed which dissolved on addition of hydrochloric acid. This is an example of the precipitating power of potassium chromate.



3. With silver nitrate solution followed by dilute nitric acid a dark precipitate of silver chromate was formed which dissolved on addition of acid to give a bright yellow solution.



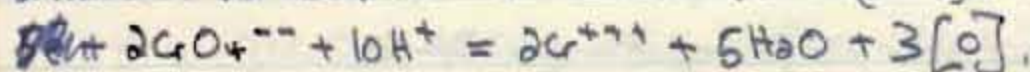
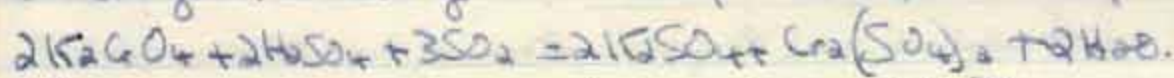
4. With lead acetate solution a yellow precipitate of lead chromate was formed.



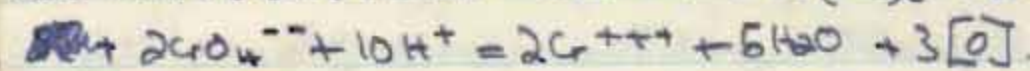
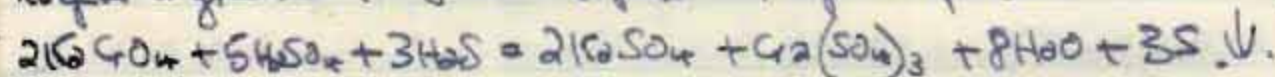
5. With hydrogen peroxide, acidified with solution of potassium chromate gave a blue colouring of perchromic acid, ~~was formed~~. The hydrogen peroxide is oxidized to give the perchromic acid ( $H_2CrO_5$ ) possibly.



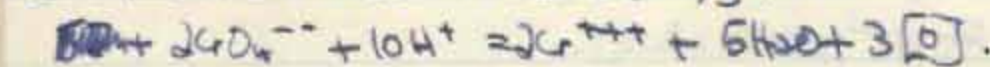
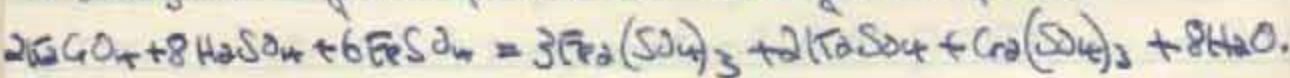
6. When sulphur dioxide gas is bubbled through a solution of potassium chromate acidified with dilute sulphuric acid the sulphur dioxide is oxidised to give a dark green solution of chromic sulphate and potassium sulphate.



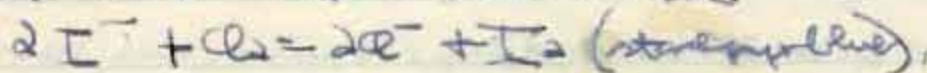
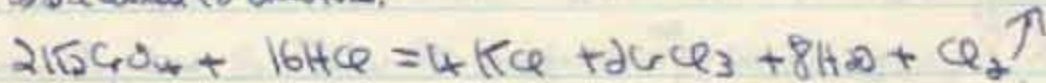
7. When hydrogen sulphide gas is bubbled through a solution of potassium chromate acidified with dilute sulphuric acid the hydrogen sulphide is oxidised to give a green solution of chromic sulphate and sulphur deposited.



8. With ferrous sulphate solution a solution of potassium chromate acidified with dilute sulphuric acid gives a green colouring of chromic sulphate together with a solution of ferric sulphate and potassium sulphate, showing that the ferrous sulphate is oxidised to ferric sulphate.



9. With concentrated hydrochloric acid ~~and~~ a solution of potassium chromate gives an orange precipitated chromic chloride ~~also~~ together with chlorine gas which turned starch paper blue. Therefore the hydrochloric acid is oxidised to chlorine.





## Reactions of Dichromates.

### Potassium Dichromate ( $K_2Cr_2O_7$ )

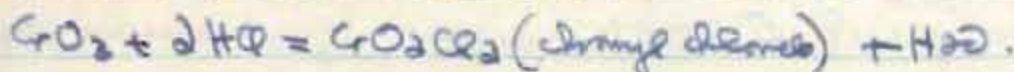
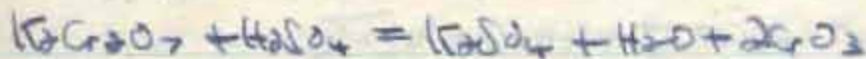
1. With potassium hydroxide solution, a solution of potassium dichromate gave yellow potassium chromate.



2. With concentrated hydrochloric acid a pale green precipitate of chromyl chloride is formed together with deposits of ~~white~~ chlorine gas which turns starch paper blue. The hydrochloric acid is oxidised to give chlorine.



3. When solid sodium chloride was added to solid potassium dichromate and concentrated sulphuric acid added and the solution warmed red deposits of chromyl chloride were formed below the sulphuric acid. The sulphuric acid is oxidised to form potassium sulphate, and chromic oxide, and the ~~white~~ hydrogen chloride, formed by the reaction of sodium chloride on sulphuric acid, joins with chromic oxide to give chromyl chloride.

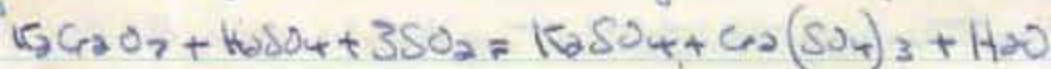


4. With ethyl alcohol, a solution of potassium dichromate mixed with dilute sulphuric acid gave a green solution of chromous sulphate and the typical smell of acetaldehyde. The alcohol is oxidised to acetaldehyde.

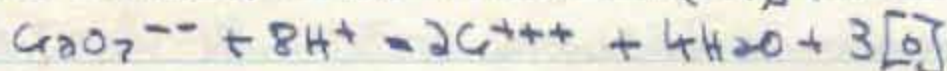
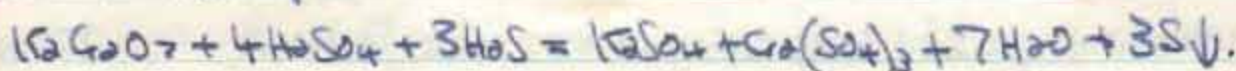




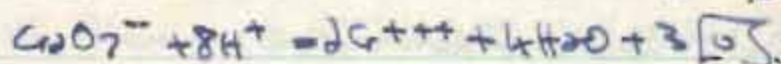
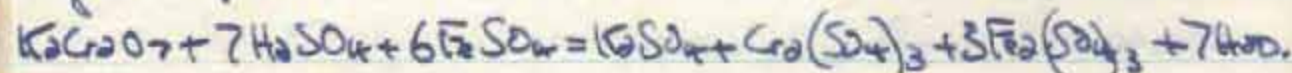
5. When sulphur dioxide gas was bubbled through a solution of potassium dichromate acidified with dilute sulphuric acid a dark green colouring of chromic sulphate was formed. The sulphur dioxide is oxidised to give chromic sulphate and potassium sulphate.



6. When hydrogen sulphide gas was bubbled through a solution of potassium dichromate acidified with dilute sulphuric acid a dark green colouring of chromic sulphate was formed together with deposits of sulphur. The hydrogen sulphide gas is oxidised to sulphur.



7. With a solution of ferrous sulphate a solution of potassium dichromate acidified with dilute sulphuric acid gave a dark green colouring of a mixture of potassium sulphate and ferrous and chromic sulphates. The ferrous sulphate is oxidised to ferric sulphate.



