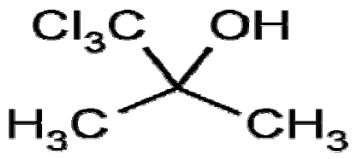
CHAPTER 16

STUDY OF FOLLOWING OFFICIAL COMPOUNDS

CHLOROBUTOL

FORMULA : $C_4H_6Cl_3OH$

STRUCTURE :



OTHER NAME : Chloretone

PREPARATION : It is prepared by direct combination of acetone with chloroform in presence of solid KOH**.**

ASSAY : A weighed quantity of substance is dissolved in alcohol and hydrolysed by being boiled under reflex with Aq NaOH. The chlorides ions are determined by Volhard's method with N/10 $AgNO_3$ an N/10 NH_4SCN. Before the thiocynate titration the mixture is shaken in presence of small quantity of nitro benzene in order to coagulate the AgCl precipitate.The result is expressed in terms of the hemi hydrate, so that 1000 ml of N/10 $AgNO_3$ are equivalent to 1/30 Of $C_4H_7Cl_3O$. 1/2 H_2O.

USES :

- Crystaline compound of characteristic odour.
- Has hypnotic properties an use as preventive of sea sickness.
- Due to variable proportion of water of crystallization the melting point is not lower than 77^0 C.

DIMERCAPROL

FORMULA : $C_3H_8OS_2$.

STRUCTURE :

OTHER NAME **:** 2,3 dimercapto-1-propanol

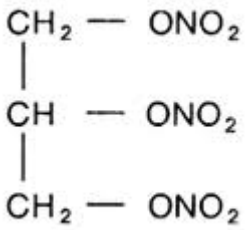
ASSAY : Dissolve about 0.12 gm,accurately weighed in 20 ml of HCl (0.1 mol/lt) Vs and titrate rapily with ioine V_s using starch is an indicator. Report the experiment test without the test liquid being examined, and make any necessary corrections. Each 1ml of iodine V_s is equvivalent to 6.211mg of $C_3H_8OS_2$.

USES : Antidote for arsenic,gold and mercury poisoning.

GLYCERYL TRINITRATE

FORMULA : $C_3H_5O_3(NO_2)$

STRUCTURE :



OTHER NAME : nitroglycerine

PREPARATION : It is prepared by slowly adding glycerol to an ice cooled mixture of conc.H_2SO_4 and conc.HNO_3. The solution is cautiously run into ice cold water, the nitroglycerine then separates as a heavy oil which is purified by repeated washing with cold water.

ASSAY : Nitrate is determined calorimetrically in a weighed quantity of the powdered tablets by interaction with phenol-2,4-disulphonic acid and subsequent classification with ammonia. The yellow colour is matched against that of solution

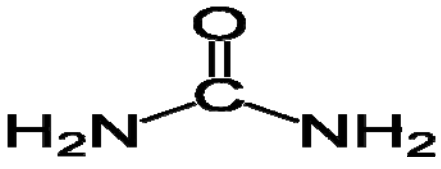
containing known quantities of potassium nitrate which hav been treated similarly. The assay depends on the formation of coloured nitro compounds by the interaction of the glycerol trinitrate with the phenol-2,4- disulphonic acid,and the sub sequent conversion of these into intensely ammonium salts. The method suffers from certain disadvantanges ,the most important of which is the difficulty of correctly matching the yellow colour.

USES :

- Manufacture of dynamite and blasting gelatin which are comparitivly safe explosives.
- In tablets it is given in certain diseases of heart

UREA

STRUCTURE :



FORMULA : NH_2CONH_2.

PREPARATION : A laboratory method is to evaporate solution of ammonium cynate to dryness (wohler) about 95% NH_4CN becomes converted into urea by reversible isomeric change.

$$NH_4COCN \rightarrow CO(NH_2)_2 \quad \text{UREA}$$

It is also synthesised by interaction of ammonia with carbonyl chloride**.**

ASSAY : The official process is based on the method of Richmond and hill. This hydrolysis the saccharin to the sodium salt of O-sulphonyl benzoic acid, without loss of ammonia.

