CADMIUM, COPPER, IRON, LEAD, NICKEL AND ZINC

Measure the content of cadmium, copper, iron, lead, nickel and zinc by the standard additions method (*2.2.23, Method II*), using reference solutions of each heavy metal and the instrumental parameters described in Table 2.4.27.-1.

The absorbance value of the blank solution is automatically subtracted from the value obtained with the test solution.

Table 2.4.27.-1

		Cd	Cu	Fe	Ni	Pb	Zn
Wavelength	nm	228.8	324.8	248.3	232	283.5	213.9
Slit width	nm	0.5	0.5	0.2	0.2	0.5	0.5
Lamp current	mA	6	7	5	10	5	7
Ignition temperature	°C	800	800	800	800	800	800
Atomisation temperature	°C	1800	2300	2300	2500	2200	2000
Background corrector		on	off	off	off	off	off
Nitrogen flow	l/min	3	3	3	3	3	3

ARSENIC AND MERCURY

Measure the content of arsenic and mercury in comparison with the reference solutions of arsenic or mercury at a known concentration by direct calibration (*2.2.23, Method I*) using an automated continuous-flow hydride vapour generation system.

The absorbance value of the blank solution is automatically subtracted from the value obtained with the test solution.

Arsenic

Sample solution. To 19.0 ml of the test solution or of the blank solution as prescribed above, add 1 ml of a 200 g/l solution of *potassium iodide R*. Allow the test solution to stand at room temperature for about 50 min or at 70 °C for about 4 min.

Acid reagent. Heavy metal-free hydrochloric acid R.

Reducing reagent. A 6 g/l solution of *sodium tetrahydroborate R* in a 5 g/l solution of *sodium hydroxide R*.

The instrumental parameters in Table 2.4.27.-2 may be used.

Mercury

Sample solution. Test solution or blank solution, as prescribed above.

Acid reagent. A 515 g/l solution of *heavy metal-free hydrochloric acid R*.

Reducing reagent. A 10 g/l solution of *stannous chloride R* in *dilute heavy metal-free hydrochloric acid R*.

The instrumental parameters in Table 2.4.27.-2 may be used.

Table 2.4.27.-2

		As	Hg
Wavelength	nm	193.7	253.7
Slit width	nm	0.2	0.5
Lamp current	mA	10	4
Acid reagent flow rate	ml/min	1.0	1.0
Reducing reagent flow rate	ml/min	1.0	1.0
Sample solution flow rate	ml/min	7.0	7.0
Absorption cell		Quartz (heated)	Quartz (unheated)
Background corrector		off	off
Nitrogen flow rate	l/min	0.1	0.1

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2.4.28. 2-ETHYLHEXANOIC ACID

Examine by gas chromatography (*2.2.28*), using *3-cyclohexylpropionic acid R* as the internal standard.

Internal standard solution. Dissolve 100 mg of *3-cyclohexylpropionic acid R* in *cyclohexane R* and dilute to 100 ml with the same solvent.

Test solution. To 0.300 g of the substance to be examined, add 4.0 ml of a 33 per cent *V/V* solution of *hydrochloric acid R*. Shake vigorously for 1 min with 1.0 ml of the internal standard solution. Allow the phases to separate (if necessary, centrifuge for a better separation). Use the upper layer.

Reference solution. Dissolve 75.0 mg of *2-ethylhexanoic acid R* in the internal standard solution and dilute to 50.0 ml with the same solution. To 1.0 ml of the solution add 4.0 ml of a 33 per cent *V/V* solution of *hydrochloric acid R*. Shake vigorously for 1 min. Allow the phases to separate (if necessary, centrifuge for a better separation). Use the upper layer.

The chromatographic procedure may be carried out using:

- a wide-bore fused-silica column 10 m long and 0.53 mm in internal diameter coated with *macrogol 20 000 2-nitroterephthalate R* (film thickness 1.0 µm),
- *helium for chromatography R* as the carrier gas at a flow rate of 10 ml/min,
- a flame-ionisation detector,

with the following temperature programme:

	Time (min)	Temperature (°C)	Rate (°C/min)	Comment
Column	0 - 2	40	–	isothermal
	2 - 7.3	40 → 200	30	linear gradient
	7.3 - 10.3	200	–	isothermal
Injection port		200		
Detector		300		

Inject 1 µl of the test solution and 1 µl of the reference solution.

The test is not valid unless the resolution between the peaks corresponding to 2-ethylhexanoic acid (first peak) and the internal standard is at least 2.0.

Calculate the percentage content of 2-ethylhexanoic acid from the expression:

$$\frac{A_T \times I_R \times m_R \times 2}{A_R \times I_T \times m_T}$$

A_T = area of the peak corresponding to 2-ethylhexanoic acid in the chromatogram obtained with the test solution,

A_R = area of the peak corresponding to 2-ethylhexanoic acid in the chromatogram obtained with the reference solution,

I_T = area of the peak corresponding to the internal standard in the chromatogram obtained with the test solution,

I_R = area of the peak corresponding to the internal standard in the chromatogram obtained with the reference solution,

m_T = mass of the substance to be examined in the test solution, in grams,

m_R = mass of 2-ethylhexanoic acid in the reference solution, in grams.