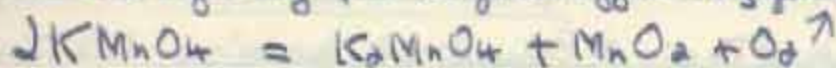
### Reactions of Permanganates

#### Potassium Permanganate ( $KMnO_4$ )

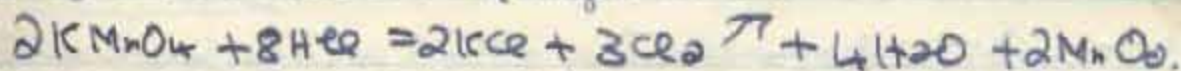
1. When solid potassium permanganate is heated in a test tube, oxygen gas



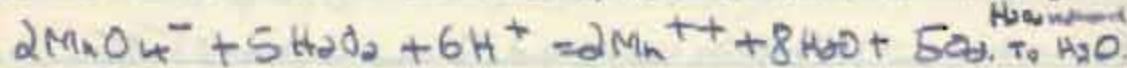
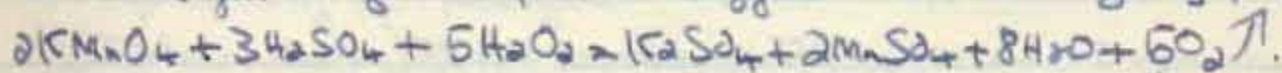
which resembles a glowing splint is given off during the reaction.



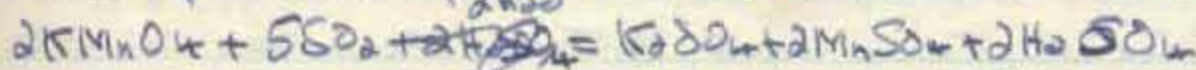
2. With concentrated hydrochloric acid the reaction of potassium permanganate with HCl and chlorine gas, which turned starch paper blue, was given off. The hydrochloric acid is oxidized to give chlorine.



3. With hydrogen peroxide, a solution of potassium permanganate acidified with dilute sulphuric acid neutralized each other, evolving oxygen in equal amounts to give manganese sulphate and oxygen which resembled a glowing splint.



4. With sulphur dioxide a solution of potassium permanganate acidified with dilute sulphuric acid was reduced to a colorless solution of manganese sulphate. The sulphur dioxide is oxidized.



5. With hydrogen sulphide gas an acidified solution of potassium permanganate was reduced to give manganese sulphate. The hydrogen sulphide was oxidized to sulphur.



6. When a solution of oxalic acid was added to a solution of potassium



### Results

White, needle shaped crystals of potassium tetrates dihydrate containing two molecules of water were collected

### 2. Sodium tetrates pentahydrate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ )

10 gm. of sodium sulphite in 50 c.c. of water. add 3 gm. of powdered sulphur. Boil gently for 1/2 hr. until the solution is no longer alkaline to litmus. (This was tested by withdrawing a drop of the solution on the end of a glass rod and dropping it on red litmus paper). Filter, and evaporate to crystallization point. Crystallization point was reached when a drop of solution withdrawn on the end of a glass rod and allowed to cool gave tiny crystals. The solution was then cooled.  $\text{Na}_2\text{SO}_3 + \text{S} = \text{Na}_2\text{S}_2\text{O}_3$

### Result

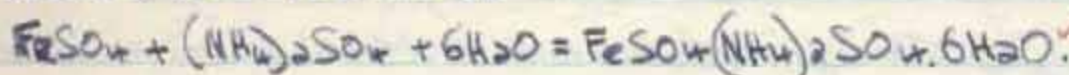
Large, transparent, monoclinic prisms, containing five molecules of water were left in the evaporating dish after crystallization

### Double Salts

### 1. Ferrous ammonium sulphate ( $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ )

5 c.c. of concentrated sulphuric acid was diluted by adding it to 50 c.c. of water. 5 gm. of iron filings were carefully added to the solution and the solution warmed. The solution was filtered and then 10 gm. of ammonium sulphate was added to it. This solution was then filtered and evaporated to crystallization point.

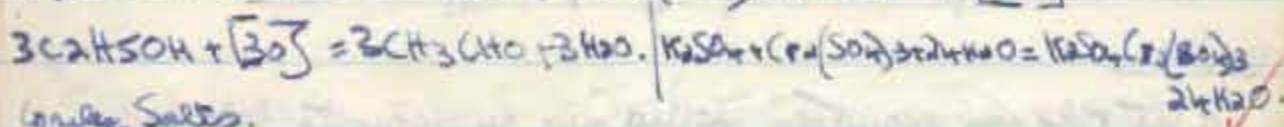
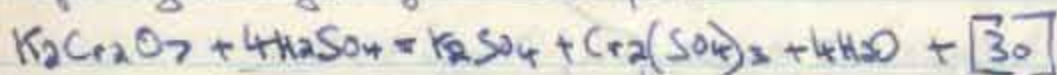




mult. Transparent bluish-green, transparent, rhombic crystals were collected  
2, chromalum. (Potassium chromium sulphate  $\text{K}_2\text{SO}_4 \cdot \text{Cr}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$ ).

3 gms. of potassium dichromate were dissolved in 100 c.c. of water. 12 c.c. of concentrated sulphuric acid was added to the solution keeping the temperature below  $25^\circ\text{C}$ . 10 c.c. of ethyl alcohol was added to this solution keeping the temperature down by cooling. The final solution was evaporated to crystallization point, the liquor was added when a drop of solution withdrawn on a glass rod gave tiny crystals. The crystals were then collected, dried, and bottled.

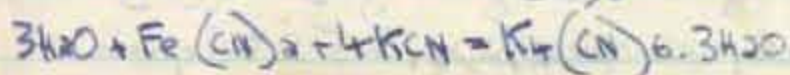
Result. Deep violet coloured crystals of octahedral shape were left in the evaporating dish after crystallization point.



Complex Salts.

The Preparation of Potassium Ferro-cyanide ( $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$ )

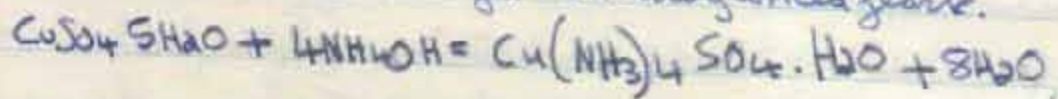
5 gms of hydrated ferrous sulphate were dissolved in 50 c.c. of water. 15 gms of potassium cyanide was also dissolved in a similar amount of water. The potassium cyanide was added to the ferrous sulphate solution and the resulting solution stirred and, if necessary, filtered until the red precipitate is dissolved. The filtrate was then evaporated to crystallization point. The test for this is as above.





### 16. The Preparation of Cuprammonium Sulfate ( $\text{Cu}(\text{NH}_3)_4\text{SO}_4 \cdot \text{H}_2\text{O}$ ) ✓

5gms of hydrated copper sulfate was dissolved in 50cc of water. Ammonium hydroxide solution was added to the solution until the precipitate redissolved. 10cc of alcohol was now added to the ink blue solution and the resulting solution was allowed to remain overnight in a loosely corked flask.



(Results. ①. Large yellow monoclinic prismatic crystals of potassium ferrioxalate were collected.

②. Tiny, thin, blue rhombic prismatic crystals of cuprammonium sulfate were collected.