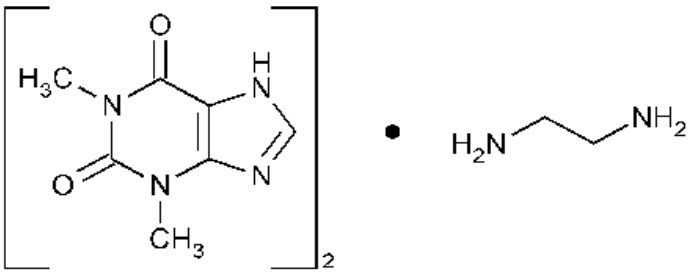
USES :

- It is widely used as a calorie free substitute sugar or diabetics and in the weight conscious.
- It also called as sweetening agent in tooth paste,mouth washes, aerated drinks.

ETHYLENE DIAMINE DIHYDRATE

FORMULA : $C_2H_4(NH_2)_2.2H_2O$

STRUCTURE :



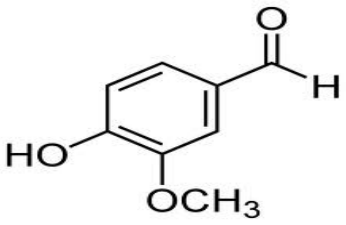
PREPARATION : From 1,2- dichloroethane and ammonia at maintaining the temperature of 180^0C.

ASSAY : By titration with N/1 HCl to the bromophenol blue indicator,this indicates the formation of the dihydrochloric acid.

VANILLIN

FORMULA : $C_8H_8O_3$

STRUCTURE :



PREPARATION : Vanillin – $C_6H_3(OMC)(OH).CHO$

It is the flavouring agent of the vanilla pods can be made synthetically from guaicol by remier – tiemann reaction. It is also prepared by eugenol. This compound is first isomerised by the alkali to isoeugenol and the later is then subjected to controlled oxidation.

ASSAY **:** Weigh accurately about 0.12gm dissolve 20ml of ethanol add 60ml of CO_2 free H_2O and titrate with 0.1m NaOH determining the end point ,potentiometrically each 1ml of 0.1m NaOH is equivalent to 0.01521g of $C_8H_8O_3$..

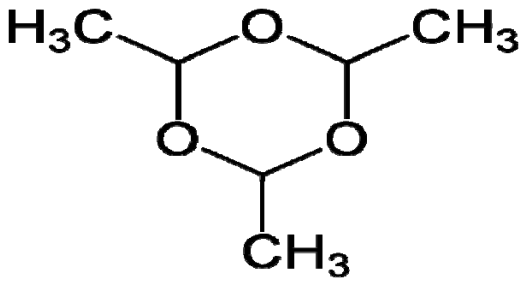
USES **:**

As a flavouring agent particularly in chocolate ice ceam and confectionary.

PARALDEHYDE

FORMULA **:** $(C_2H_4O)_3$

STRUCTURE :



PREPARATION : When acetaldehyde is treated with a small amount of con. H_2SO_4 of room temperature a cyclic trimer paraldehyde is formed.

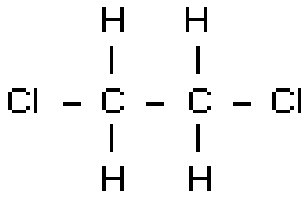
USES :

- sedative and hypnotic
- obstetric analgesic

ETHYLENE CHLORIDE

FORMULA : $C_2H_4Cl_2$

STRUCTURE :



PREAPARATION : Ethylene chloride is produced by the reaction of ethylene and chlorine.

$$C_2H_4+Cl_{2-}\rightarrow C_2H_4Cl$$

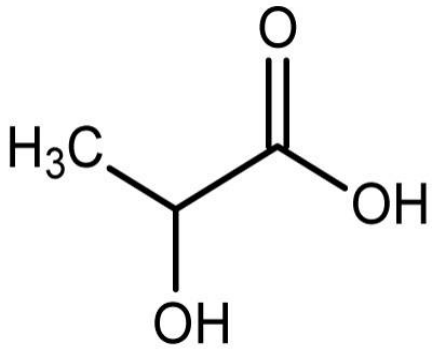
ASSAY : Stock, standard gas was created by static dilution form 100% analyte, mixed volumetrically with input of O_2, sample is verified by chromatography.

