ANNEX

DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE
IDENTIFICATION AND DETERMINATION OF ORGANOMERCURY COMPOUNDS
SCOPE AND FIELD OF APPLICATION

B. **DETERMINATION**

5. PROCEDURE

Take all normal precautions for trace mercury analysis.

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 ^{o}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 ^{o}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 ^{o}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 ^{o}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).

5.2. ***Determination***

5.2.1. Place 10 ml of the test solution (5.1.6) in the glass vessel for the cold vapour mercury determination (4.2). Dilute with 100 ml of water (3.3) and subsequently 5 ml of sulphuric acid (3.2) and 5 ml of tin dichloride solution (3.7). Mix after each addition. Wait 30 seconds to reduce all ionic mercury to the metallic state and take a reading (n).

5.2.2. Place some palladium dichloride impregnated glass wool (3.9) between the mercury reduction vessel and the flow cell of the instrument (4.2). Repeat 5.2.1 and record the reading. If the reading is not zero mineralization was incomplete and analysis must be repeated.