

**INTERNATIONAL OZONE ASSOCIATION, Quality Assurance Committee,
Revised Standardized Procedure 001/96.**

IODOMETRIC METHOD FOR THE DETERMINATION OF OZONE IN A PROCESS GAS

OBJECT

The present standard method concerns the determination of ozone in air, oxygen or other process gases.

RANGE OF APPLICATION

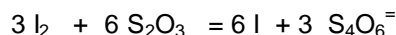
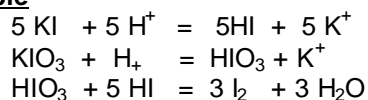
The method is directly applicable in the range of 1 g/m³ to 200 g/m³ of ozone, the volume being expressed at NTP (Normal Temperature Pressure, conditions which equal to: 0°C or 273.15 K and 1.01325 x 10⁵ Pa or 1 ATM).

REAGENTS (all of analytical grade)

- Quality of the water for make-up of solutions shall comply with ISO No.3696-1987 Grade 1).
- Buffered KI (potassium iodide) in water:
KI 20 g/L; Na₂HPO₄·2H₂O (disodium hydrogen phosphate) 7.3 g/L and
KH₂PO₄ (monopotassium dihydrogen phosphate) 3.5g/L.
- Sodium thiosulfate: Na₂S₂O₃ 0.1 mol/L in water.
- Acidifying solution: H₂SO₄ (sulfuric acid): 4.5 mol/L
- Powered KIO₃ (Potassium periodate).
- Crystalline KI.
- HCl (hydrochloric acid) or H₂SO₄ 0.1 N (certified).
- Starch indicator: ZnI₂ (zinc iodide)-starch, prepared by dispersing 4 g starch into an aliquot of water. The dispersion is added to a solution of 20 g ZnCl₂ (zinc chloride) in 100ml water. The solution is boiled until the volume has been reduced to 100mL and is finally diluted to 1-L while adding 2 g of ZnI₂. The indicator is stable for at least one month when stored in the dark at room temperature.

STANDARDIZATION OF TITRANT

Principle



Procedure

To 50mL of water in a 250 conical flask (Erlenmeyer) are added 0.05 g KIO₃ and 0.5 g KI, followed by another volume of about 50mL water. After mixing, 10 mL of certified 0.1 N acid are added. The iodine formed is titrated with the thiosulfate solution.

Results

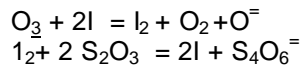
Normality of S₂O₃²⁻ equals: Normality of acid multiplied by the volume of acid (mL) and divided by the volume of thiosulfate titrant (mL).

DETERMINATION OF OZONE

Procedure

- 200mL of KI solution are added to a gas washing bottle equipped with an open gas bubbling device (tube or diffusor) under a reagent depth of 15 cm or more; (the use of fritted glass diffusors is not recommended).
- A second identical flask is connected in series as a guard detector for ozone transfer and reaction in the first flask.
- Process gas containing ozone is bubbled at a flow rate of 1 L/minute or less, until a total (estimated or expected) quantity of approximately 1 mM O₃ (it equals 0.048 g) has passed.
- The iodine formed in the solutions of KI in the flasks, immediately after acidification with 5mL of the acidifying reagent, is titrated with a freshly standardized sodium thiosulfate solution.
- After titration to a pale yellow color, optionally, 0.5mL of the starch indicator solution can be added to complete and record the final result. (This addition is recommended, but can be optional, depending on the skill and experience of the operators).

Results



Concentration of ozone in g/L equals: 24 x volume of thiosulfate in L x Normality of thiosulfate divided by the inlet volume of gas passed in L.

PRECAUTIONS

- All upstream transfer and pressure reducing equipment must be in materials which do not react with ozone, e.g., glass, PTFE,
- The gas contacting systems must have a free exit to ambient pressure.
- All gas flow must be expressed at NTP, (for high precision or when analyzing high ozone concentrations, the volume must be corrected for local existing atmospheric pressure).
- Gas flows should be measured with an accuracy of 1%: totalizing volumetric gas meter or with a bubble trap).

PRECISION AND ACCURACY

- Detection limit of the analytical procedure: 0.1 mg/L
- Repeatability: 2% of the measured ozone concentration.

INTERFERENCES

Nitrogen oxides, other oxidants of iodide ion, if present.