Determination of Methylmercury Compounds in Fish

1. SCOPE

The method is designed for methylmercury compounds in fish and other marine organisms at levels down to about 0.01mg/kg* but can also be used, with modifications where necessary having regard to recovery levels, for other foods. Normally, the sample is first examined for total mercury residues. Examination for methylmercury compounds is unnecessary if no residues are found.

2. FIELD OF APPLICATION

The procedure has been mainly designed for a variety of fish, including tuna, salmon, pike, cod and herring and is capable of estimating 0.02mg/kg of methylmercuric chloride in a 2g sample.

3. PRINCIPLE OF METHOD

Methylmercury compounds are extracted with benzene, extracted into aqueous cysteine solution, acidified, back extracted into benzene and the benzene extract is examined gas chromatographically.

4. REAGENTS

Alkaline sodium sulphate solution, 2N Sodium hydroxide in 10% w/v sodium sulphate solution. Benzene, redistilled from charcoal. Hydrochloric acid, concentrated. Cysteine solution, 1% w/v–dissolve 0.5g cysteine hydrochloride and 0.4g sodium acetate in 50ml 10% sodium sulphate solution. Methylmercuric chloride, standard solutions in benzene.

5. APPARATUS

Conical flasks, 100ml; Steam bath; Separators, 100ml; Büchner filter, 10cm; Kuderna-Danish evaporator; Gas chromatographic column; Polyethylene glycol succinate; Chromosorb G, acid-washed DCMS treated, 80-100 mesh; Nitrogen carrier gas, oxygen-free.

*Based on a sample weight of 5g.

6. PROCEDURE

6.1 *Extraction*

Place 2g of homogenized sample in a 100ml conical flask, add 10ml 2N sodium hydroxide in 10% sodium sulphate solution and disperse on a steam bath (up to 20min). Transfer to 100ml separator, add 25ml benzene, 6ml concentrated hydrochloric acid, shake for 2min. Centrifuge, or filter under suction through a 10cm Whatman 1 paper; transfer to another 100ml separator, warming on the steam bath gently as necessary to break any stable emulsion. Separate the aqueous layer, extract with 20ml benzene, combine the benzene extracts and concentrate to not less than 4ml in a Kuderna-Danish evaporator in the steam bath. Extract.

6.2 *Clean-up (7.1)*

Extract with 2 x 1.5ml cysteine solution, combine the extracts, add 1ml concentrated hydrochloric acid and extract with a measured volume (5.0 or 10.0ml) of benzene.

6.3 *Gas chromatography*

Prepare a glass column 1.5m x 3mm (internal diameter) by packing with 2% diethylene glycol succinate on 80-100 mesh chromosorb G for operation at 160°C, using nitrogen carrier gas at a flow rate adjusted to give a retention time of about 3min for methylmercuric chloride. Examine 5 microlitre portions of the purified benzene extract obtained above. Prepare a calibration curve using standard methylmercuric chloride solutions. A peak height of about 5% full scale deflection should be obtained with 0.05 methylmercuric chloride in a 5ml volume.

7. REFERENCES

7.1 Westöö, G. *Acta Chem. Scand.*, **21**, 1,790, 1967.