

Oxygen-Flask-Combustion Method

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Schoniger first proposed the technique of oxygen flask combustion in 1955 for organically combined halogens. In a closed assembly, in presence of oxygen organic compound is burnt, organic matter gets destroyed and halogen is released which is absorbed in an appropriate solution, which subsequently is analyzed. Besides halogen like fluorine, chlorine, bromine and iodine, sulphur is also determined by this method. This method is simple, rapid and accurate and can be applied to formulated products such as tablets, capsules, creams and ointments. However,

great care must be taken in carrying out the test especially during the combustion stage. Use of safety screen should be made while working.

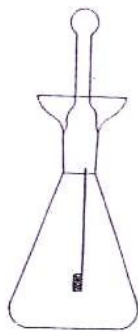


Fig.: Oxygen flask combustion apparatus

Apparatus: The apparatus (Above Figure) consists of a thick walled, 500 ml conical flask, fitted with a ground-glass stopper to which is fused a platinum wire about 13 cm long and 1 mm diameter. To this is attached a piece of platinum gauze for holding sample. The gauze is 2 cm x 1.5 cm in dimensions and is of No.36 sieve.

The general method is as follows:

Weigh accurately a suitable quantity of sample (about 20 mg) if solid on a halide free filter paper of 5 cm x 3 cm, wrap it and place in the platinum gauze sample holder. For ointments/cream weigh the sample on small size greaseproof paper, fold it and place in sample holder.

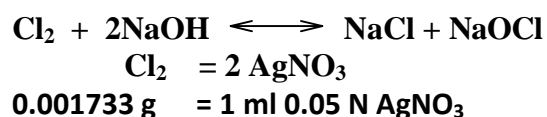
For the liquid samples use gelatin capsule or mix with methylcellulose powder and place on ash less grade paper. Insert one end of narrow strip of filter paper in the roll to serve as a fuse. Flush the flask with oxygen, moisten the neck with water and place specified quantity of absorbing liquid in the flask, fill it with oxygen and light the free end of paper strip and immediately insert stopper. Hold the stopper firmly during combustion of the sample, then shake the flask vigorously for about five minutes. Place few ml of water in the cup, carefully remove the stopper and rinse the stopper, platinum wire gauze with water. Then proceed for analysis for the particular ion or element.

For fluorine: Twenty ml of water is used as absorbing liquid. After combustion of sample add sufficient water to produce 50 ml. To 2 ml of above solution, add 20 ml water, 10 ml alizarin fluorine blue solution, 3 ml of solution containing 2 per cent sodium acetate and 6 % w/v glacial acetic acid, 10 ml of cerous nitrate and sufficient water to produce 50 ml. Allow this to stand in dark for one hour and measure the extinction in 1 cm cell at 610 nm. Prepare the blank similarly, using 2 ml water instead of test solution. Calculate the fluorine content from calibration graph, prepared by using suitable quantity of sodium fluoride solution (22 mg/2 lit).

NOTE: *For Fluorine determination combustion is carried out in silica or soda glass flask. Borosilicate glass is affected by fluorine and should be avoided.*

For Chlorine: Absorbing liquid is 20 ml of 1 N sodium hydroxide solution. To the solution add 25 ml nitric acid, 10 ml or 0.1 N silver nitrate and titrate with 0.05 M ammonium thiocyanate using ferric ammonium sulphate solution as indicator. Carry out a blank titration.

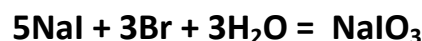
The difference between two titrations represents the amount of 0.1 N silver nitrate required for the sample. Each ml of 0.1 N silver nitrate is equivalent to 0.001773 g of Cl. Organically combined chlorine is converted to free chlorine during combustion and this is absorbed in sodium hydroxide to yield a mixture of sodium chloride and sodium hypochlorite. Addition of silver nitrate precipitates chloride as silver chloride and excess silver nitrate is back- titrated with ammonium thiocyanat,solution



For Bromine: Absorbing liquid is 15 ml of mixture of 1 volume of hydrogen peroxide (30%) and 9 volume of 1 N sulphuric acid. After the reaction is complete, add 5 ml of 2N nitric acid and 10 ml of 0.1 N silver nitrate and titrate with 0.05 N ammonium thiocyanate using ferric ammonium sulphate as indicator. A blank determination is carried. The difference between the titrations represents the number of ml of 0.05 N silver nitrate required for the sample. Each ml of 0.05 N silver nitrate is equivalent to 0.003995 g. of Br.

For Iodine: A mixture of 10 ml of water and 2 ml of 1 N sodium hydroxide is used as absorbing liquid. When the process is complete, add excess of acetic-bromine solution (5-10 ml) and allow to stand for two minutes. Remove the excess of

bromine by the addition of formic acid (about 1 ml), drive off vapour of bromine with current of air. Add 1 g of potassium towards the end of titration. Each ml of 0.02 N sodium thiosulphate is equivalent to 0.000423 g of I



For Sulphur: There are two methods for sulphur determinations. Method I is for estimation of sulphur in the absence of halogens and phosphorous. Method II is for sulphur determination in presence of halogens or phosphorous. Both methods are similar to treatment upon combustion and differ in use of absorbing liquid and further treatment.

Method-I : Burn the specified quantity of sample material in the flask. 10 ml water containing

0.1 ml of hydrogen peroxide solution (30%) is used as absorbing liquid. When the process is complete, cool the solution in ice for 15 minutes. Boil the solution gently for 2 minutes, cool, add 50 ml of ethanolic-acetic-ammonia buffer (pH 3.7) and titrate with 0.05 M barium perchlorate using 0.3 ml alizarin red S as indicator until the solution becomes orange pink in colour. Each ml of 0.05 M barium perchlorate is equivalent to 0.001603 g of S.

