

Application Bulletin



Of interest for:
Pottery industry
Foodstuffs inspection laboratories

No. 105/1 e

Determination of permissible lead and cadmium levels in crockery and glassware

Summary

Lead and cadmium are metals which, when absorbed by the human organism over a long period in quantities exceeding 0.5 mg Pb or 0.05 mg Cd per day, will lead to toxic symptoms. For this reason, efforts are now being made in various countries to eliminate the most likely sources of these metals, whether in the air (exhaust gases from leaded motor fuels) or from eating and drinking vessels, or at least to reduce the risks of their being absorbed by human beings.

These metals are determined by extraction with 4% acetic acid followed by either atomic absorption spectrometry (AAS) or polarography. Of the two alternative methods, polarography offers certain advantages over AAS. Firstly, the apparatus required is generally lower in first cost and cheaper to maintain, and secondly, the polarographic method enables Cu, Cd, Pb, As and Sb to be determined together in a single operation. This Bulletin describes the determination of the above metals by means of differential pulse polarography.

Apparatus

- ▶ 2.506.00XX Polarecord and 2.663.002X VA Stand

Reagents

- ▶ **4% acetic acid** Dissolve 40 g (38.1 mL) of glacial acetic acid, AR grade, in distilled water and make up to 1 litre.
- ▶ **Standard lead solution** Dissolve 1.8308 g $\text{Pb}(\text{OOCCH}_3)_2 \cdot 3\text{H}_2\text{O}$ in 4% acetic acid and make up to 1 litre.
(1 mL $\hat{=}$ 1 mg Pb)
- ▶ **Standard cadmium sol.** Dissolve 2.3177 g $\text{Cd}(\text{OOCCH}_3)_2 \cdot 3\text{H}_2\text{O}$ in 4% acetic acid and make up to 1 litre.
(1 mL $\hat{=}$ 1 mg Cd)
- ▶ **Standard copper sol.** Dissolve 3.1421 g $\text{Cu}(\text{OOCCH}_3)_2 \cdot \text{H}_2\text{O}$ in 4% acetic acid and make to 1 litre.
(1 mL $\hat{=}$ 1 mg Cu)
- ▶ **Standard arsenic sol.** Dissolve 1.3205 g As_2O_3 in 40 mL of 10% NaOH and make up to 1 litre with 4% acetic acid.
(1 mL $\hat{=}$ 1 mg As)
- ▶ **Standard antimony sol.** Dissolve 1.1971 Sb_2O_3 in 40 mL of 10% NaOH and make up to litre with 4% acetic acid.
(1 mL $\hat{=}$ 1 mg Sb)

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Method	<p>The surfaces of the tableware, or those portions which are of interest, are extracted with 4% acetic acid at room temperature for 24 h. The area of the extracted surface should be noted. Finally, a suitable quantity of the extract, such as 25 mL, is pipetted off into a polarography vessel, deaerated with nitrogen and then polarographed under the following conditions:</p> <table style="margin-left: 40px;"> <tr><td>Method</td><td>DP</td></tr> <tr><td>Reference electrode</td><td>Ag/AgCl 4 mol/L KCl</td></tr> <tr><td>Auxiliary electrode</td><td>Pt</td></tr> <tr><td>U start</td><td>+0.1 V</td></tr> <tr><td>ΔU</td><td>- 1.5 V</td></tr> <tr><td>Amplitude</td><td>- 20 mV</td></tr> <tr><td>Damping</td><td>0</td></tr> <tr><td>Voltage: paper</td><td>0.6 s/drop / 0.5 mm:drop</td></tr> </table> <p>The half-wave potentials are at the following voltages:</p> <table style="margin-left: 40px;"> <tr><td>Cu</td><td>- 0.02 V</td></tr> <tr><td>Sb</td><td>- 0.28 V</td></tr> <tr><td>Pb</td><td>- 0.34 V</td></tr> <tr><td>Cd</td><td>- 0.55 V</td></tr> <tr><td>As</td><td>- 0.78 V and - 1.01 V</td></tr> </table>	Method	DP	Reference electrode	Ag/AgCl 4 mol/L KCl	Auxiliary electrode	Pt	U start	+0.1 V	ΔU	- 1.5 V	Amplitude	- 20 mV	Damping	0	Voltage: paper	0.6 s/drop / 0.5 mm:drop	Cu	- 0.02 V	Sb	- 0.28 V	Pb	- 0.34 V	Cd	- 0.55 V	As	- 0.78 V and - 1.01 V
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Calculation	The quantity is determined by the known addition method, the result being finally expressed as mg of the element concerned per 100 cm ² of extracted surface.																										
Remarks	<ul style="list-style-type: none"> ▶ Quite good determinations can be carried out even as low as 0.01 mg of an element/25 mL of extract. ▶ With very small quantities it may be necessary to carry out a blank control determination on the 4% acetic acid solution used. ▶ When all the metals mentioned are present, only 10 to 15 min are required for the analysis and calculation. ▶ The method was tried experimentally on a lead-crystal wineglass. The extract contained 0.162 mg Pb and 0.26 (!) mg Cd per 100 cm². 																										
Literature	<ul style="list-style-type: none"> ▶ Z. Lukaszewski und H. Szymanowska <i>Polarographische Bleibestimmung in den Extrakten von Edelkeramikfarbstoffen</i> Chem. Anal. (Warsaw) <u>17</u>, 387-392, 1972 Ref: Z.Anal.Chem. <u>265</u>, 374-375, 1973 ▶ B. Krinitz and V. Franco <i>Collaborative study of the atomic absorption method for the determination of lead and cadmium extracted from glazed ceramic surfaces</i> J. Assoc. Offic. Anal. Chemists <u>56</u>, 869-875, 1973 Ref: Z.Anal.Chem. <u>270</u>, 65, 1974 ▶ Eidgenössische Lebensmittelverordnung Art. 453 (betr. Töpfereiwaren) 																										