### 17. To determine the Equivalent weight of Sn, Oxygen Method.

A test tube was accurately weighed very in accurate balance. Some tin was now put into the test tube and the test tube containing the tin was again accurately weighed. A drop of concentrated nitric acid was now added to the tin in the test tube. A vigorous action occurred in which fumes of brown nitrogen peroxide were given off. The shiny zinc was converted into a white powder, metallic zinc acid. When the fumes began to be given off less vigorously, the test tube was now heated at the top of the Bunsen tube and brought to its length, until all fumes of any kind (e.g. water vapour and excess nitrogen peroxide) were given off. The test tube, now

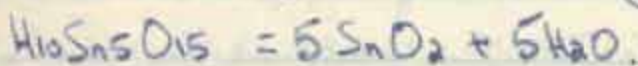
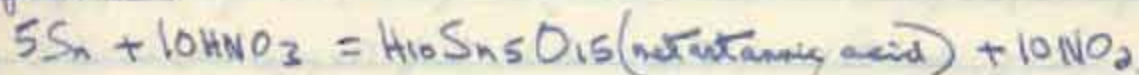


✓  
 The stannous oxide was again accurately weighed after it was cool. The test  
 tube containing the stannous oxide was now weighed to constant weight.  
Final results.

Weight of test tube	= 14.387 gms	14	387
Weight of test tube + Tin	= 16.414 gms	8	0.9031
Weight of test tube + stannous oxide	= 16.919 gms	2.027	0.3069
∴ weight of tin = 16.414 - 14.387	= 2.027 gms ✓		1.2100
Weight of oxygen = 16.919 - 16.414	= 0.505 gms	0.505	1.7033
∴ 0.505 gms of oxygen joins with 2.027 gms of tin			1.5067
∴ 8 gms of oxygen joins with	8 × 2.027 gms of tin		
	0.505.		
	= 32.11 gms of tin		

∴ equivalent weight of tin = 32.11. (Correct 25.75)

Equation



Conclusion

The equivalent weight of stannous (tin) was found to be 32.11 by  
 the oxygen method.



An Experiment To Find the atomic weight of Potassium by precipitating Silver Chloride from Potassium Chloride Solution.

Method

A watch glass was weighed accurately. Some potassium chloride powder was added to the watch glass and the weight of the potassium chloride (about one gram) accurately determined. The potassium chloride was now dissolved in a beaker of distilled water and the solution transferred to a clean, dry beaker. A solution of silver nitrate was prepared similarly and an excess of silver nitrate was added to the potassium chloride solution to precipitate silver chloride. A little of the silver nitrate solution was kept back. The solution was now filtered and the filtrate tested for complete precipitation by adding the silver nitrate solution kept back. If the filtrate contained any potassium chloride a white precipitate of silver chloride is formed. If negative, the filtration was completed, the precipitate was completely transferred to the filter paper with a bobby, and the precipitate washed. The filter paper was added in an oven ~~and~~, the precipitate transferred to a weighed crucible and lid, and the filter paper was incinerated. The crucible, lid and contents were now weighed and the weight of silver chloride found, subtracting the weight of the ash (0.0006 gms). The crucible and contents were now heated to constant weight.

Results.

Weight of watch glass	= 8.842 gms
Weight of watch glass + potassium chloride	= 9.9314 gms
∴ Weight of potassium chloride	= 1.042 gms.

Weight

Weight

Weight

∴ wt of

∴ 1.85

potassium

∴ 143

∴ equi

∴ atomic

potassium

∴ 249

Method

∴ app

liquid

100. 10

20/10/16



Weight of crucible + lid = 19.132 gms

Weight of crucible + lid + silver chloride = 20.987 gms

Weight of air = ~~1.855 gms~~ 0.0004 gms

∴ wt of silver chloride = 1.855 gms

∴ 1.855 gms of silver chloride are precipitated by 1.042 gms of silver  
potassium chloride

∴ 143.5 gms of silver chloride are precipitated by  $\frac{1.042 \times 143.5}{1.855}$

∴ equivalent weight of potassium chloride = 80.48

∴ atomic weight of potassium = equivalent weight of  
potassium chloride - equivalent of chlorine  
= 80.48 - 35.5  
= 44.98.

	Ag	Cl
1.855	1.042	0.0178
143.5		156.8
		2.1746
1.855		0.2689
		1.9057

### Molecular Weights of Volatile Liquids

Experiment to find the Vapour Density of a volatile liquid by Victor  
Meyer's Method

#### Method:

The apparatus was assembled as shown in the diagram. Boil also a  
liquid (usually water) with a boiling point at least  $30^\circ\text{C}$  above  
that of the volatile liquid whose vapour density is being  
measured. The bottom of the tube is covered with dry sand or



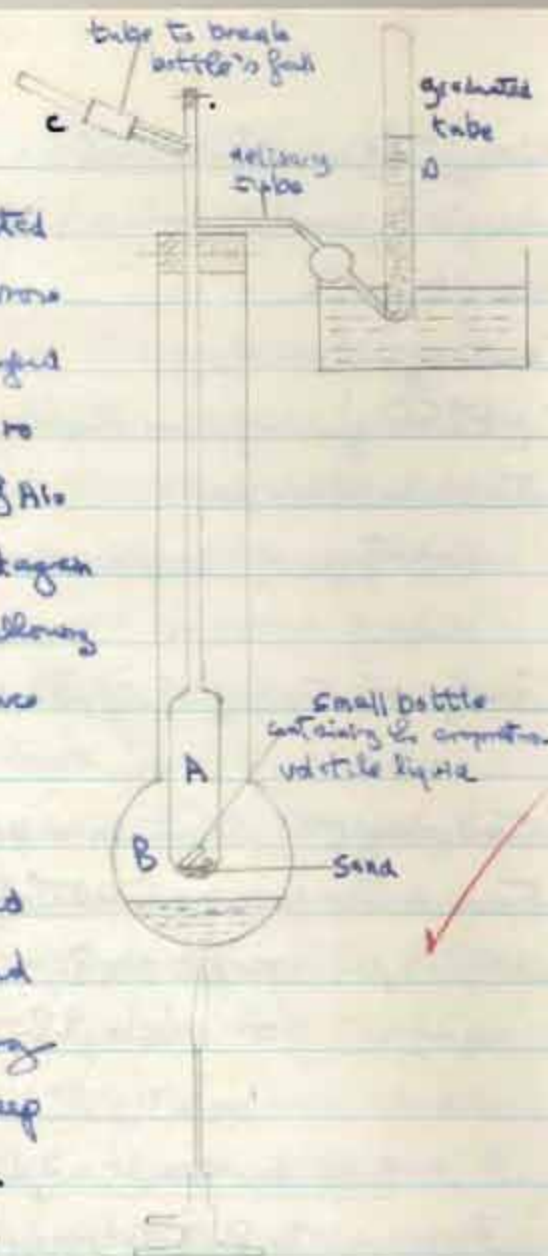
ambient as to break the fall of the small tube  
 containing liquid and the bath B is then heated  
 until all the air in A has expanded so that no more  
 escapes from the delivery tube. The liquid is weighed  
 in a previously weighed well stoppered bottle. When no  
 more air escapes from A, the cork at the top of A is  
 opened, the bottle dropped in and the cork closed again  
 directly. The glass rod C is then withdrawn allowing  
 the bottle to fall to the bottom of A and it is  
 replaced.

The stopper is blown off the bottle and  
 the liquid evaporated quickly. The displaced  
 air is collected in the graduated tube D and  
 when no more air is expelled from the delivery  
 tube the graduated tube is removed to a deep  
 vessel full of water and levels equalized.

The vapor pressure of water vapor at the  
 temperature measured is subtracted from the volume of air in the graduated  
 tube and the true vol of dry air found. The barometric pressure is noted.  
 The experiment is conducted rapidly in order to avoid diffusion and loss of  
 vapors.

### Results

Weight of small bottle = 0.825 gms.



Weight of  
 ∴ weight of  
 Volume of  
 Temperature  
 Pressure  
 ∴ pressure  
 to reduce  
 by gas

$$\therefore V_2 = 3$$

1000 c.c.  
 ∴ 31.74 c.c.

