

Chromium (VI) analysis a

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Hardware and software for accurate determination of



Figure 2: 1,5-diphenyl carbazide

The European Union has officially announced in its official bulletin of 13 February 2003, new directives regulating electrical and electronic used equipment (WEEE, Waste Electrical and Electronic Equipment) as well as the Restriction of the use of certain Hazardous Substances in electrical and electronic equipment (RoHS). This marks the ratification of both directives in the EU (2002/95/EC and 2002/96/EC) which were adopted into national law in January 2005.

According to the RoHS guidelines, as of 1 July 2006, a threshold value will apply for lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls (PBB) and polybrominated diphenyl ethers (PBDE). However, RoHS does not apply to all ten equipment categories listed in WEEE. According to Article 2 of the RoHS directive the equipment categories 8 (medical devices) and 9 (monitoring and control instruments) as well as certain other equipment classes (computer servers, memory systems and network infrastructure in telecommunications) are currently exempted from the directives under RoHS guidelines.

Where the equipment has been on the market prior to July 2006, use of these prohibited substances is allowed in replacement parts for repair purposes. They are, however, banned in new equipment.

Determination of hexavalent chromium

X-ray fluorescence or atomic absorption spectrometry only enables determination of total chromium content. X-ray fluorescence is a fast technique for the determination of total chromium content and requires virtually no sample pretreatment. After dissolution of the sample, atomic absorption spectrometry in the flame atomisation mode can detect the elements cadmium, lead and chromium in a concentration range of 0.1 up to 5 mg/L

and using electrothermal atomisation mode in a concentration range of 0.1 up to 20 µg/L.

Photometric quantification of hexavalent chromium (Cr⁶⁺) can be carried out with the UVmini-1240 (Figure 1) using 1,5-diphenyl carbazide (Figure 2) as reactant to form colored chromium complexes.

This procedure is suitable for the determination of chromium (VI), primarily used as a corrosion-protective layer on metallic surfaces, but also found in small parts such as screws, washers and spring rings.

The sample material is eluted into a reaction vessel during a predefined timeframe. A blank value as well as the extinction at 540 nm is subsequently measured. Chromi-



Figure 1: UVmini-1240 with cuvette

According to the RoHS guidelines

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hazardous substances

um (VI) oxidizes 1.5-diphenyl carbazide to 1.5-diphenyl carbazone, forming a red/violet-colored complex with chromium. The extinction of the dye is linear with respect to the chromium (VI) concentration (Figures 3 and 4).

Pentavalent vanadium (V^{5+}), trivalent iron (Fe^{3+}) and tetravalent molybdenum (Mo^{4+}) also react with 1.5-diphenyl carbazide and, in the presence of the reaction solution, result in an apparently higher Cr^{6+} concentration. The concentrations of these ions must therefore be determined in a preliminary experiment.

Summary

UV-VIS spectrometry is highly suitable for fast and straightforward determination of hexavalent chromium and can be applied in routine analysis using the UVmini-1240. The UVmini offers the following advantages for the analysis of ions in aqueous solutions using the specially developed water analysis program pack:

- Fast and accurate analysis of selected ions in aqueous solutions.
- 55 measuring programs (optional water analysis program pack) for 34 different ions including hexavalent chromium.
- All analysis parameters including the analytical wavelength, calibration curve, measuring time etc. are adjusted automatically by the selected measuring program.

- After measurements are completed, the concentrations are displayed immediately.

The application example described above offers an overview of the current state-of-the-art in the determination of hazardous compounds in electrical and electronic equipment as well as electrical scrap materials according to WEEE and RoHS. The threshold values for lead, mercury, chromium, polybrominated biphenyls and polybrominated diphenyl ethers are determined as 1,000 mg/kg and for cadmium as 100 mg/kg, corresponding to concentrations already used in the earlier ELV directive.

Hardware and software for accurate determination of hazardous substances

Shimadzu offers an extensive product range featuring a complete hardware and software solution for accurate determination of hazardous substances, as well as the competence and know-how of a market leader in analytical instrumentation.

Please have a look at the European seminar tour dates on page 17.

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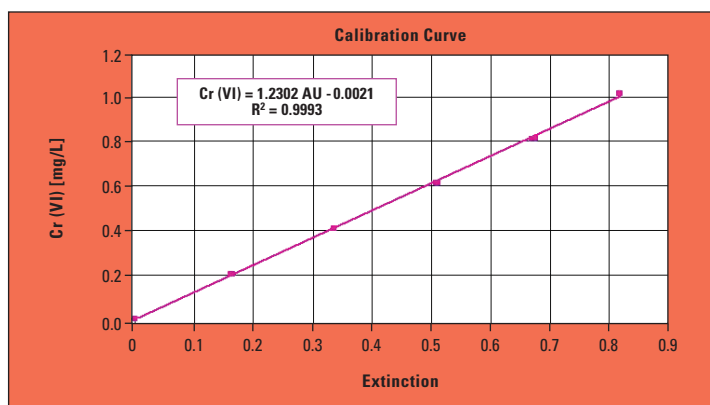


Figure 3: Chromium (VI) calibration curve

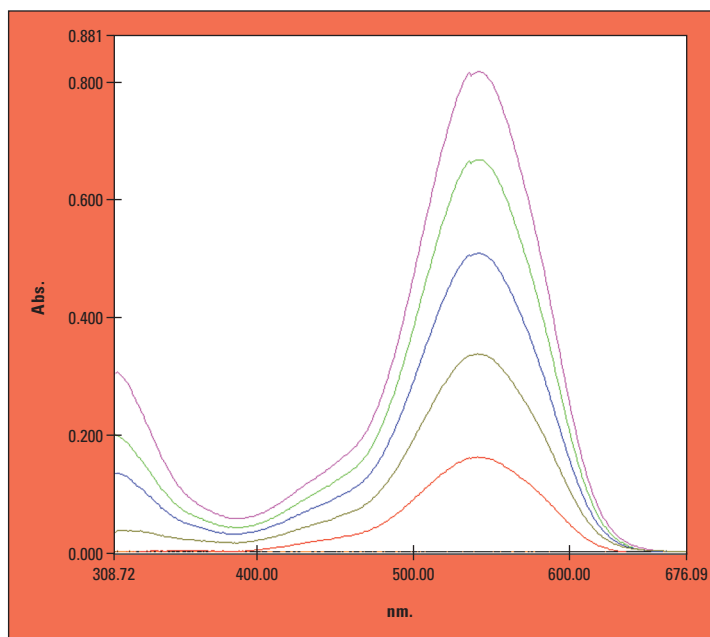


Figure 4: UV-VIS spectra of different concentrations of Cr^{6+} -solutions