EXPERIMENT 32  DETERMINATION OF COPPER, ZINC, LEAD AND CADMIUM IN FOOD PRODUCTS BY ATOMIC ABSORPTION SPECTROSCOPY

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32.0 OBJECTIVES
After attending to this experiment, we shall be able to:

- learn to perform determination of copper, zinc, lead and cadmium in food products by Atomic Absorption Spectroscopy.

32.1 INTRODUCTION
Food products contain substantial quantity of organic matter which must be destroyed prior to the estimation of minerals. Dry ashing or wet digestion is generally used for the destruction of organic matter. The wet digestion method is more preferable to dry ashing method because some of minerals may loss during dry ashing at high temperature. Atomic absorption spectroscopy is an important analytical technique used for the detection and determination of metals in foods. Atomic absorption spectrophotometer consists of hollow cathode lamp or electrodeless discharge lamp, chopper, atomizer, monochromator, detector and electronics. AAS technique offers specificity and more accuracy as compared to the chemical methods.

32.2 PRINCIPLE
The organic matter is removed by dry ashing or wet digestion. The residue is dissolved in dilute acid and sprayed into the flame of an atomic absorption spectrophotometer (AAS). The absorption or emission of the metal to be analysed is measured at a specific wavelength. AAS determines a wide range of elements based upon the principle of absorption of atoms. The liquid sample is aspirated and atomized in the flame where it converts into atomic vapour in their ground state. Atoms of metal of interest absorb the intensity of light from hollow cathode lamp. The amount of light absorbed in flame is proportional concentration of metal in solution.
**32.3 REQUIREMENTS**

**Apparatus**

*Silica crucible*

*Muffle furnace*

*Desiccator*

*Hot plate*

*AAS :* Any instrument operating in absorption mode may be used providing it has facilities for the selection of required oxidant/fuel combination from a choice of air, nitrous oxide and acetylene and has wavelength ranges from 180 to 600 nm.

*Light source :* Hollow cathode or electrodeless discharge lamps

**Reagents**

*Nitric acid (sp gr 1.42)*

*Sulphuric acid-98%*

*Hydrochloric acid-sp gr 1.16 to 1.18)*

**Standard solutions**

*Copper (100 ppm):* Dissolve 3.928 g of pure copper/copper sulphate (CuSO₄·5H₂O) in water, dilute to 1000 ml at 20°C with water in a volumetric flask. Dilute 10 ml to 100 ml with water in a volumetric flask. One ml of solution contains 100 μg Cu.

*Zinc (100 ppm):* Dissolve 1.000 g of pure zinc powder in 10 ml water, 5 ml of HCl and dilute to 1000 ml at 20°C with water in a volumetric flask. Dilute 10 ml to 100 ml with water in a volumetric flask. One ml of solution contains 100 μg Zn.

*Lead (100 ppm):* Dissolve 1.000g of pure lead in 10 ml HNO₃ in 1 litre volumetric flask, dilute to 1000 ml at 20°C with water in a volumetric flask. Dilute 10 ml to 100 ml with water in a volumetric flask. One ml of solution contains 100 μg Pb.

*Cadmium (100 ppm):* Dissolve 2.282 g of CdSO₄·8H₂O in distilled water, dilute to 1000 ml at 20°C with water in a volumetric flask. Dilute 10 ml to 100 ml with water in a volumetric flask. One ml of solution contains 100 μg Cd.

**32.4 PROCEDURE**

**Sample Preparation**

*By dry ashing :* Accurately weigh 5-10 g of sample into a dried, tared crucible. Heat the dish on hot plate until fume cease and place the crucible in furnace maintained at 550±10°C till white ash results. To the ash, add 5-10 ml of 6N HCl, and heat to dryness at low temperature on a hot plate. Add 15 ml of 3N HCl and heat on a hot plate until the solution just boils. Cool and filter through ashless filter paper (Whatman 42) into 100 ml volumetric flask. Make up the volume with distilled water. Prepare a reagent blank along with sample.

*By Wet digestion :* In case of dried products, take 2 g of the sample. If the sample contains more moisture (>10%), take 5 g or more and transfer to a 500 ml Kjeldahl flask. Add 10 ml of conc. sulphuric acid and shake vigorously. Ensure that there are no dry lumps. Add 5 ml of conc. nitric acid and mix. Heat gently in a fume cupboard until the initial vigorous reaction has subsided. Thereafter, heat vigorously until most of the
nitrous fume has ceased to evolve. Add nitric acid dropwise and continue heating until all the organic matter is destroyed and white fumes of sulphuric acid evolve. Cool the digest, add water again cool transfer to a volumetric flask and make up the volume with water. Carry out a blank through the operation using the same amount of reagents as in the case of sample.

Instrument conditions

Select the wavelength and gases to be used for the particular element under consideration from table below:

<table>
<thead>
<tr>
<th>Element</th>
<th>Wavelength (nm)</th>
<th>Gases</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zinc</td>
<td>213.9</td>
<td>Air/Acetylene</td>
</tr>
<tr>
<td>Copper</td>
<td>324.8</td>
<td>Air/Acetylene</td>
</tr>
<tr>
<td>Lead</td>
<td>217.0</td>
<td>Air/Acetylene</td>
</tr>
<tr>
<td>Cadmium</td>
<td>228.8</td>
<td>Air/Acetylene</td>
</tr>
</tbody>
</table>

The recommended settings for the various instrumental parameters vary from model to model and certain parameters require optimization at the time of use to obtain the best results. Instrument should therefore be adjusted as described in the manufacturer’s instructions using the type of flame and wavelength settings specified above.

Set the AAS to appropriate condition. Measure the absorbance of the standards. Instrument automatically will plot a graph showing net absorbance against the concentration of element in standard solution. Now read the blank and the sample solutions.

32.5 CALCULATION

\[
\text{Element in sample, mg/kg} = \frac{\text{Conc. of element (}\mu\text{g/ml}) \times \text{Dilution}}{\text{Weight of sample (g)}}
\]

32.6 RESULTS AND INFERENCE

The difference between the results of two determinations of any element carried out simultaneously or in rapid succession by the same analyst shall not exceed 0.1 mg/kg.

32.7 PRECAUTIONS

- Make sure that the sample is free from organic matter before reading in the AAS.
- Digest sample always in fuming cupboard.
- Use hand gloves, mask and goggles for protection from acid fumes while digesting the sample.
- Thoroughly washed glassware should be used
- Prepare standard solutions accurately.