Presentation, handling and operation instructions TRACERLAB Electro-Deposition Systems "S" / "S-L" Sample preparation for the Alpha-Spectroscopy

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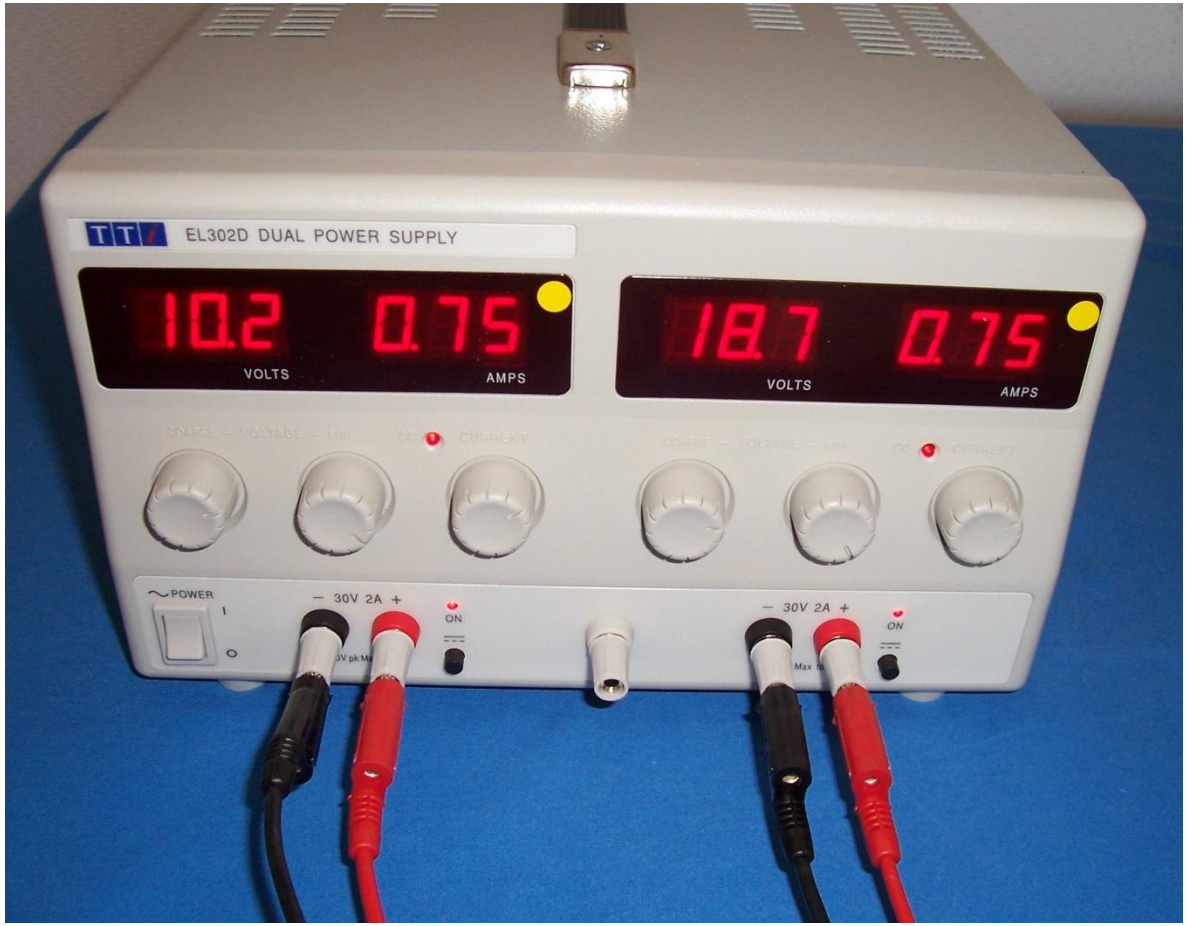
Alpha-Electro-Deposition-System