∴ Vapor pressure

∴ volume



Weight of small bottle + stopper = 1.025 gms

∴ Weight of stopper = 0.2 gms

Volume of air displaced = 44.0 - 7.9 = 36.1 c.c.

Temperature = 24°C = 297° A

Pressure = 749 mm. - 22 mm (vapour pressure of water vapour)

∴ pressure of dry air = 727 mm.

To reduce to N.T.P.

$$\text{By Gas Laws } \frac{V_1}{T_1} = \frac{V_2 P_2}{T_2}$$

$$\therefore V_2 = \frac{36.1 \times 273 \times 727}{297 \times 760} = 31.74 \text{ c.c.}$$

1000 c.c. of Hydrogen @ N.T.P. weigh 0.09 gms

∴ 31.74 c.c. of Hydrogen @ N.T.P. weigh  $\frac{0.09 \times 31.74}{1000}$

$$= 2.856$$

1000

$$= 0.002856 \text{ gms}$$

∴ Vapour density of liquid =  $\frac{\text{wt. of vapour}}{\text{wt. of equal vol. of hydrogen}}$

wt. of equal vol. of hydrogen

$$= 0.2$$

$$0.002856$$

$$= 70.02$$

(high)

$$\therefore \text{molecular wt} = 70.02 \times 2 = 140.04$$

(correct 59.8)

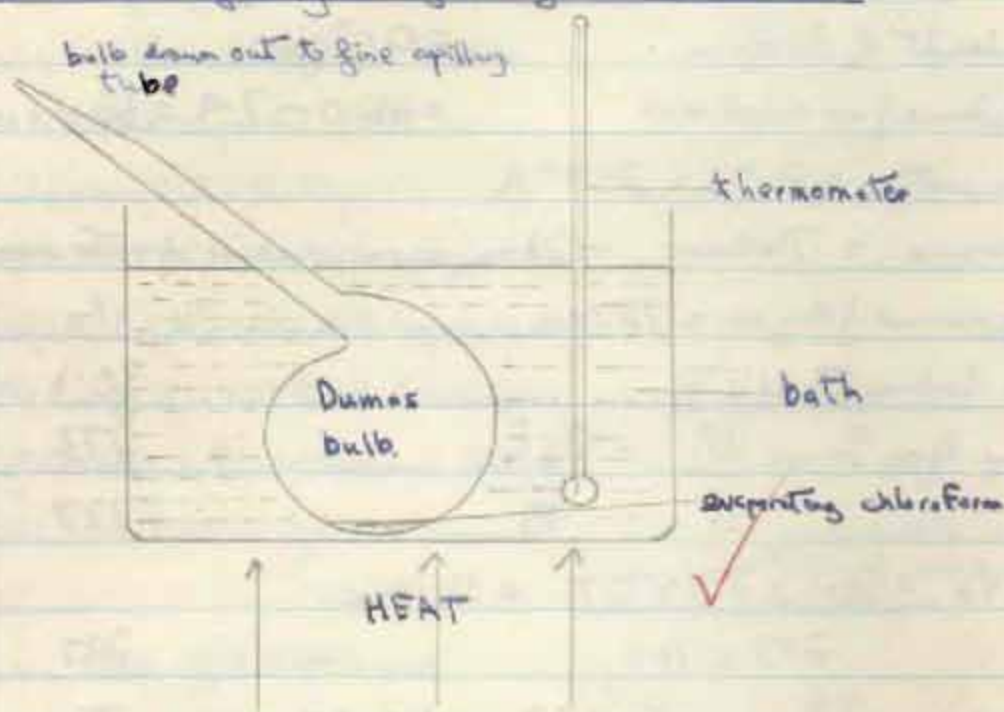
Po	Log
36.1	1.5575
273	2.4352
727	2.8615
	6.8552
297	2.4728
760	2.8808
	5.3536
	1.5016

Po	Log
0.09	5.9542
31.74	1.5016
	0.4558

Po	Log
0.2	1.3010
0.002856	3.4558
	1.9452



## To find the Molecular Weight of chloroform by Dumas' Method.



### Method.

A Dumas bulb was weighed full of air. The Dumas bulb was then very gently heated to expand the air inside it and therefore reduce the pressure. The narrow end was then put into a watch-glass containing a little chloroform and the chloroform was sucked into the bulb. The bulb containing the chloroform was now immersed as completely as possible into a bath of water (which has a B.P. of at least  $20^{\circ}\text{C}$  above that of chloroform) and the bath heated to evaporate the excess chloroform vapour. After the temperature of boiling water was taken and the excess vapour extracted from the bulb the bulb was sealed. After cooling the bulb was weighed containing the chloroform vapour and then the seal was broken under water, and the water sucked into the bulb to take the place of the vapour because of the reduced pressure. The bulb was then

weighed.

Conclusion

The vapor weight  $12.5$

Results.

Weight of bulb  
Weight of bulb  
Weight of bulb  
Temperature of  
Lab. temperature  
atmosphere pressure

Volume of water  
water occupies  
 $\therefore$  Volume of

To reduce T

By Gas Laws

$$\frac{V_1 P_1}{T_1} = \frac{V_2 P_2}{T_2}$$

$$T_1 \quad T_2$$

$$\therefore V_2 = 218$$

1000 c.c.



Weighted full of water with the pieces of glass broken off.

### Conclusion

The vapour density of chloroform was found to be 61.03 and molecular weight 122.06 by the following method. ✓

### Results

$$\text{Weight of bulb + air} = 28.14 \text{ gms}$$

$$\text{Weight of bulb + air + vapour} = 28.75 \text{ gms}$$

$$\text{Weight of bulb full of water} = 246.69 \text{ gms}$$

$$\text{Temperature of boiling water} = 100^\circ \text{C} = 373^\circ \text{A}$$

$$\text{Lab. temperature} = 20^\circ \text{C} = 293^\circ \text{A}$$

$$\text{atmospheric pressure} = 763 \text{ mm.}$$

$$\text{Volume of water filling bulb} = 246.69 - 28.14 = 218.55 \text{ c.c. (as 1 gm of water occupies 1 c.c.)}$$

$$\therefore \text{Volume of air in bulb at } 20^\circ \text{C} + 763 \text{ mm pressure} = 218.55 \text{ c.c.}$$

So Reduce To N.T.P.

By Gas Laws

$$\frac{V_1 P_1}{T_1} = \frac{V_2 P_2}{T_2}$$

$$; V_1 = 218.55 \text{ c.c.} \quad V_2 = ?$$

$$T_1 \quad T_2$$

$$P_1 = 763 \text{ mm.}$$

$$P_2 = 760 \text{ mm.}$$

$$T_1 = 293^\circ \text{A}$$

$$T_2 = 273^\circ \text{A.}$$

$$\therefore V_2 = \frac{218.55 \times 763 \times 273}{760 \times 293}$$

$$760 \times 293$$

1000 c.c. of dry air @ N.T.P. weigh 1.293 gms.



$$\therefore V_2 \text{ cc. of dry air at N.T.P. weigh } \frac{28.55 \times 763 \times 273 \times 1.293}{760 \times 293 \times 1000} \text{ gm}$$

$$= 0.2644 \text{ gm}$$

$$\therefore \text{Weight of bulb - air} = 28.14 - 0.2644 = 27.8756 \text{ gm}$$

$$\therefore \text{Weight of vapour} = 28.75 - 27.8756 \text{ gm}$$

$$= 0.8744 \text{ gm}$$

$$\text{Volume of vapour at } 100^\circ\text{C and } 763 \text{ mm pressure} = 218.6 \text{ cc}$$

Reduce To N.T.P.

