8.14 RECOMMENDED METHOD OF ANALYSIS FOR THE DETERMINATION OF TOTAL IODINE IN FOODS OTHER THAN MILK

101st session of Council, June 1986

1 SCOPE

This method utilises the catalytic effect of iodide on the destruction of iron (III) thiocyanate by nitrite.

2 APPLICATION

This method is applicable to most foods in general.

3 PRINCIPLE

Ferric ions form a stable orange-red complex with thiocyanate ions. This complex is reduced to a ferrous complex by nitrite ions in the presence of iodide as a catalyst.

The ferrous complex is colourless. At low nanogram quantities of iodide the reduction is proportional to iodide concentration.

4 REAGENTS AND MATERIALS

4.1 General requirements

4.1.1 All the reagents should be of analytical grade and distilled water should be used in preference to deionised water.

4.2 Reagents

4.2.1 **Standard iodide solutions**

4.2.1.1 Stock standard iodide 1 g/L:
Dissolve 1.3080 grams potassium iodide in distilled water and dilute to 1 litre in a volumetric flask.

4.2.1.2 Working standards:
Dilute appropriately stock standard to obtain working standards of 4, 8, 12, 16 and 20 µg iodide per mL.

4.2.2 **Potassium carbonate (30% m/V):**
Dissolve 30 g of potassium carbonate in water and dilute to 100 mL.

4.2.3 **Zinc sulphate (10% m/V):**
Dissolve 10 g of zinc sulphate ($ZnSO_4.7H_2O$) in water and dilute in 100 mL.

4.2.4 **Potassium thiocyanate (0.023% m/V):**
Dissolve 0.23 g of potassium thiocyanate in water and dilute to 1 L.

4.2.5 **Sodium nitrite (2.07% m/V):**
Dissolve 2.07 g of sodium nitrite in water and dilute to 100 mL (Stable only for 1 day).

4.2.6 **Ammonium iron sulphate reagent:**
Dissolve 77 g of ammonium iron (III) sulphate ($NH_4 Fe (SO_4)_2 . 12H_2O$) in approximately 400 mL of water. Add 167 $\pm$ 1 mL of nitric acid (S.G 1.42) and dilute to 1 L, warming until all traces of solid dissolve.

5 APPARATUS

5.1 Muffle furnace which can be thermostatically controlled to 550°C

5.2 Porcelain crucibles, 45mm diameter, with lids.

5.3 Centrifuge, with centrifuge tubes

5.4 Vortex mixer

6 PROCEDURE

6.1 Dry ashing

6.1.1 Into a clean, dry crucible, accurately weigh approximately 1 g of sample with a content of iodine not exceeding 1 ug.

6.1.2 Add 1 mL of potassium carbonate solution and then 1 mL of zinc sulphate solution. Slurry the mixture with a glass rod and wash any residue left on the rod back into the crucible.

6.1.3 Dry the crucible at 95°C overnight.

6.1.4 Place the dried crucible in a muffle furnace at 100°C and cover with a lid.

6.1.5 Raise the temperature evenly to 550°C in approximately 90 mins and maintain at this temperature for 1 hour.

6.1.6 Remove the crucible and allow to cool to room temperature.

6.1.7 Add 1 mL of zinc sulphate solution and slurry the charred residue.

6.1.8 Repeat the drying and ashing as before.

6.2 Centrifugation

6.2.1 Transfer the cooled ash, normally white or grey in colour to a centrifuge tube with 50 ± 0.5 mL of distilled water.

6.2.2 Centrifuge for 5 mins to settle insolubles.

6.2.3 Decant about half of the clear solution and store in a clean polyethylene container prior to analysis.

6.3 Photometric determination

6.3.1 Pipette 4 mL of each working standard, containing 4 to 20 ~~µg~~ ng/mL iodide, into a 15 x 1.5 cm test tube.

6.3.2 A reagent blank is prepared using distilled water.

6.3.3 To each tube add 1 mL of distilled water, 1 mL of potassium thiocyanate solution and 2 mL of ammonium iron (III) sulphate solution.

6.3.4 Mix the contents of each tube on a vortex mixer.

6.3.5 Add, at 90 second intervals, 1 mL sodium nitrite solution and mix again on a vortex mixer.

6.3.6 After 20 minutes, at 90 second intervals measure the absorbance of each solution at 450 nm.

6.3.7 Sample digestion solutions are treated by the above procedure.

6.3.8 It is essential that all solutions should be maintained at the same temperature.

6.3.9 All photometric determinations should be carried out in duplicate.

7 CALCULATION AND EXPRESSION OF RESULTS

7.1 Prepare a calibration curve from the absorbances of standard solutions and iodide concentration.

7.2 From the calibration curve read the iodide concentration of sample solutions and reagent blank.

7.3 Calculate the iodide concentration ug/kg, using the following equation.

7.4 Iodide Content (ug/kg) = $\left(\frac{(A-B) \times 50}{W} \right)$

Where A = Iodide content of sample solution (~~ug~~/mL)

B = Iodide content of reagent blank (~~ug~~/mL)

W = Weight of sample (g)

8 REFERENCES

8.1 Semi-Automated Method for the Determination of Total Iodine in Food (1980) R.E.D. Moxon and E.J. Dixon: Analyst 105, 344-52.

8.2 The Determination of Iodine in Dairy Products (1984) A.M. Chaffey and T.L. Lewis: AGAL Report No 95.