USES : Used as degreasen and paint remover.

LACTIC ACID

FORMULA : $CH_3.CH(OH).CO_2H$

STRUCTURE :



PREAPARATION: Lactic acid has been prepared by brominating propionic acid and hydrolysing the resultant 2-bromopropionic acid to sodium lactate by heating with dil.alkali.

$$CH_3CH_2CO_2H-\rightarrow CH_3.CHBr.CO_2H-\rightarrow CH_{3.}CH(OH)CO_2NO$$

ASSAY : A weighted quantity is diluted with H_2O and boiled with N/1 sodium hydroxide and excess of alkali is then determined by titration with N/1 HCl by using phenolphthalein as indicator. The boiling alkali hydrolyses the condensation products.

$NaOH+CH_3.CH(OH).CO_2H \Leftrightarrow CH_3.CH(OH).CO_2$

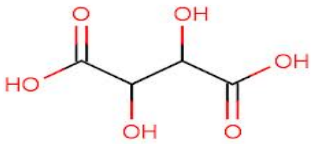
USES :

- Used in dairy products
- For determining of tides
- Ethyl and butyl lactates are used as plasticers.

TARTARIC ACID

FORMULA :$CO_2H.CH(OH).CH(OH).CO_2H$

STRUCTURE :



PREPARATION: From malic acid- malic acid produced industrially by oxidation of cyclohexone is treated with alkaline $KMNO_4$ to get m-tartaric acid.

ASSAY **:** Titration with N/1 NaOH using phenolphthalein as indicator.

$$2NaOH+C_4H_6O_6 \Leftrightarrow Na_2C_4H_4O_6+2H_2O$$

1,000ml of N/1 alkali are equivalent to ½ $C_4H_6O_6$

USES **:**

- In carbonated beverages and effervescent tablets
- In baking powder
- In silvering of mixtures

CITRIC ACID

FORMULA **:**$C_6H_8O_7.H_20$

STRUCTURE :

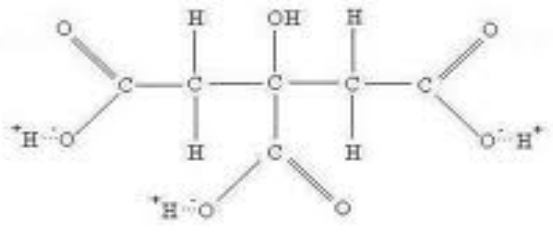


Fig. - Citric Acid ($C_6H_8O_7$) - a two dimensional structural representation.

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PREPARATION: From molasses- it containing sucrose is diluted to water and subjected to fermenting with a micro organism Aspergillus niger.

$$C_{12}H_{22}O_{11}+H_2O \rightarrow HO\text{-}C$$

ASSAY : The fermentation process is carried out for 7-10 days at 26-28^{0}C.The resulting solution of citric acid is neutralized with $Ca(OH)_2$ to form insoluble calcium citrate.This is washed with H_2O and decomposed with dilute H_2SO_4.The calcium sulphate is filtered of and the solution concentration under vaccum to get crystals of citric acid.

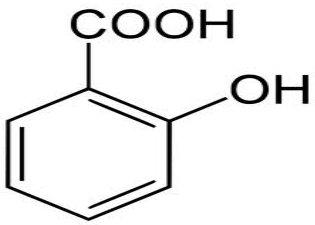
USES :

- Used as a mordant
- As a esters that are good plasticizers for lacquers and vanishers**.**

SALICYLIC ACID

FORMULA :$C_6H_4(OH).CO_2H$

STRUCTURE :



PREPARATION: This involves the treatment of sodium phenoxide with CO_2 at 125^0C under 6atm of pressure followed by acid hydrolyses therefore acid is formed.