By Gas Laws

$$\frac{V_1 P_1}{T_1} = \frac{V_2 P_2}{T_2}; V_1 = 218.6 \text{ cc} \quad V_2 = ?$$

$$T_1 = 373^\circ\text{A} \quad P_1 = 763 \text{ mm} \quad P_2 = 760 \text{ mm}$$

$$T_2 = 273^\circ\text{A}$$

$$\therefore V_2 = \frac{218.6 \times 763 \times 273}{760 \times 373} \text{ cc}$$

$$\therefore \text{Volume of equal volume of hydrogen} = V_2 \text{ cc}$$

$$1000 \text{ cc. of dry hydrogen @ N.T.P. weigh } 0.09 \text{ gm}$$

$$\therefore V_2 \text{ cc. of dry hydrogen @ N.T.P. weigh } \frac{218.6 \times 763 \times 273 \times 0.09}{760 \times 373 \times 1000} \text{ gm}$$

$$= 0.01435 \text{ gm}$$

$$\therefore \text{V.D. of chloroform} = \frac{\text{wt. of chloroform vapour}}{\text{wt. of an equal volume of hydrogen}}$$

$$= \frac{0.8744}{0.01435} = 61.03$$

No	Temp	Volume
218.6	2.3397	In bulb
763	2.8825	Carbonate
273	2.4362	Na2CO3
1.293	0.1116	106.
	7.7700	2 equivalent
293	2.4669	1 equivalent
760	2.8808	∴ N. wt.
1000	3.0000	10
	8.3477	∴ 2500 cc.
	7.4223	

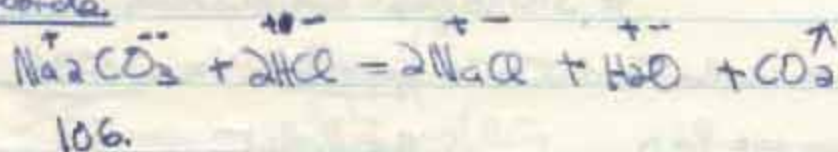
No	Temp	Method
218.6	2.3397	Same as before
763	2.8825	To 250 cc.
273	2.4362	acid no. 2
0.09	2.9542	carbon dioxide
	6.6126	∴ determine
760	2.8808	∴ not diff
373	2.5747	Results
1000	3.0000	Weight
	8.4555	Weight
	2.1571	∴ weight

(59.8).



### Volometric Analysis

To find the Strength of Hydrochloric acid using Pure anhydrous Sodium Carbonate



2 equivalents of Hydrochloric acid react with 106 parts of sodium carbonate.  
1 equivalent of Hydrochloric acid react with 53 parts of sodium carbonate.

$\therefore$  11 sodium carbonate = 53 gms per litre.

$\frac{11}{10}$  sodium carbonate = 5.3 gms per litre.

$\therefore$  250 c.c. of ~~sodium~~  $\frac{11}{10}$  sodium carbonate solution contains  $\frac{5.3}{4}$  gms or (1.2 - 1.5) gms.

### Method.

Some anhydrous sodium carbonate was dissolved in distilled water and the solution was made up to 250 c.c. 25 c.c. portions of the solution was titrated against the Hydrochloric acid in a burette using 1 drop of methyl orange as indicator, which is insensitive to carbon dioxide. The colour changed from yellow to orange-pink. One trial only was obtained and two accurate readings. The accurate readings not coincident and it must differ by only 0.1 c.c. ✓

### Results

Weight of weighing bottle = 13.865 gms

Weight of weighing bottle + sodium carbonate powder = 15.114 gms

$\therefore$  weight of sodium carbonate = 1.259 gms. ✓

$\therefore$  In 250 c.c. of sodium carbonate solution 1.259 gms of sodium carbonate



are dissolved.

$\therefore$  in 1 litre of sodium carbonate solution  $1.259 \times 4$  gms of sodium carbonate  
 $= 5.036$  gms are dissolved.

$\therefore$  Normality of sodium carbonate solution  $= \frac{\text{gms/litre}}{\text{equivalent}} = \frac{5.036}{53}$

Let normality of hydrochloric acid be  $n$

Normality of sodium carbonate  $=$  no of c.c.s of hydrochloric acid

normality of hydrochloric acid  $=$  no of c.c.s of sodium carbonate

Number of c.c.s of sodium carbonate solution  $= 25$

No of c.c.s of hydrochloric acid  $= 25.65$  (trial 25.6)

$$\therefore \frac{5.036}{53n} = \frac{25.65}{25}$$

$$53n = 25.65$$

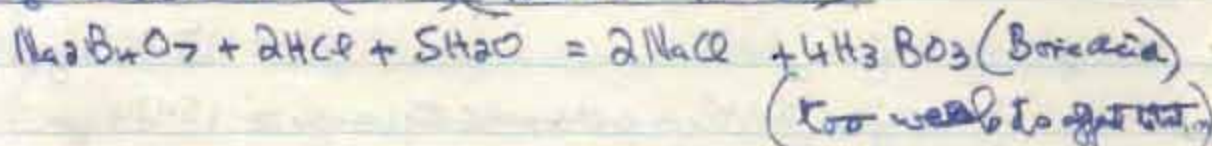
$$\therefore n = \frac{5.036 \times 25.65}{25 \times 53} = 0.0975$$

$\therefore$  normality of acid  $= 0.0975 N$

Conclusion

The normality of the hydrochloric acid was found to be  $0.0975 N$

An Experiment to Standardize Hydrochloric acid using Sodium Tetraborate decahydrate Solution (Borax) ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ )



2 equivalents of hydrochloric acid  $\equiv \text{Na}_2\text{B}_4\text{O}_7 \equiv \text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$   
(391.4)

$\therefore$  1 equivalent

$\therefore$  equivalent

$\therefore$  N factor

$\therefore$  10 litres

Method.

Between 1  
solution made  
against the hydro  
chloric acid  
accurate result

Results

Weight of

Weight of

Weight of

$\therefore$  On 25 c.c.s of

$\therefore$  On 1 litre

$\therefore$  Normality

No of c.c.s of

No of c.c.s

normal

normal



$$\therefore 1 \text{ equivalent of hydrochloric acid} = \frac{\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}}{2} = \frac{391.4}{2} = 195.7$$

$$\therefore \text{equivalent of Borax} = 195.7$$

$$\therefore \text{N Borax} = 195.7 \text{ gms/litre}$$

$$\therefore \frac{11}{10} \text{ Borax} = 19.57 \text{ gms/litre}$$

$$= 4.89 \text{ gms/250 c.c.}$$

### Method.

Between 4 and 5 c.c. of borax was dissolved in distilled water and the solution made up to 250 c.c. 25 c.c. portions of the solution were titrated against the hydrochloric acid in a burette using a drop of methyl orange as indicator. The colour changed from yellow to orange-pink. One trial reading was obtained and then accurate readings. The accurate readings must only differ by at most 0.1 c.c.

### Results

$$\text{Weight of weighing bottle} = 13.907 \text{ gms.}$$

$$\text{Weight of weighing bottle + borax powder} = 18.331 \text{ gms}$$

$$\therefore \text{Weight of borax} = 4.424 \text{ gms.}$$

$$\therefore \text{On 250 c.c. of borax solution } 4.424 \text{ gms of borax are dissolved.}$$

$$\therefore \text{On 1 litre of borax solution } 4.424 \times 4 = 17.696 \text{ gms are dissolved.}$$

$$\therefore \text{Normality of borax solution} = \frac{\text{gms/litre}}{\text{equivalent}} = \frac{17.696}{195.7} = 0.09046$$

(a)

$$\text{No of c.c. of hydrochloric acid solution} = 25.00$$

$$\text{No of c.c. of hydrochloric acid required} = 25.00 \text{ all readings?}$$

$$\frac{\text{Normality of borax solution}}{\text{Normality of hydrochloric acid}} = \frac{\text{No of c.c. of hydrochloric acid}}{\text{No of c.c. of borax solution}}$$



$$\therefore \text{normality of hydrochloric acid} = \frac{25}{25} \times 0.09046.$$

$$= 0.09046.$$

### Conclusion

The normality of the hydrochloric acid was found to be 0.09046.

