Set the zero of the instrument using *methyl isobutyl ketone R* treated as described for the test solution without the substance to be examined. Measure the absorbance at 283.3 nm using a lead hollow-cathode lamp as source of radiation and an air-acetylene flame.

The substance to be examined contains not more than 0.5 ppm of lead, unless otherwise prescribed.

01/2008:20411

2.4.11. PHOSPHATES

To 100 ml of the solution prepared and, if necessary, neutralised as prescribed add 4 ml of *sulphomolybdic reagent R3*. Shake and add 0.1 ml of *stannous chloride solution R1*. Prepare a standard in the same manner using 2 ml of *phosphate standard solution (5 ppm PO_4) R* and 98 ml of *water R*. After 10 min, compare the colours using 20 ml of each solution.

Any colour in the test solution is not more intense than that in the standard.

01/2008:20412

2.4.12. POTASSIUM

To 10 ml of the prescribed solution add 2 ml of a freshly prepared 10 g/l solution of *sodium tetraphenylborate R*. Prepare a standard in the same manner using a mixture of 5 ml of *potassium standard solution (20 ppm K) R* and 5 ml of *water R*.

After 5 min, any opalescence in the test solution is not more intense than that in the standard.

01/2008:20413

2.4.13. SULPHATES

All solutions used for this test must be prepared with distilled water R.

Add 3 ml of a 250 g/l solution of *barium chloride R* to 4.5 ml of *sulphate standard solution (10 ppm SO_4) R1*. Shake and allow to stand for 1 min. To 2.5 ml of this solution, add 15 ml of the solution to be examined and 0.5 ml of *acetic acid R*. Prepare a standard in the same manner using 15 ml of *sulphate standard solution (10 ppm SO_4) R* instead of the solution to be examined.

After 5 min, any opalescence in the test solution is not more intense than that in the standard.

01/2008:20414

2.4.14. SULPHATED ASH

Ignite a suitable crucible (for example, silica, platinum, porcelain or quartz) at 600 ± 50 °C for 30 min, allow to cool in a desiccator over silica gel or other suitable desiccant and weigh. Place the prescribed amount of the substance to be examined in the crucible and weigh. Moisten the substance to be examined with a small amount of *sulphuric acid R* (usually 1 ml) and heat gently at as low a temperature as practicable until the sample is thoroughly charred. After cooling, moisten the residue with a small amount of *sulphuric acid R* (usually 1 ml), heat gently until white fumes are no longer evolved and ignite at 600 ± 50 °C until the residue is completely incinerated. Ensure that flames are not produced at any time during the procedure. Allow the crucible to cool in a desiccator over silica gel or other suitable desiccant, weigh it again and calculate the percentage of residue.

If the amount of the residue so obtained exceeds the prescribed limit, repeat the moistening with *sulphuric acid R* and ignition, as previously, for 30 min periods until 2 consecutive weighings do not differ by more than 0.5 mg or until the percentage of residue complies with the prescribed limit.

The amount of substance used for the test (usually 1-2 g) is chosen so that at the prescribed limit the mass of the residue (usually about 1 mg) can be measured with sufficient accuracy.

01/2008:20415
corrected 6.0

2.4.15. NICKEL IN POLYOLS

Determine the nickel by atomic absorption spectrometry (*2.2.23, Method II*).

Test solution. Dissolve 20.0 g of the substance to be examined in a mixture of equal volumes of *dilute acetic acid R* and *water R* and dilute to 100.0 ml with the same mixture of solvents. Add 2.0 ml of a saturated solution of *ammonium pyrrolidinedithiocarbamate R* (about 10 g/l) and 10.0 ml of *methyl isobutyl ketone R* and then shake for 30 s protected from bright light. Allow the layers to separate and use the methyl isobutyl ketone layer.

Reference solutions. Prepare 3 reference solutions in the same manner as the test solution but adding 0.5 ml, 1.0 ml and 1.5 ml respectively of *nickel standard solution (10 ppm Ni) R* in addition to the 20.0 g of the substance to be examined.

Set the zero of the instrument using *methyl isobutyl ketone R* treated as described for preparation of the test solution omitting the substance to be examined. Measure the absorbance at 232.0 nm using a nickel hollow-cathode lamp as source of radiation and an air-acetylene flame.

The substance to be examined contains not more than 1.0 ppm of nickel, unless otherwise prescribed.

01/2008:20416

2.4.16. TOTAL ASH

Heat a silica or platinum crucible to redness for 30 min, allow to cool in a desiccator and weigh. Unless otherwise prescribed, evenly distribute 1.00 g of the substance or the powdered herbal drug to be examined in the crucible. Dry at 100 °C to 105 °C for 1 h and ignite to constant mass in a muffle furnace at 600 °C ± 25 °C, allowing the crucible to cool in a desiccator after each ignition. Flames should not be produced at any time during the procedure. If after prolonged ignition the ash still contains black particles, take up with hot water, filter through an ashless filter paper and ignite the residue and the filter paper. Combine the filtrate with the ash, carefully evaporate to dryness and ignite to constant mass.