DX-720

Routine determination of hazardous compounds

Atomic absorption spectrometry and WEEE, RoHS, ElektroG

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tomic absorption spectrometry is primarily suitable for quantitative determination of hazardous compounds such as lead, cadmium, mercury and chromium in sample materials according to ElektroG (WEEE/RoHS). AAS is a relative method for quantification and is based on the elemental composition of the sample and absorption according to Lambert-Beer’s law. In principle, calibration curves are calculated in the appropriate concentration ranges for each element to be determined. The calibration curves are then used to evaluate all unknown samples. A prerequisite for accurate results is, however, that calibration standards and samples represent the same composition with respect to other elements and matrix. This prerequisite is not always met and can, therefore, lead to problems – for example when, in addition to the elemental absorption, background absorption of the matrix contributes to the signal.

Interferences such as molecular absorption, particulate caused scattering and spectral interferences caused by absorption line overlap can be eliminated via high-performance background compensation techniques. For complete compensation of all known AAS interferences in the flame- as well as in the electrothermal atomization modes, the high-speed self-reversal method is well established. Another widely used method, deuterium background compensation is, however, only usable in the wavelength range up to 420 nm, while self-reversal background compensation can be applied over the entire 185 – 900 nm range.

**Cadmium in polymers**

Quantitative determination of elemental cadmium in polymers was carried out using an AA-6300 Shimadzu atomic absorption spectrometer (Figure 1), which is equipped as standard with deuterium- and self-reversal background compensation modes. For electrothermal atomization, the highly sensitive GFA-EX7i graphite furnace with digital control was used.

The experimental results were obtained from standard solutions, diluted measuring solutions and dissolved reference materials. For sample preparation of polymers, several dissolution procedures are possible, for example dry ashing or microwave-assisted acidic digestion using nitric acid and, if necessary, hydrogen peroxide under addition of hydrofluoric acid.

Cadmium determination (Fig. 2) was carried out in a concentration range of 0.1 up to 0.4 mg/L using flame atomization and in the concentration range of 0.1 up to 0.4 μg/L using electrothermal atomization. Due to spectral interference of the cadmium line at 228.8 nm by arsenic and iron, the deuterium method can lead to overcompensation. In this case, the self-reversal method was applied for background compensation. In this way, AAS can be applied as a suitable routine analysis method for the reliable determination of cadmium and other hazardous compounds according to the ElektroG directive.

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**Table 2: Results of the calibration for Pb (Ls1)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Pb (Ls1)</th>
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<tbody>
<tr>
<td>Measuring time</td>
<td>300 s</td>
</tr>
<tr>
<td>LLD</td>
<td>24.8 ppm</td>
</tr>
</tbody>
</table>

**Table 3: Results of the repeated measurements**

The average value of 259.3 ± 7.4 ppm obtained via the EDX-720 reflects an excellent reproducibility (Table 3).

**Results**

The results show that even without any sample preparation, high accuracy and precision are attained already after a measuring time of 300 seconds. The EDX-720 is therefore, the ideal tool for fast analysis of elements ranging from sodium to uranium in solid and liquid samples. Without adjusting the method, one measurement can cover the entire concentration range from ppm up to 100%. The possibility of carrying out analyses without using standards (fundamental parameter method) enables the investigation of unknown samples with very high precision. In addition, the large sample compartment (300 mm internal diameter x 150 mm height) offers enough room for non-destructive analysis of most samples without the need for prior sample fractionation.

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