An Experiment To Find The Strength of Sodium Hydroxide solution using the Hydrochloric acid of normality as above



40. equiv.

Equivalent of hydrochloric acid reacts with 40 parts of sodium hydroxide

$\therefore$  N sodium hydroxide = 40 gms/litre.

### Method.

The burette was filled with sodium hydroxide solution and a wire-glass tube fitted to minimise the action of atmospheric carbon dioxide on the solution. The solution was titrated against 25 c.c. volume of hydrochloric acid of strength as above using methyl orange, methyl red, and phenolphthalein indicators. It was found that two accurate readings were obtained using methyl orange and 1 reading using methyl red and phenolphthalein.

### Results.

10 gms of sodium hydroxide

1) with methyl orange - true 20.2; accurate, 20.75, 20.7

2) with methyl red - 20.75.

with phenolphthalein  
normality of  
normality of  
normality of  
normality of

### Conclusion

The normality

### An Experiment

(COOH)<sub>2</sub>

(COOH)<sub>2</sub>

$\therefore$  2 equiv

$\therefore$  1 equiv

$\therefore$  N

$\therefore$  N

### Method.

The burette

gms of sodium

250 c.c. 25



GLAMORGAN  
EDUCATION COMMITTEE

Name *Myron Wyn Evans*

Subject *Chemistry*

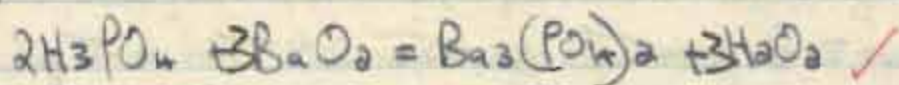
E. J. Arnold & Son Ltd. Leeds



## The Preparation and Properties of Hydrogen Peroxide

### Method

Measure out 100 c.c. of nuggy phosphoric acid. Dilute with its own volume of water. Gradually add barium peroxide while cooling under the tap until the barium peroxide is in excess. Filter the ~~white~~ solution.

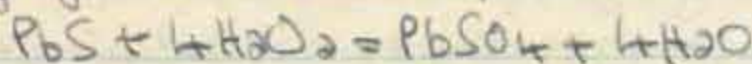


### Tests and Properties

1. When powdered manganese dioxide was added to the solution of hydrogen peroxide there was a vigorous <sup>reaction</sup> to liberate a gas which rekindled a glowing splint showing that it was oxygen. The hydrogen peroxide had been decomposed to water with the manganese dioxide as catalyst with oxygen given off.



2. When lead acetate which had been blackened to lead sulphide was added to a solution of hydrogen peroxide the paper was oxidized to white lead sulphate. Therefore hydrogen peroxide can act as an oxidizing agent. It was reduced to water.



3. With purple potassium permanganate acidified with dilute sulphuric acid was added to a little of the solution, the solution became nearly white and a gas which rekindled a glowing splint showing that it was oxygen was given off. The acidified potassium permanganate

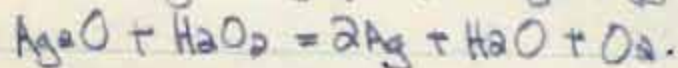


was reduced to give potassium sulphate and manganese sulphate  
 $2\text{KMnO}_4 + 3\text{H}_2\text{SO}_4 + 5\text{H}_2\text{O}_2 = \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + 8\text{H}_2\text{O} + 5\text{O}_2$   
 $[\text{H}_2\text{O}_2 + \text{O} = \text{H}_2\text{O} + \text{O}_2]$ .

Therefore hydrogen peroxide can also act as a reducing agent  
 4. When some hydrogen peroxide solution was added to potassium iodide solution acidified with dilute sulphuric acid the solution became dark red due to the formation of iodine which turned starch paper blue. The potassium iodide was oxidised to potassium sulphate and the hydrogen peroxide, the oxidising agent, was reduced to water.



5. With silver oxide solution, prepared from sodium hydroxide and silver nitrate solution, hydrogen peroxide reduced it to give silver, and oxygen, which rekindled a glowing splint, was given off.



6. With ether and orange potassium dichromate acidified with dilute sulphuric acid, one drop of a solution of hydrogen peroxide gave a blue layer of perchromic acid which is formed in the ether layer. This is a special test for hydrogen peroxide.

### Conclusion

Hydrogen peroxide can behave both as an oxidising agent and a reducing agent. Formula =  $\text{H}_2\text{O}_2$  or  $\text{HOOH}$ .

### Summary

A solution of hydrogen peroxide may be concentrated to 50% by

careful evaporation  
by distillation  
by freezing  
peroxide, a  
common

volume was  
may be obtained  
10 volume of  
at N.T.P.

a true  
oxide with

Simple Salts

Potassium

$2\text{H}_2\text{O}$ .

binds

this solution

more effervescent

neutralised

quickly filled

point. Then

$\text{COOH} + \text{K}_2$

$\text{COOH}$



careful evaporation between 75°C and 100°C. Further concentration is possible by distillation and reduced pressure. Further, the water may be removed by freezing it out as ice to leave concentrated, anhydrous hydrogen peroxide, a thick, colourless liquid of S.G. 1.6.

Commercially it may be brought under various strengths from 10 volume concentrations i.e. the number of c.c. of oxygen at N.T.P. that may be obtained by the decomposition of 1 c.c. of hydrogen peroxide. Thus 10 volume of hydrogen peroxide means that 10 c.c. of oxygen are obtained at N.T.P. by the decomposition of 1 c.c. of hydrogen peroxide.

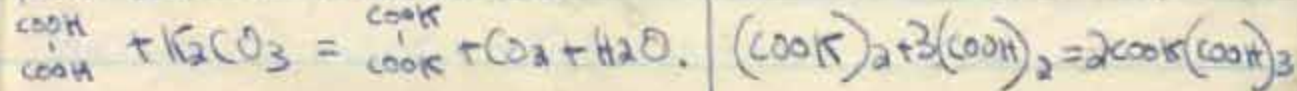
A true peroxide is a higher oxide able to give a solution of hydrogen peroxide with any cold dilute acid.

### Preparation of Salts

#### Simple Salts

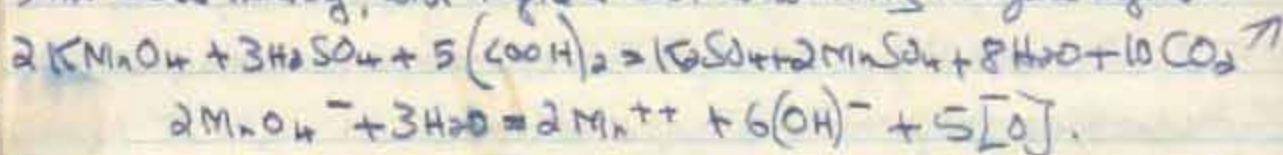
Potassium Tetroxalate di-hydrate  $(KH_2(C_2O_4)_2 \cdot 2H_2O \cdot COOK(COOH))$   
 $(2H_2O)$ .

Redissolve 10 gms of oxalic acid in 100 c.c. of water. Measure out 25 c.c. of this solution and boil. Add excess potassium carbonate until there is no more effervescence. Boil the remaining 75 c.c. of solution and add to it the neutralised portion. Redissolve any precipitate by adding boiling water. Cool quickly. Filter and evaporate to crystallisation point. Test for crystallisation point. Then allow solution to cool.





permanganate acidified with dilute sulphuric acid and the mixture warmed, the oxalic acid was oxidized to give off fumes of carbon dioxide gas, which turned lime-water milky, and to give a colorless solution of manganese sulphate.



