

EXPERIMENT NO: 4

LEAD ANALYSIS WITH DITHIZON EXTRACTION METHOD

A) General Information:

In order to isolate metallic poisons from the biological material, the organic matter in the environment must first be destroyed. In this way, metals which are resistant to degradation are left in the environment. For this purpose, as well as methods of incineration and drying in aqueous media, as well as microwave incineration are also used.

Dry incineration; The biological material is dried first, then the organic substances are destroyed at 450° C. This method is applied for all metals other than metals such as Hg, As, Sb and Pb which are volatile.

a) Basic incineration; It is the method in which the biological material is dried into ash at 450 ° C for 5-6 hours after drying at 110°C. The ash obtained is dissolved in HCl and made suitable for qualitative and quantitative analysis.

b) Incineration in the presence of a chemical substance: Biological material is burnt by adding lime, KNO₃. During combustion, the surface of the mixture with these chemicals is expanded. As it gets easier to burn, the incineration is also carried out at a lower down temperature.

c) incineration methods in aqueous media: It can be applied with chlorine, acids and other oxidants. Sulfuric acid is not used in samples with high alkaline earth alkalinity, since the collapsed sulphates of these metals adsorb and cover trace elements. Perchloric acid is suitable for degradation but is dangerous because of its explosive properties. The demolition is usually carried out in the Kjeldahl balloon. The biological material is heated after acid addition; the acid addition - heating - cooling process is continued until certain volume and clarity are reached. The product obtained is called acid digestion. Necessary analyzes are performed on acid digestion.

Lead analysis by Dithizone method

Dithizone extraction method used for the recognition of metallic poisons from acid digestion is important in terms of toxicological analyzes. It is applied in qualitative and quantitative analysis.

Dithizone is a green substance that is insoluble in water and soluble in aqueous alkalis and organic solvents. It forms chelates which can be extracted with metals using organic solvents. Metals which are known to be chelated with dithizone and which are important for toxicology are: Hg, Cu, Bi, Zn, Cd, Pb, Sn, Tl, Ni and As. Metals are chelated under certain conditions with dithizone and they are separated by selective solvent extraction.

- When the **pH of the aqueous medium is below 2**, inert metals and mercury can be removed by solvent extraction.

- **pH between 2-3**, Cu, Bi, Sn,

- **pH between 4-7** Zn, Cd, Pb, Tl

It can be taken in case of dithizonate with solvent. Dithizone-metal complexes can be quantitatively determined by spectrophotometric methods.

B) Principle and Practice of Experiment

Principle:

The lead is extracted from the acid digestion adjusted to pH 8 with the solution of the dithizone in chloroform. Excess of dithizone is removed by KCN washing solution. The amount of Pb in the extract is determined as the dithizone-Pb complex.

Experimental Procedure:

- 10 mL of distilled water and 4 mL of di ammonium hydrogen citrate solution are added to the 1 mL of solution (sample / standard)
- Adjusted to pH 8 with di ammonium hydrogen citrate buffer (controlled by pH indicator paper).
- Transfer to separating funnel; Add 5 mL of KCN buffer and 8 mL of dithizone solution. Extraction is carried out (no violent agitation to prevent emulsion formation) Sub-phase is taken.
- When lead and dithizone are combined, depending on Pb concentration, it produces bright colors ranging from pink to red.
- The lower phase is washed 4-5 times with 10 mL KCN wash solution to remove the excess of dithizone. When color is formed, the washing process is terminated. Samples are compared to standards.