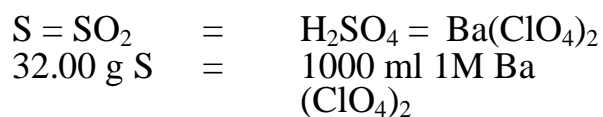
Method-II: Burn the specified quantity of sample as usual in flask. Absorbing fluid is 15 ml water containing 1 ml of hydrogen peroxide solution (10%). When the process is complete, boil solution for 10 minutes, cool and add 60 ml ethanol. Titrate the mixture with 0.01 M barium perchlorate, used as titrant and 0.1 ml of 0.2 % thoron and 0.1 ml of 0.0125 % methylene blue as indicator until the colour changes from yellow to pink.

Each ml of 0.01 M barium perchlorate is equivalent to 0.00032 g of S.

If the determinations as under Method-I and II are performed at different temperatures, the titrant volumes are corrected by the formula.

Where,	V_c	=	$V [1+0.008(t_1-t_2)]$
	V_c	=	the corrected volume of titrant
	V	=	the volume of titrant used
	t_1	=	temperature of titrant during standardization
	t_2	=	temperature of titrant during determination

During combustion, sulphur is converted to sulphur dioxide, which upon oxidation with hydrogen peroxide is transformed into sulphuric acid.



The following pharmacopoeial drugs are assayed by oxygen flask combustion method.

S.No.	Name	Identification	Absorbing liquid
1.	Diazepam	Chlorine	
2.	Diiodohydroxy quinoline	Iodine	
3.	Diiodohydroxy quinoline Tablets	Iodine	10 ml H ₂ O + 2ml 1N NaOH
4.	Diodone injection	Iodine	10 ml H ₂ O + 2ml 1N NaOH
5.	Ethacrynic acid	Chlorine	
6.	Flurouracil	Fluorine	
7.	Idoxuridine	Iodine	10 ml H ₂ O + 2ml 1N NaOH
8.	Niclosamide	Chlorine	1 N NaOH
9.	Quinodochlor	Chlorine and Iodine	5 ml 1% NaOH + 3 ml saturated sulphur solution
10.	Sulphobromophthalein sodium	Sulphur and Bromine	
11.	Throxine sodium	Iodine	

Determination of Alcohol

Ethyl alcohol is commonly known as alcohol. It is very widely used in pharmacy for various purposes. It serves as vehicle in pharmaceutical preparations and manufacture of various formulations. The amount and strength of alcohol used varies from preparation to preparation. In some preparations different strength of alcohol is used, while in others industrial methylated spirit is used. The denatured spirit or industrial methylated spirit contains upto 5 % methyl alcohol in alcohol. Detection of it is usually recommended in pharmacopoeias, since it is toxic and harmful to the health.

Determination of alcohol content is very routinely carried out in pharmaceutical industry. Different methods are used for its determination. The methods adopted by pharmacopoeia of India are discussed below.

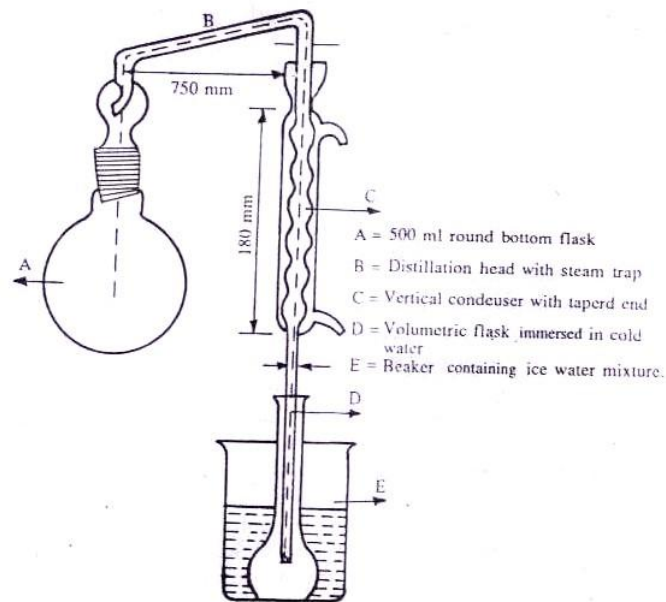
Traditionally, determination of alcohol in galenicals is carried out by distillation method followed by physical measurements of the distillate. During the distillation following steps are carried out which decides particular method to be used.

1. Distillation of all the ethyl alcohol from the sample.
2. Removal of volatile impurities and interfering materials.

Test on the distillate for identification of methyl alcohol or isopropyl alcohol; and dilution of distillate to record specific gravity to be in measurable range. **Distillation Methods :** The basic principle of this method is the distillation of the liquid sample using an appropriate distillation assembly. The distillate is then analyzed either by

determining specific gravity of distillate at specified temperature or by refractive index. In distillation method care is to be taken to minimize the loss of alcohol by evaporation. Three methods are used in distillation depending upon the nature of the sample. An appropriate treatment is given to the sample before placing it in distillation assembly. The distillation apparatus used in alcohol determination is described below.

Apparatus :The apparatus consists of four parts. Part A is a 500 ml capacity round bottom flask. To this is fitted distillation head a (B) with a steam trap. A vertical condenser (C) is fitted to the distillation head. The distillate is collected in 100 to 200 ml capacity volumetric flask (D). This flask is generally immersed in a ice-water mixture during distillation.



1 Fig.: Apparatus for Alcohol determination

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