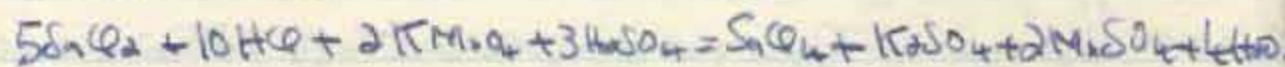
7. With a solution of ferrous sulphate, a solution of potassium permanganate acidified with dilute sulphuric acid was reduced to a colorless solution of manganese sulphate together with the potassium sulphate. The ferrous sulphate was converted to ferric sulphate.  $(2\text{MnO}_4^- + 3\text{H}_2\text{O} = 2\text{Mn}^{++} + 6(\text{OH})^- + 5[\text{O}])$

$$2\text{KMnO}_4 + 10\text{FeSO}_4 + 8\text{H}_2\text{SO}_4 = \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + 5\text{Fe}_2(\text{SO}_4)_3 + 8\text{H}_2\text{O}$$

8. With sodium nitrite solution an acidified solution of potassium permanganate was reduced to manganese sulphate (colorless solution), the sodium nitrite being oxidized to give sodium nitrate.



9. With stannous chloride solution an acidified solution of potassium permanganate, with hypochlorous acid, was reduced to a colorless solution of manganese sulphate and stannous sulphate, the stannous chloride being oxidized to stannic chloride.



To find the

Sulphate

molecular

2 KMnO<sub>4</sub>

2 (39 +

316

80 parts of

8 parts of

1. square

Ferric acid

= 56 + 3

2 Fe SO<sub>4</sub>

2 (39.2)

16 parts of

8 " "

1. equivalent

for N

for 260.

Method was

accounted

ratios of the



3) with phenolphthalein - 20.6.44

Normality of Hydrochloric acid = 0.09046.

Normality of Hydrochloric acid =  $\frac{\text{no of c.c. of sodium hydroxide}}{\text{no of c.c. of Hydrochloric acid.}}$

Normality of sodium hydroxide

Normality of sodium hydroxide =  $0.09046 \times 25$

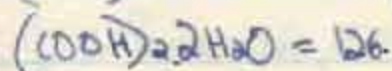
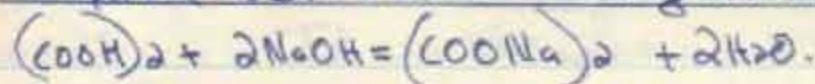
20.75.

= 0.1090. *N*

### Conclusion

The normality of the sodium hydroxide solution was found to be 0.109.

An Experiment to Standardize a solution of Sodium Hydroxide using oxalic acid.



$\therefore$  2 equivalents of sodium hydroxide  $\equiv$  90 parts of anhydrous oxalic acid.

$\equiv$  126 parts of hydrated oxalic acid.

$\therefore$  1 equivalent of sodium hydroxide  $\equiv 126/2 = 63$  parts of hydrated oxalic acid.

$\therefore$  N oxalic acid = 63 gms / litre

$\therefore$   $\frac{N}{10}$  oxalic acid = 6.3 gms / litre

= 1.4 - 1.8 gms / 250 cc.

### Method.

The burette was filled with sodium hydroxide solution. Between 1.4 and 1.8 gms of oxalic acid crystals were dissolved in distilled water and the solution made up to 250 cc. 25 cc. portions of the solution were titrated against the sodium hydroxide solution.



in a little more phenolphthalein coloration (This indicator is always used with weak acids). A faint pink tinge which permanently remained in the solution was observed. Re-neutralization occurred. One trial was again obtained and two accurate readings which differed by 0.1 c.c. at the end.

### Results

Weight of weighing bottle = 13.868 gms

Weight of weighing bottle + oxalic acid = 15.378 gms

$\therefore$  wt of oxalic acid crystals = 1.51 gms

In 25 c.c. of oxalic acid solution 1.51 gms of oxalic acid was dissolved.

In 1 liter of oxalic acid solution 6.04 gms of oxalic acid are dissolved.

$\therefore$  normality of oxalic acid solution =  $\frac{\text{gms/liter}}{\text{equivalent}} = \frac{6.04}{63} = 0.0992$

One trial = 21.93 c.c. another reading = 21.93, 21.96 c.c.

normality of oxalic acid =  $\frac{\text{no. of c.c. of sodium hydroxide solution}}{\text{no. of c.c. of oxalic acid}}$

normality of sodium hydroxide sol. =  $\frac{\text{no. of c.c. of oxalic acid}}{\text{no. of c.c. of sodium hydroxide solution}}$

$\therefore 0.0992 = \frac{21.93}{25}$

normality of sodium hydroxide sol. = 25

$\therefore$  normality of sodium hydroxide solution =  $25 \times 0.0992$

21.93

= 0.1092 N

### Conclusion

The normality of the sodium hydroxide solution was found to be 0.1092

So find the

$\text{Mg} + 2\text{HCl}$

2 equiv

$\therefore$  1 equivalent

### Method

Between 0.

magnesium was

a funnel was in

was complete to

and with water

now 25 c.c. of

to find the con

One trial and

### Result

Weight of

Volume of N

So find the

Strength

titration V

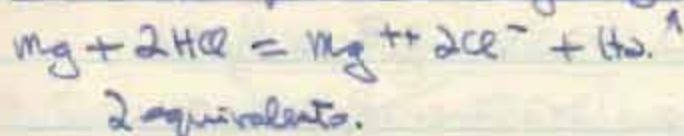
Spice

10.200

$\therefore$  25 c.c. R



To find the Equivalent weight of Magnesium



$\therefore$  1 equivalent of magnesium  $\equiv$  1 equivalent of N acid  
 $= 1000 \text{ c.c. of N HCl.}$

#### Method:

Between 0.1 and 0.3 gms of clean magnesium was accurately weighed. The magnesium was put into a flask containing 25 c.c. of N Hydrochloric acid. A funnel was inserted to prevent loss of acid through spray. When the action was complete the solution was transferred to a 250 c.c. graduated flask and with water was made up to 250 c.c. It was shaken well and two 25 c.c. portions were titrated against 0.1N caustic soda to find the excess acid present. Indicator used was methyl orange. One trial and two accurate readings were obtained.

#### Results:

Weight of magnesium  $= 0.158 \text{ gms.}$

Volume of N HCl added  $= 25 \text{ c.c.}$

To find excess acid present.

Strength of caustic soda  $= 0.1 \text{ N}$

Titration Volume of caustic soda  $= 25 \text{ c.c.}$

Spent	accurate 1	accurate 2
12.2 c.c.	12.35 c.c.	12.35 c.c.

$\therefore$  25 c.c. of solution required 12.35 c.c. of 0.1N sodium hydroxide



to neutralise excess acid.

$\therefore$  250 c.c. of solution required 12.35 c.c. of 0.1N NaOH.

$$= 12.35 \text{ c.c. of } 1N \text{ NaOH.}$$

$$= 12.35 \text{ c.c. of } 1N \text{ HCl (excess acid)}$$

$\therefore$  Volume of acid required to react with 0.158 gms of magnesium

$$= 25 - 12.35$$

$$= 12.65 \text{ c.c.}$$

$$\therefore 12.65 \text{ c.c. of } 1N \text{ HCl} = 0.158 \text{ gms of magnesium}$$

$$\therefore 1000 \text{ c.c. of } 1N \text{ HCl} = \frac{0.158 \times 1000}{12.65} = 12.49.$$

$$12.65$$

$$\therefore \text{Equivalent weight of magnesium} = 12.49.$$

100  
15/12/66

Enter Sample

The Solution

mixture of alcohol + ether + sulphate powder

Method.

