PARAQUAT 56

$$H_3C-N^+$$
 N^+
 CH_3

ISO common name Paraquat

Chemical name 1,1'-Dimethyl-4,4'-bipyridinium (IUPAC);

1,1'-dimethyl-4,4'-bipyridyldiylium

(CA; 4685-14-7)

Empirical formula $C_{12}H_{14}N_2$

C₁₂H₁₄Cl₂N₂ (paraquat dichloride)

RMM 186.3

257.2 (paraquat dichloride)

m.p. Paraquat dichloride decomposes at about

300 °C

Solubility In water (paraquat dichloride) very soluble;

slightly soluble in short-chain alcohols, insoluble

in hydrocarbons

Description Paraquat dichloride forms colourless crystals

Stability Stable in acidic media, but unstable in alkaline

media. Decomposed by UV light in aqueous

solutions

Formulations Aqueous solutions

Note Paraquat dichloride (CA; 1910-42-5),

paraquat di(methylsulphate) (CA; 2074-50-2)

PARAQUAT 56

PARAQUAT SALT AQUEOUS SOLUTIONS *56/SL/M/-

1 Sampling. Take at least 500 ml.

2 Identity test. -

3 Paraquat

OUTLINE OF METHOD Paraquat is reduced by alkaline sodium dithionite solution to a blue, free radical. Its absorbence is measured at 600 nm against a permanent blue glass reference standard and the paraquat content obtained from a calibration graph prepared at the same time from solutions of known paraquat content.

REAGENTS

Sodium dithionite 1% m/m solution in sodium hydroxide solution, 0.1 mol/l. Do not keep the solution for more than 3 h. Solid sodium dithionite is unstable in presence of moisture. Store in 25 ml bottles with air tight closures and keep these in a vacuum desiccator containing an efficient desiccant (e.g., "Drierite"). Paraquat dichloride pure, PP 56

Calibration solution. Dissolve pure paraquat dichloride previously dried to constant weight at 100 to 120 °C (0.1728 g) in water, dilute to 500 ml with water, and mix. Prepare the solution fresh as required. One ml of standard solution contains 0.25 mg of paraquat.

APPARATUS

Spectrophotometer with 1 cm glass cuvettes; a suitable filter is fitted into the spectrophotometer cell carrier in contact with one of the faces of the reference cell. A piece cut from a Chance B2A blue glass filter, 2.8 mm thick (obtainable from Precision Optical Instruments (Fulham) Ltd), is suitable but any permanent material, e.g., a neutral glass filter or metal gauze, giving an absorbence of about 1.5 at 600 nm may be substituted.

Volumetric flasks 100, 250 and 500 ml Burette 25 ml Pipettes 10 ml

Note: DO NOT wash the apparatus with acetone as any traces left will interfere with the method.

^{*} CIPAC method 1968, revised 1972. Prepared by the Diquat and Paraquat Subcomittee of PAC-GB; Chairman M G Ashley (Plant

PROCEDURE

(a) Preparation of the sample solution. Weigh (to the nearest 0.1 mg) enough of the well mixed sample (w g) to contain about 1.6 to 1.8 g of paraquat, transfer to a 250 ml volumetric flask, dilute to the mark with water, and mix. (Solution A). Pipette "Solution A" (10.00 ml) into a 250 ml volumetric flask, dilute to the mark with water, and mix. (Solution B).

(b) Determination. Measure 10.0 ml of "Solution B" and 9.0, 10.0, 11.0, 12.0 and 13.0 ml portions of paraquat calibration solution containing 2.25, 2.5, 2.75, 3.0 and 3.25 mg of paraquat into 100 ml volumetric flasks, and dilute the contents of each flask to about 80 ml with water. Add the sodium dithionite solution (10 ml) to the contents of one of the flasks and make up to the mark with water. Mix the contents by inverting the flask end-over-end three times at such a speed that the air bubble travels from one end to the other. Do not shake the flask vigorously as this tends to cause fading of the colour due to oxidation. Immediately transfer the solution to a 1 cm cuvette and measure the absorbence of the solution at 600 nm relative to the permanent reference standard. Similarly treat each flask in turn, completing the absorbence measurement without delay, before adding dithionite to the next solution. Draw the graph relating differential absorbences of the calibration solution to their paraquat contents in mg and read off the paraquat content of the sample solution (y).

(c) Calculation

Paraquat content
$$=\frac{625 y}{w}$$
 g/kg

where:

y = paraquat content of the final sample solution, read from the calibration graph (mg)

w = mass of sample taken (g)

4 Free 2,2'-bipyridyl.

REAGENTS AND As for 55/SL/M/4. CIPAC H, p 161.

except : substitute for 2,2'-dipyridyl: 4,4'-bipyridyl RE 60 zone refined.

PROCEDURE As for 55/SL/M/4. CIPAC H, p 161 except: substitute 4,4'-bipyridyl for 2,2'-dipyridyl throughout.