ASSAY :Titration with N/2 NaOH in alcoholic solution using phenol as red indicator. The end point marks the completion of sodium salt. The presence of hydroxyl group makes the acid stonger than benzoic. The constant is 1×10^{-3} and the pH of sodium salicylate is therefore close to 7.

$$NaOH+C_6H_6(OH).CO_2H \rightarrow C_6H_4.CO_2Na+H_2O$$

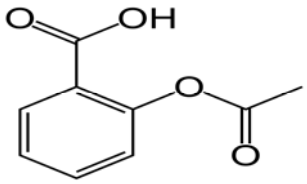
1,000ml of N/2 alkali are equivalent to ½ $C_6H_4(OH)CO_2H$

USES : Used as antiseptic and disinfectant**.**

ASPIRIN

FORMULA :$C_6H_4(O.CO.CH_3)CO_2H$

STRUCTURE :



PREPARATION: It is prepared by heating salicylic acid with acetyl chlorine in the presence of phosphoric acid. Notice that only OH group isinvolved in the reaction.

ASSAY **:** Hydrolysis with a measured volume of N/2 NaOH and titration of excess of alkali with N/2 acid.The final neutralized solution contains sodium acetate and monosodium salicylate.

1,000ml of N/2 alkali are equivalent to ¼ $C_6H_4(OCOCH_3)CO_2H$

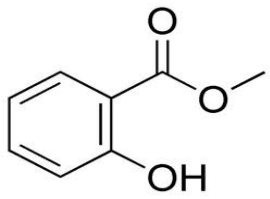
USES **:**

- Used as a painkiller
- It is sold under the trade name aspirin.

METHYL SALICYLATE

FORMULA : $C_8H_9O_3$

STRUCTURE :



PREPARATION: It is prepared by esterfying salicylic acid with methanol in presence of H_2SO_4.

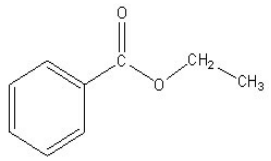
ASSAY : By a modification of the method for the determination of the esters. Instead of being first neutralized to phenolphthalein, on alcoholic solution is saponified by directly with alc.KOH. No correction is made for any free acid present, but permiscible amount of this is controlled by the above mention test. To ensure complete hydrolysis longer boiling is necessary than in the official process for the determination of the esters.

USES **:** It is used in hair tonics and ointments for treating aches, sproins and bruises.

ETHYL HYDROXY BENZOATE

FORMULA :$C_9H_{10}O_3$

STRUCTURE:



ethyl benzoate
$C_9H_{10}O_2$

PREPARATION: Ethyl alcohol is treated with benzyl chloride.

$$C_2H_5OH+C_6H_5CO_2H \rightarrow C_6H_5COOC_2H_5+H_2O$$

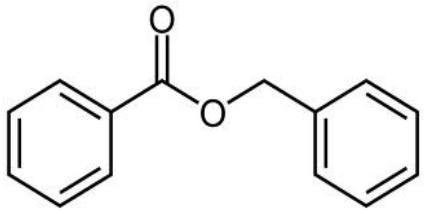
ASSAY **:** Place about 0.08g accurately weighted in a round glass stopped flask, add 2.5ml of NaOH and boil gently under reflex condenser for 3mins.

USES **:**Used as artificial fruit flavouring agent.

BENZYL BENZOATE

FORMULA :$C_{14}H_{12}O_2$

STRUCTURE :



PREPARATION:

1. Prepared by esterification of benzyl alcohol with benzoic acid of catalyst.
2. By heating benzyl chloride with potassium benzoate in the presence of diethyl amine.
3. Condensation of sodium benzyl oxide in non aqueous solution.

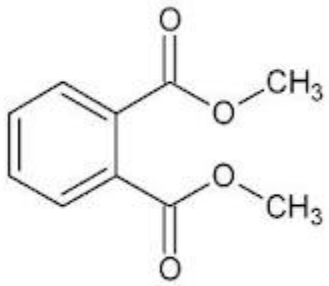
ASSAY : Add about 2.0g accurately weighted 240ml of KOH/C_2H_5OH Vs and boil under reflex for 1hr. Cool and titrate with HCl Vs using phenolphthalein or ethanol as indicator. Repeat the operation without the test liquid being examined and make any necessary correction. Each 1ml of KOH/C_2H_5OH Vs equivalent to 106.1mg of $C_{14}H_{12}O_2$.

USES : Used as a antiparasitic(scabicide-topical use).

