Draft Tanzania Standard

Lead determination in fish and fishery products

TANZANIA BUREAU OF STANDARDS

Lead determination in fish and fishery products (Draft for comments only)

0. FOREWORD

This draft Tanzania standard prescribes the spectrophotometric method for the determination of lead content in fish and fishery products.

In the preparation of this Tanzania standard assistance was derived from AOAC Official Method 972.23, Lead in fish.

In reporting the result 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(See clause 2)

1.0 SCOPE

This Tanzania standard specifies the spectrophotometric method for the determination of lead content in fish and fishery products.

2.0 REFERENCES

The following referenced documents are indispensable for the application of this standard For dated references, only the edition cited applies. For undated references, the latest edition of the referenced documents (including any amendments) applies;

TZS 4, Rounding off numerical values
TZS 59, Water - Distilled quality – Specification

3.0 APPARATUS

a) **Atomic absorption spectrophotometer** – ranging 0 -10µg/mL;
b) **Lead lamp** –Hollow cathode Pb lamp; and
c) **Crucible** –Porcelain, about 50 mL capacity and 5cm deep; or tall-form Vycor or quartz beaker,100 mL

4.0 REAGENTS

4.1 Hydrochloric acid -1M.Dilute 82 mL HCL to 1 L water.

4.2 Lead standard solutions

a) **Stock solution** - 1mg Pb/ 1M HNO_3; and
b) **Working solution -**10µg Pb/ mL Pipet 10 mL stock solution into 1L volumetric flask, add 82 mL HCL, and dilute to volume with water.

4.3 Buffer solution-disperse 163g EDTA in 200 water in 2L volumetric flask and add enough NH_4OH to dissolve. Dilute 60 mL 70.5% $HClO_4$ by pouring carefully into about 500 mL water and cool. Dissolve 50g La_2O_3 in $HClO_4$ solution. Add 8 drops methyl orange indicator to ammoniacal EDTA solution and add La_2O_3 solution to EDTA solution while stirring vigorously. If necessary, add NH_4OH to maintain alkalinity of above solution to methyl orange. Dilute to 2L.

5.0 REAGENT BLANK

Before proceeding with analysis, test purity of reagents as follows: Evaporate 4ml HNO_3 in crucible to dryness on hot plate or steam bath, dissolve residue in 1M HCL, and transfer to 25ml volumetric flask. Heat residue again successively with two 5 ml portions 1M HCL and add to flask. Cool, dilute to

volume with 1M HCL, and mix. Proceed with determination. Total reagent blank should be ≤10 µg Pb (equivalent to 0.4 mg/kg in material) for determinations at levels ≥1mg/kg. For determinations at < 1mg/kg, purify reagents to attain blank < 50% of limiting level of concern.

6.0 PREPARATION OF MATERIAL

Weigh about 25 g (to nearest 0.1g) test portion into crucible, **(3.3),** and dry 2h at 135 -150 ˚C. Transfer to cold, temperature controlled furnace and slowly raise temperature to 500˚C. Set, control and check for maintenance of 500˚C (temperature as low as 550˚C may cause loss of Pb). Ashes overnight (16h), remove crucible, let it cool to room temperature and cautiously add 2 mL HNO_3 and swirl. Evaporate carefully just to dryness on warm hot plate or steam bath. Transfer to cooled furnace, slowly raise temperature to 500ºC, and hold at this temperature for 1h,remove dish and cool. Repeat HNO_3 ashing, if necessary, to obtain clean, practically c-free ash. Add 10 mL 1M HCl and dissolve ash by heating cautiously on hot plate. Transfer to 25 mL volumetric flask. Heat ash residue again successively with two 5 mL portions 1M HCl and add to flask. Cool, dilute to volume with 1M HCl, and mix.

7.0 PREPARATION OF STANDARD CURVE

Transfer 0,1,3,5,15, 25 and 50 ml Pb working solution,(**4.2)**,to separate 50 mL volumetric flasks and dilute to volume with 1M HCl (0,0.2,0.6,0.6,1.0,3.0,5.0 and 10.0 µg Pb /ml, respectively).set spectrophotometer to previously established optimum conditions for maximum signal at 283.3nm.Use air – C_2H_2 flow rates recommended by manufacturer for standard conditions for Pb. For digital concentration readout, calibrate in concentration mode with solutions containing 0.2 and 10.0 µg Pb/ mL. Record concentration directly after calibration of instrument. For strip chart readout, set amplification to give ≥1% absorption reading for 0.2 µg/ mL working solution and prepare standards curve of A against µg Pb/ mL

8.0 DETERMINATION

Use aliquot of test solution, (**6.0),** and proceed as in **8.1 or 8.2.** Treat reagent blank, (**5.0)** as test solution and subtract reading from a of test solutions.

8.1 Clear solutions

Determine a of test and standards solutions as in **7.0**, using following sequence 3 times: Read standard solution first, then test solution, alternating until all test and standard solutions have been read. When many test solutions are to be analysed, standard solutions may be read after series of 3 test solutions instead of after each.

$$\text{Pb, ppm}(\mu g/g) = \frac{\mu g\ Pb/\ mL\ \text{test solution} \times 25}{g\ \text{test portion}}$$

8.2 Cloudy solutions

Proceed as in **8.1**, but add 1 mL buffer solution**, (4.3),** to aliquots of test and standard solutions before reading.

If additional dilutions are necessary or if buffer is added:

Pb, ppm (µg/g) =µg Pb mL l test solution x (mL diluted test solution/ml original aliquot) x (25/g test portion)