TRACERLAB Electro-Deposition-System

The photo shows ALPHA-ELEC-2 2 Channel system (one cell as the new, one cell as the standard version)



Standard-Systems Specifications:

- Electric Power input:
- 115 / 230 Volt, single phase, neutral and ground
- 2-Channel constant-current power-supply to connect two ea. Cells
- Upgrading of the system for the use as a 2-, 4- or 6- channel version
- Optional with housing and exhaust connection
- Set and Display of Voltage, up to 30 Volt
- Set and Display of constant-current, up to 2,0 Amp.
- Set and Display of adjust and working conditions
- Connection of one or two ea. Electrodeposition-Cells to ea. channel of the Power-Supply
- max. volume of the standard-cell: 40 ml
- max. volume of the new-designed-cell: 80 ml
- use of standard planchets 25 mm dia x 0,5 mm thickness
- standard-deposition spot 12 or 22 mm dia, others on request
- Requested ground-space: appr. 500 x 400 mm

The photo shows ALPHA-ELEC-6 3 x 2 Channel with housing





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The photo shows the Two-Channel-Power-Supply



The photo shows the available Electro-Deposition-Cells

TRACERLAB Standard Electro-Deposition cell



Details of the Standard-Electrodeposition-Cell

- Stainless-Steel-Groundplate (Cathode)
- Pt-Electrode (Anode)
- Cell body, made of Teflon
- Silicone-O-ring
- Stainless-Steel planchet, standard 25 mm x 0,5 mm

TRACERLAB new advanced Electro-Deposition cell



Details of the NEW Electrodeposition-Cell

- Stainless-Steel-Groundplate (Cathode)
- Pt-Electrode (Anode)
- Cell body, made of Plexi-Glass
- Plastic-Funnel to avoid any kind of contamination and / or memory effect
- O-ring, washer
- Stainless-Steel planchet, standard 25 mm x 0,5 mm

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Electrodeposition Procedure

The actinides are deposited electrolyticly as basic hydrated oxides on stainless steel planchets with 25 mm diameter. These are cleaned successively by means of Ethanol (96 %) and deionized water. The discs (cathode) are placed into electrolytic cells consisting of disposable plastic powder funnel, stainless steel bottom with a 25 mm diameter milling out and a power connection, perspex[™] part supporting the plastic funnel, and perspex[™] screw lid with holder for the anode. A platinium-gauze is used as the anode, it is placed at a distance of 2 mm ... 3 mm above the stainless steel disc. The DC-power supply should be designed for constant current with 2 A and 25 V.

Procedure

- 1. The beaker or evaporation dish with the residue of the radiochemical separation is fumed with 500 L each of HNO₃ (65 %) and H₂O₂ (30 %) in order to remove organic traces. The result should be a colourless residue. Alternatively, fuming with H₂SO₄ (98 %) and calcination with NaHSO₄ at 520 °C for 15 minutes can be used (1). The residue is dissolved in 4 mL NaHSO₄- solution (0,25 M) at elevated temperature for about 5 minutes. The dissolution in bisulfate has to be thoroughly done, otherwise losses of matter up to 20 % will result.
- The sample is transferred into the electrolytic cell and beaker is rinsed successively with 1 mL 1,5 M-(NH₄)₂SO₄-solution three times and with 1 mL deionized water twice.
- The electrolytic conditions are: I = 1,6 A, U: 5 V ... 10 V, t = 45 min. Voltage is decreasing during the electrodeposition.

4. After 44 minutes 2 mL NH₄OH (25 %) are added by pipetting it through the drill-hole in the lid. The electrolyte is discarded, the planchet is washed twice with 2 mL 0.1 M-NH₄OH, and after rinsing with ethanol (96 %) the planchet is heated on a hot plate at about 350 °C for 5 minutes.

Remarks

- 1. 2 mL NH₄OH (25 %) are added 1 min before the disconnection of the voltage in order to prevent the partial dissolution of the actinide layer until the electrolyte (pH \approx 2) has been poured out. Without that quenching the loss amounts to 5 % ... 30 %.
- 2. Residues of the electrolyte are removed with diluted NH₄OH and with ethanol from the stainless steel planchet. Since sublimating ammonium salts could drag along the actinides during the following heating to 350 °C. The heating serves for the sublimation of Po-isotopes and for the formation of an oxide layer, respectively, whereby the risk of contaminating the detector's surface due to recoil effects is reduced.
- 3. Complexing anions like SO_4^{2-} , PO_4^{3-} , and carboxylic-acid anions extremely trouble to some extent. For example, about 10 µg citrate suffice in order to reduce the degree of deposition to 5 % at most; oxalate has a disturbing effect from 50 µg, nitrate from 50 mg. Above 0.6 mol·L⁻¹ the degree of deposition is decreased to 60 % ... 70 % by sulfate. Bisulfate above 0.3 mol·L⁻¹ causes a similar effect, it reduces the degree of deposition up to 50 %.

- 4. As long as the current density does not exceed 0.8 A·cm⁻², the electrode spacing in the range of 1 mm ... 5 mm does not influence perceptibly the degree of deposition. A greater spacing and so voltages above 10 V effects a stronger heating of the electrolyte so that boiling may occur. The emerging gas bubbles markedly reduce the degree of deposition. Even boiling for five minutes results in an average deposition of only 40 % ... 50 %.
- 5. The cathode surface should be as fat free and polished as possible in order to guarantee a unique and firmly adhering basic actinide layer. Beyond that, the pH of the electrolyte and so the degree of deposition can be controlled by the form and surface of the anode, for its concentration polarisation is increased by a smoother surface so that the anodic oxidation of special electrolyte anions $(SO_4^{2-}, C_2O_4^{2-}, NH_4^+)$ is reduced.

The degree of deposition amounts to (90 ± 10) % (n = 10; p = 95 %).

The necessary times for the quantitative deposition fluctuate according to the isotope up to 10 minutes. This can be explained by differences in:

- charge density of the respective ion, which influences its mobility in the electric field,
- specific activity and resulting molar concentration (achieving the solubility product of the actinide hydroxid).

References:

S. Bajo, J. Eikenberg, *Electrodeposition of actinides for alpha-spectrometry*, J.
Radioanal. Nucl. Chem., **242**/ 3 (1999) 745 - 751.