DIMETHYL PTHALATE

FORMULA :$C_{10}H_{10}O_4$

STRUCTURE :



PREPARATION: It is prepared by oxidation of p-xylene and subsequent, esterification with methyl alcohol.

$$CH_3C_6H_4CH_3 + (O) \rightarrow HOOC.C_6H_4COOH$$

$$HOOC.\ C_6H_4COOH + 2CH_3OH \rightarrow CH_3COO.C_6H_4COOH$$

ASSAY :Weigh accurately about 2g of dimethyl phthalate into a 250ml flask attached to reflex condenser and add 50cc of 0.5NaOH and add 10cc of H_2O and reflex for 1 hr. wash down the sides of the container and glass connection with about 2.5cc of water, cool and add 0.2cc of thymol blue and titrate excess alkalie with 0.5N HCl to the production of yellow colour performance.

USES :

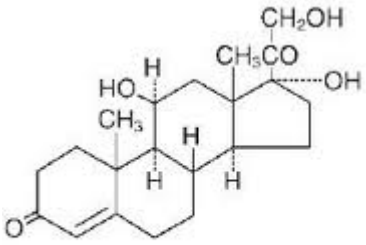
- Used as ectoparasiticide
- Solid rocket propallents

- In preparation of plastic
- Insect repellant**.**

SODIUM LAURYL SULPHATE

FORMULA :$NaC_{12}H_{25}SO_4$

STRUCTURE :



MOLECULAR FORMULA	MOLECULAR WEIGHT	CAS REGISTRY NUMBER
$C_{21}H_{30}O_5$	362.47	50-23-7

PREPARATION: It is prepared by sulphating long chain fatty alcohols and neutralizing with alkali to form the sodium salts. The alcohols are prepared by reduction of coconut oil by high pressure hydrogenation using the catalyst copper-chromium oxide.

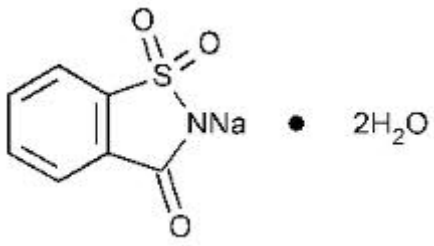
ASSAY **:**Weigh accurately about 1.15g, dissolve in sufficient water to produce 1,000ml warming if necessary to 20.01ml add 15ml of $CHCl_3$ 10ml of dil.H_2SO_4 and dimethyl oracet blue solution and titrate with 0.004M benzethonium chloride, until the $CHCl_3$ layer acquires a permanent clear green colour. Each 1ml of 0.004M benzethonium chloride is equivalent to 0.00115g of sodium alkali sulphates, calculated as $C_{12}H_{25}NaO_4S$.

USES : Pharmaceutical aid, acts as surfactant, as an emulsifier.

SACCHARIN SODIUM

FORMULA **:**$C_7H_4NNaO_3S$

STRUCTURE:



PREPARATION: When saccharin is treated with aqueous NaOH to form saccharin sodium.

ASSAY **:**Weigh accurately about 0.15g, dissolve in 50ml of anhydrous glacial acetic acid. With slight heating if necessary and carry out method A for non aqueous titration, determining the end potentiometrically perform a blank determination and make any necessary correction.

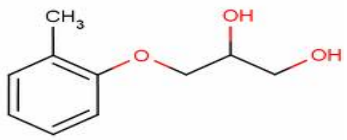
Each ml of 0.1M perchloric acid is equivalent to 0.02052g of $C_7H_4NNaO_3S$.

USES : Pharmaceutical aid, preparation of toothpaste.

MEPHENSIN

FORMULA : $(C_{11}H_{17}N)_2H_2SO_4$

STRUCTURE :



PREPARATION: It is synthesized from o-cresol. It is treated with aq.NaOH to convert sodium salt which undergoes nucleophillic substitute with 3-chloro-1,2 propane diol to give mephensin.

ASSAY **:** Test for mephensin, injection equivalent is measured accurately 1g of mephensin in position

USES **:**Used as analgesic, axiolytic, anti-fungal sometimes, muscular relaxant.

BY,

G.DEEPA