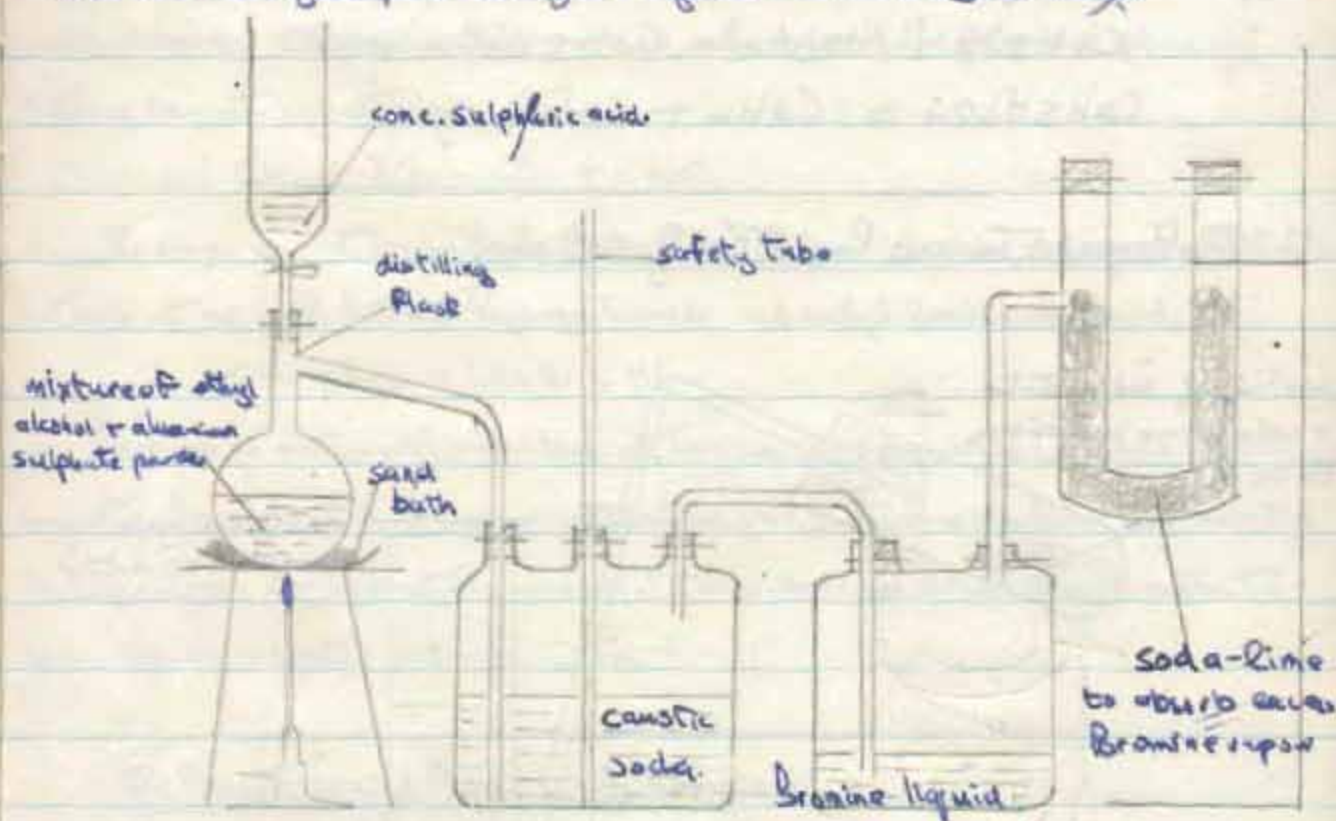
distilling off  
put into the  
sulphuric acid  
had been pre-  
lubricated into



Enter Term 1967

Organic Preparations

The Laboratory Preparation of Ethylene Dibromide ( $C_2H_4Br_2$ )

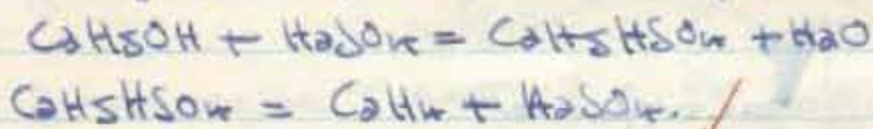


Method.  $(C_2H_5 + Br_2 = C_2H_4Br_2)$

Some aluminum sulphate powder and <sup>30 c.c.</sup> ethyl alcohol were put into a distilling flask and the apparatus assembled as in the diagram. Bromine was put into the three washed flasks and the safety tube replaced. 80 c.c. of conc. sulphuric acid was now allowed to run slowly into the flask. When the acid had been put into the flask it was gently warmed and the ethylene gas evolved bubbled into the bromine and gradually decolorized it to form the oily colorless

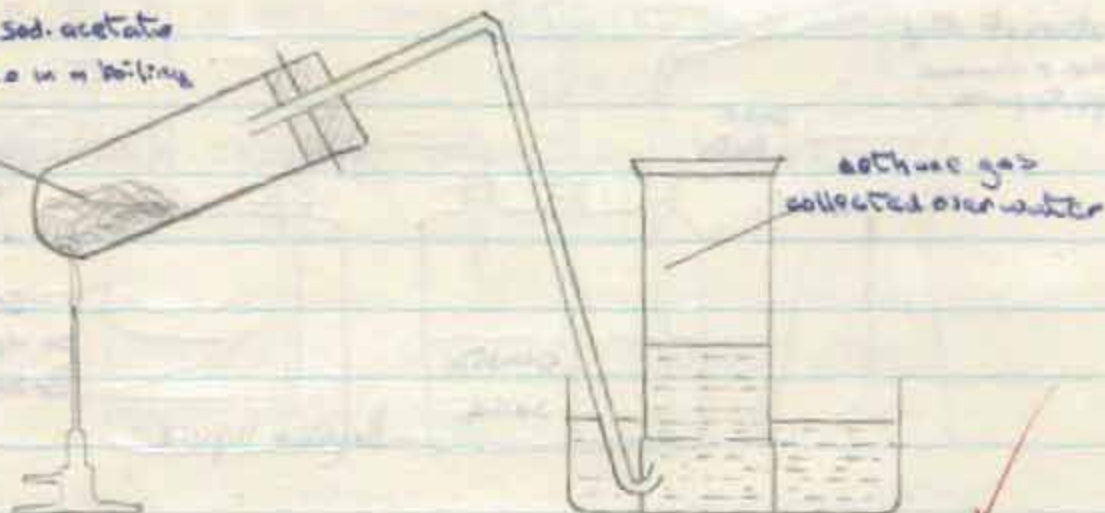


ethylene dichloride in the latter. Sulphuric acid removed to the flask. a little of caustic soda may be included before the three necked bottle to purify the ethylene which may contain sulphur dioxide and other impurities



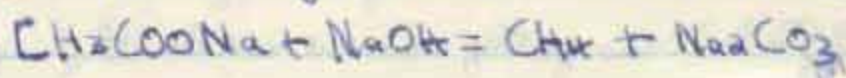
### The Preparation and Properties of Methane.

mixture of sod. acetate  
+ soda-lime in a boiling  
tube.



#### Method

a mixture of 20 gms of sodium acetate and 20 gms of soda-lime was put in a boiling tube and the apparatus assembled as in the diagram. Soda-lime acts as a catalyst and is partially heated and cooled. The boiling tube was heated and the methane gas evolved collected over water.



#### Tests and

1. St.

2. When

carbon dioxide

Methane dioxide

3. Reaction

action with

4. Reaction

agents, the

hydrogen gas

carbon dioxide

#### The P.

mixture of ethyl  
alcohol and  
chromium  
sulphate  
powder

ethyl  
alcohol