

## ANNEX

**DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE  
DETERMINATION OF TOTAL FLUORINE IN DENTAL CREAMS**

## 4. REAGENTS

All reagents should be of analytical purity.

- 4.1. Sodium fluoride, dried at 120 °C to constant mass.
- 4.2. Water, double distilled or equivalent quality.
- 4.3. Hydrochloric acid,  $d_4^{20} = 1,19$  g/ml.
- 4.4. Cyclohexane (CH).
- 4.5. Xylene with no peaks in the chromatogram prior to the solvent peak when chromatographed under the same conditions as the sample (6.1). If necessary purify by distillation (5.8).
- 4.6. Chlorotriethylsilane (TECS Merck or an equivalent).
- 4.7. ***Fluorine standard solutions***
  - 4.7.1. Stock solution, 0,250 mg F<sup>-</sup>/ml. Weigh accurately 138,1 mg of sodium fluoride (4.1) and dissolve in water (4.2). Quantitatively transfer the solution into a 250 ml volumetric flask (5.5). Dilute to the mark with water (4.2) and mix.
  - 4.7.2. Diluted stock solution, 0,050 mg F<sup>-</sup>/ml. Transfer by pipette 20 ml of the stock solution (4.7.1) into a 100 ml volumetric flask (5.5). Dilute to the mark with water and mix.
- 4.8. ***Internal standard solution***

Mix 1 ml of cyclohexane (4.4) and 5 ml of xylene (4.5).
- 4.9. ***Chlorotriethylsilane/internal standard solution***

Transfer, by pipette (5.7), 0,6 ml of TECS (4.6) and 0,12 ml of the internal standard solution (4.8) into a 10 ml volumetric flask. Dilute with xylene (4.5) to the mark and mix. Prepare fresh daily.
- 4.10. Perchloric acid, 70 % (m/v).
- 4.11. Perchloric acid, 20 % (m/v) in water (4.2).