

ANNEX

DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE IDENTIFICATION AND DETERMINATION OF ORGANOMERCURY COMPOUNDS

SCOPE AND FIELD OF APPLICATION

B. DETERMINATION

5. PROCEDURE

5.1. *Breakdown*

- 5.1.1. Weigh accurately 150 mg of the sample (m). Add 10 ml of nitric acid (3.1) and leave to digest for three hours in an airtight flask in a water bath at 55 °C, shaking at regular intervals. At the same time, carry out a blank test on the reagents.
- 5.1.2. After cooling, add 10 ml of sulphuric acid (3.2) and return to the water bath at 55 °C for 30 minutes.
- 5.1.3. Place the flask in an ice bath and add carefully 20 ml of water (3.3).
- 5.1.4. Adding 2 ml aliquots of 7 % potassium permanganate solution (3.4) until the solution remains coloured. Return to the water bath at 55 °C for a further 15 minutes.
- 5.1.5. Add 4 ml of dipotassium peroxodisulphate solution (3.6). Continue to warm in the water bath at 55 °C for 30 minutes.
- 5.1.6. Allow to cool and transfer the contents of the flask into a 100 ml standard flask. Rinse the flask with 5 ml of hydroxylammonium chloride (3.5) and then rinse four times with 10 ml of water (3.3). The solution should be completely decolorized. Make up to the mark with water (3.3).