of a given solvent.) Add to 1.2 mL of *thioacetamide reagent R*. Mix immediately and allow to stand for 2 min. Filter the solutions through a suitable membrane filter (nominal pore size 0.45 µm). Compare the spots on the filters obtained with the different solutions.

System suitability: the spot obtained with the reference solution shows a brownish-black colour compared to the spot obtained with the blank solution.

Result: the brownish-black colour of the spot obtained with the test solution is not more intense than that of the spot obtained with the reference solution.

01/2008:20409

2.4.9. IRON

Dissolve the prescribed quantity of the substance to be examined in *water R* and dilute to 10 mL with the same solvent or use 10 mL of the prescribed solution. Add 2 mL of a 200 g/L solution of *citric acid monohydrate R* and 0.1 mL of *thioglycollic acid R*. Mix, make alkaline with *ammonia R* and dilute to 20 mL with *water R*. Prepare a standard in the same manner, using 10 mL of *iron standard solution (1 ppm Fe) R*.

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

01/2008:20410

2.4.10. LEAD IN SUGARS

Determine the lead 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 clear 10 g/L solution of *ammonium pyrrolidinedithiocarbamate R* 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 *lead standard solution (10 ppm Pb) 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 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 *sulfomolybdic 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 corrected 8.0

2.4.13. SULFATES

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 *sulfate standard solution (10 ppm SO_4) R1*. Shake and allow to stand for 1 min. To 2.5 mL of this suspension add 15 mL of the prescribed solution and 0.5 mL of *acetic acid R*. Prepare a standard in the same manner using 15 mL of *sulfate standard solution (10 ppm SO_4) R* instead of the prescribed solution.

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

04/2010:20414

2.4.14. SULFATED ASH(1)

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 *sulfuric 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 *sulfuric 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 *sulfuric 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.

(1) This chapter has undergone pharmacopoeial harmonisation. See chapter *5.8. Pharmacopoeial harmonisation.*