AFDC 3(4791) P3
(REV TZS 268:1986)

DRAFT TANZANIA STANDARD

General atomic absorption spectrophotometric method for determination of lead in food and food stuffs.

TANZANIA BUREAU OF STANDARDS

0 Foreword

The development of this standard was necessitated by the need to address the risks associated with lead in human health.

This standard deals with the quantitative estimation of lead in foods so as to determine whether the quantity is within the acceptable limit.

In the preparation of this test method standard due consideration was given to the availability of test equipment in the country.

In the preparation of this Tanzania standard considerable assistance was derived from National Institute of Public Health, Netherlands.

In reporting the results of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off it shall be done in accordance with TZS 4: rounding off numerical values.

1.0 Scope

This Tanzania standard prescribes a method for determination of lead in food and foodstuffs. The detection limit of the method is 0.1 mg/kg as lead content in the food sample for flame AAS

2.0 References

For the purpose of this Tanzania standard the following references shall apply whereby for dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

TZS 59: – Water for analytical laboratory use – Specification and test method

TZS 4:- Rounding off numerical values.

3.0 Principle

The method involves ashing the sample at 500°C and dissolving the ash in a mixture of nitric and hydrochloric acid. Lead content is measured by flame atomic absorption spectroscopy.

4.0 Apparatus

4.1 Glassware including reagent bottles shall be of chemically resistant glass, preferably Pyrex or equivalent. It should be reserved for the estimation of lead. Before its first use it must be cleaned with warm nitric acid (4mol/ l) and water. All glassware is best kept full of 1 mol/ l HNO_3 when not in use. The neck of the bottles should be protected against dust. Cork and rubber bungs must not be used.

4.2 Crucibles of ultra-pure silica should be used. Platinum crucibles should not be used.

4.3 Measuring pipettes 1 ml, and 10ml

4.4 funnels

4.5 Muffle furnace

4.6 Atomic absorption spectrophotometer with background corrector, equipped with lead hollow cathode lamp. It is recommended to use lamp current of 6-7 mA for best sensitivity or otherwise as provided in the equipment manufacturer's manual.

4.7 Hollow cathode lamp for lead

4.8 Air, cleaned and dried through a suitable filter to remove oil water and other foreign substances.

4.9 Fuel: *Acetylene gas*

4.10 Plastic rod

4.11 Hot plate

5.0 Quality of reagents

Unless otherwise specified lead free analytical grade chemicals, and distilled water (see clause 2) shall be employed.

6.0 Reagents

6.1 Nitric acid, 65% (m/m), density= 1400kg/ m^3

6.2 Pure hydrochloric acid 35-37% (m/m), density = 1175 kg/m^3

6.3 Nitric Hydrochloric solution

Add 60 ml water to a 250-ml volumetric flask, followed by 62.5ml of nitric acid (6.1) and 62.5 ml hydrochloric acid (6.2) make up to 250 ml with water after cooling. Keep in a glass bottle.

6.4 Lead stock standard solution

Pure lead standard solution(1000mg/l)

6.5 Lead working standard solution

6.5.1 Working standard solution shall be prepared by appropriate dilution from the stock standard solution in such a way that the peak height of the working standard matches with that of the sample if possible.

6.5.2 Daily prepare serial dilutions of the lead working solutions, Dilute the stock solution (6.4) to obtain a desired working concentration range 0-5ppm. The diluent and its percentage should consider the solvent used in making stock solution. Use these concentrations for preparation of calibration curve for the preparation of the calibration curve (see 8.6).

7.0 Procedure

7.1 Preparation of the sample

7.1.1 Make the sample homogenous, avoid contact with metals by using porcelain wherever possible (spoons etc.). if metal food grinders are used, check then for possible lead contamination.

7.1.2 Weigh into a crucible 2 – 10g to the nearest 10 mg (depending on sample type) and amount of trace element expected.

7.1.3 Char the sample on a hot plate until smoke is no longer evolved or water remains.

7.1.4 Ash in a muffle furnace at 500°C until the ash is white or grayish. If ashing takes longer than the established time, it is advisable to bleach the ash with a few drops of water, evaporate on a hot plate, using low temperature to avoid spattering and continue the ashing in the furnace.look for time required for ashing.

7.1.5 Allow the crucible to cool to room temperature

7.1.6 Moisten the ashes with 2 nitric hydrochloric acid solution and a small amount of water. Heat slightly to obtain adequate dissolution. By means of a funnel, transfer into a 10-ml volumetric flask. Rinse the crucible carefully with small amounts of water (3 – 4) ml) and use this rinsing liquid to make the flask content to the mark.

7.1.7 If the mineralized is not to be analyzed immediately, transfer into a flask and keep at 4°C.

8.0 Determination

8.1 Following the manufacturers manual, switch on the atomic absorption spectrophotometer and allow the lamp and the background corrector to warm up for at least 30 minutes. Adjust the monochromator to give maximum signals nearer the recommended wavelength. Adjust slit width according to instructions of the apparatus used.

8.2 Adjust the flame – stoichiometry in such a way that the maximum absorbance and minimum noise is obtained (non-luminous flame).

8.3 Aspirate the standard solutions and not the absorption values or the peak heights, using a chat recorder.

8.4 Aspirate the sample solutions and take readings.

8.5 Blank

Carry out at least one blank along with an unknown sample through all the subsequent steps using the same amounts of reagents throughout.

8.6 Calibration curve

8.6.1 for non-automated machines: Plot the absorption values or peak heights measured against the concentration of the working standard solutions and construct the best fitting straight line through the plotted points and the origin.

> **NOTE** – It is recommended to carry out regular recovery experiments by adding standards to the sample.

The performance of the laboratory should be checked by analyzing standard reference materials.

8.6.2 For automated machine the calibration curve is generated automatically

9.0 Calculation of results

9.1 Calculate the amounts of lead present in the sample after correction for blank if necessary and by reference to the standard curve, and express results in mg lead per kg of the sample as follows:

$$C = \frac{(A - B_1)\, V}{H.M}$$

Where

C = mass concentration of metal in the sample, mg/kg
A = read out of the sample (absorbance)
B_1 = average read out of the blank (absorbance)
A-B_1 = net read out of the sample (absorbance)
H = slope of the calibration curve
M = mass of the weighed amount of the test portion in grammes.
V = volume in ml of the original sample prepared in solution.

9.2 For automated machine the results are generated automatically

9.3 Repeatability

The difference between the results of a determination in duplicate (obtained simultaneously or in rapid succession but the same analyst) shall not exceed 5% of the lead content at 1 mg/ kg level.