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# ICP-spectrometric determination of foreign atoms in Zr metal- or ZrH<sub>2</sub>-powder

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## Basic principle

The sample is subjected to an appropriate disintegration process to give rise to a complete dissolution. Foreign elements are then determined by flame-emission spectrometry using inductively coupled plasma (ICP).

## Test procedure

### Sample preparation

The sample initially has to be dissolved. The choice of a suitable disintegration process depends on the composition of the respective sample and the type of the ion which is to be determined.

An adequate disintegration process for Fe, Al, Ca, Mg, Ti, Hf, Cr, Ni, Mn and Co in Zr metal- or ZrH<sub>2</sub>-powder is described below:

0.500 g of the sample is weighed into a platinum dish and suspended with approx. 20 mL distilled water. 3 mL concentrated hydrofluoric acid are slowly and cautiously added. The sample heats up remarkably accompanied by the formation of hydrogen. 5 mL concentrated sulfuric acid and 1 mL concentrated nitric acid are subsequently added to the suspension. Volatile compounds are fumed off until the residue is still slightly moist with sulfuric acid.

After cooling down, approx. 40 mL distilled water and 15 mL hydrochloric acid are added to the platinum dish, which is warmed until the sample is completely dissolved. The solution is transferred into a 100 mL volumetric flask and once it is cooled down is filled up with distilled water to the 100 mL mark.

A second platinum dish serves for the preparation of the blank sample: an analogous mixture of distilled water, hydrofluoric acid, sulfuric acid and nitric acid is fumed off and taken up with hydrochloric acid. The solution is transferred into a 100 mL volumetric flask which is then filled up to its mark with distilled water.

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## Realization of measurement

Measurements are carried out with an ICP spectrometer at fixed conditions in accordance with manufacturer's instructions. The elements are detected as follows:

Element	Wavelength [nm]	Minimum evaluable concentration [mg/L]
Fe	238.204 (simultaneous)	0.10
Al	167.080 (sequential)	0.05
Ca	393.366 (simultaneous)	0.02
Mg	279.553 (simultaneous)	0.02
Ti	334.941 (sequential)	0.10
Hf	282.022 (sequential)	0.50
Cr	283.563 (sequential)	0.10
Ni	352.454 (sequential)	0.20
Mn	257.610 (simultaneous)	0.05
Co	238.892 (sequential)	0.50
Si	251.611 (simultaneous)	0.10

Wavelength of the monitor line:

Ar 430.010 nm (simultaneous)

The system is calibrated with two aqueous hydrochloric multi-element standard solutions and a solution containing only distilled water acidified under the same conditions as the standard solutions. The sample and a blank sample are analyzed following the calibration. The analyte concentration is obtained as mg/L. If high precision is demanded, the results are calculated on the basis of the standard addition method.

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## Calculation

The calculation is carried out according to:

$$\frac{(a - b) \cdot 100 \text{ mL} \cdot 100}{c} = \% \text{ of the respective ion in the sample}$$

with:

- a: determined analyte concentration in the sample (mg/1000 mL)
- b: determined analyte concentration in the blank sample (mg/1000 mL)
- c: weight of original sample subjected to disintegration (mg)

The error of this analytical method depends on the absolute concentration of the respective foreign ion to be determined. The minimum quantitative limit of detection is based on plausibility studies and listed in the table above.

## Multi-element standard solutions

The standard solutions employed for calibration of the spectrometer are composed of the following elements:

	Standard 1 [mg/L]	Standard 2 [mg/L]
Fe	2	10
Al	2	10
Ca	2	10
Mg	5	20
Ti	10	20
Hf	10	50
Cr	0.5	2
Ni	2	10
Mn	0.5	2
Co	2	10

The standard solutions each have a total volume of 100 mL including 15 mL concentrated hydrochloric acid and 1 mL concentrated sulfuric acid.

Distilled water containing the same concentrations of hydrochloric and sulfuric acid as the standard solutions is used as a zero-solution.

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