

ANNEX

DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE
IDENTIFICATION AND DETERMINATION OF QUINOLIN-8-OL AND BIS(8-HYDROXYQUINOLINIUM) SULPHATE

4. REAGENTS

All reagents should be of analytical purity.

- 4.1. Quinolin-8-ol.
- 4.2. Benzene. In view of its toxicity great care must be taken when working with benzene.
- 4.3. Chloroform.
- 4.4. Aqueous sodium hydroxide, 50 % (m/m) solution.
- 4.5. Copper sulphate pentahydrate.
- 4.6. Potassium sodium tartrate.
- 4.7. M hydrochloric acid.
- 4.8. 0,5 M sulphuric acid.
- 4.9. M sodium hydroxide solution.
- 4.10. Ethanol.
- 4.11. Butan-1-ol.
- 4.12. Glacial acetic acid.
- 4.13. 0,1 hydrochloric acid.
- 4.14 'Celite 545' or equivalent.
- 4.15. **Standard solutions**
 - 4.15.1. Weigh 100 mg of quinolin-8-ol (4.1) into a 100 ml standard flask. Dissolve in a little sulphuric acid (4.8) Make up to the mark with sulphuric acid (4.8).
 - 4.15.2. Weigh 100 mg of quinolin-8-ol into a 100 ml standard flask. Dissolve in ethanol (4.10). Make up to the mark with ethanol (4.10) and mix.
- 4.16. **Fehling's solution**

Solution A

Weigh 7 g of copper sulphate pentahydrate (4.5) into a 100 ml standard flask.

Dissolve in a little water. Make up to the mark with water and mix.

Solution B

Weigh 35 g of potassium sodium tartrate (4.6) into a 100 ml standard flask. Dissolve in 50 ml of water. Add 20 ml of sodium hydroxide (4.4). Make up to the mark with water and mix. Immediately before use, pipette 10 ml of solution A and 10 ml of solution B into a 100 ml standard flask. Make up to the mark and mix.

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4.17. ***Eluting solvents for thin-layer chromatography***

- I : Butan-1-ol (4.11) /acetic acid (4.12) /water (80: 20: 20; v/v/v).
II : Chloroform (4.13) /acetic acid (4.12) (95: 5; v/v).

4.18. 2,6-dichloro-4-(chloroino)cyclohexa-2,5-dienone, 1 % (m/v) solution in ethanol (4.10).

4.19. Sodium carbonate, 1 % (m/v) solution in water.

4.20. Ethanol (4.10), 30 % (v/v) solution in water.

4.21. Disodium dihydrogen ethylenediaminetetraacetate, 5 % (m/v) solution in water.

4.22. ***Buffer solution, pH 7***

Weigh 27 g of potassium dihydrogenorthophosphate anhydrous and 70 g of dipotassium hydrogenorthophosphate trihydrate into a one litre standard flask. Make up to the mark with water.

4.23. ***Prepared thin-layer plates***

Ready made thin-layer plates of a thickness of 0,25 mm (e.g. Merck Kieselgel 60 or equivalent). Before use, spray with 10 ml of reagent (4.21) and dry at 80 °C.