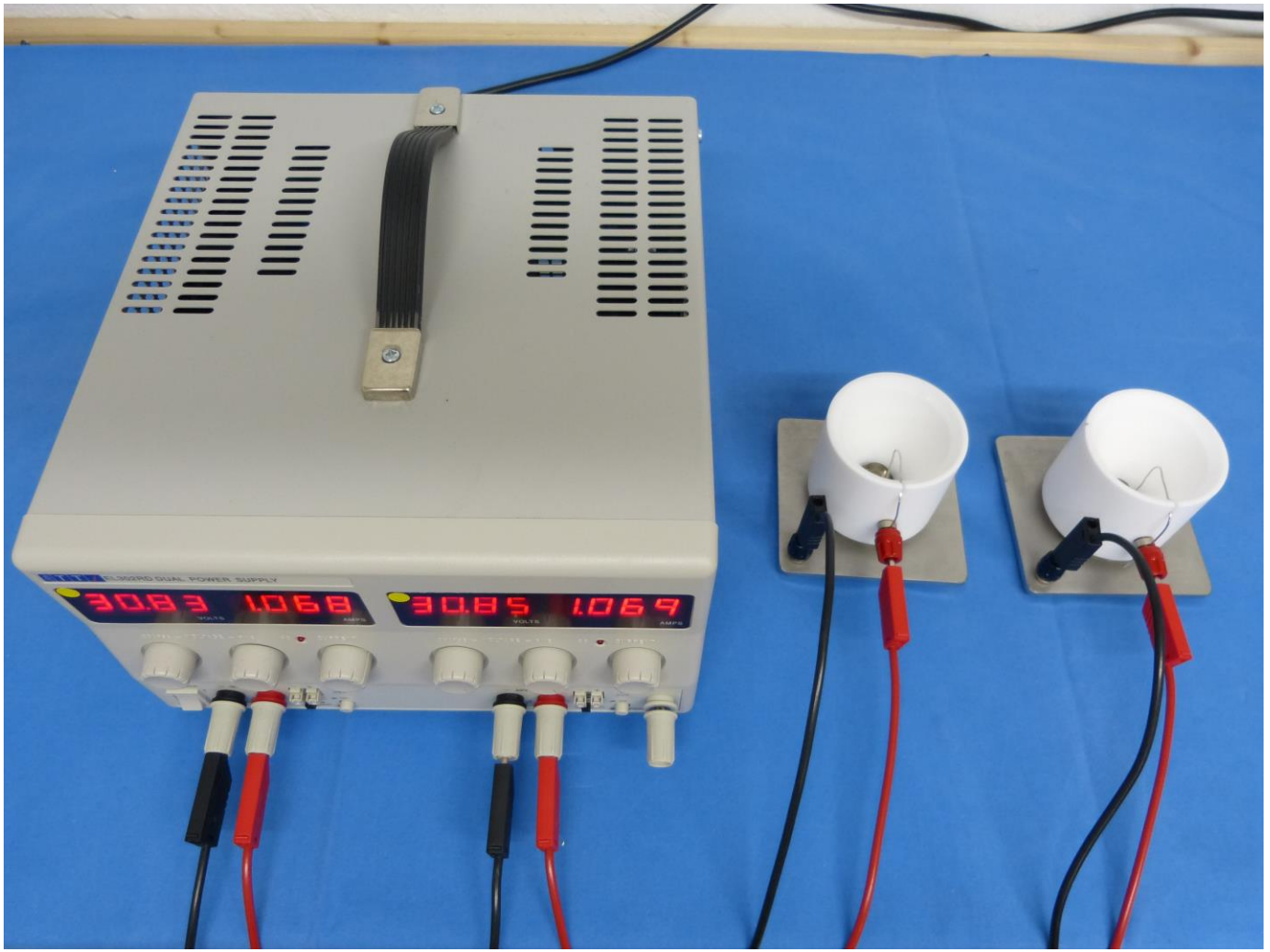
Standard-Systems Specifications:

- Electric Power input:
  230 Volt AC, 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 frame and housing for each 4, or 6 channels
- 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(S) or 22(L) mm dia, others on request

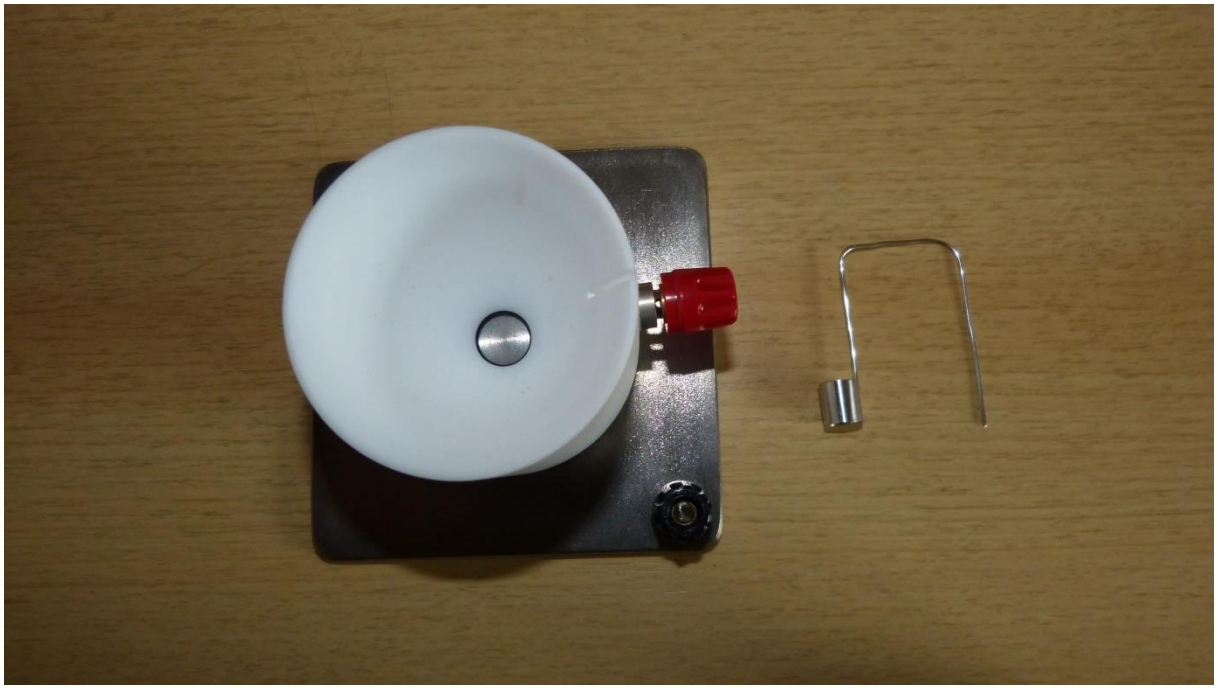
Electrodeposition System
Dual-Channel constant current Power-Supply



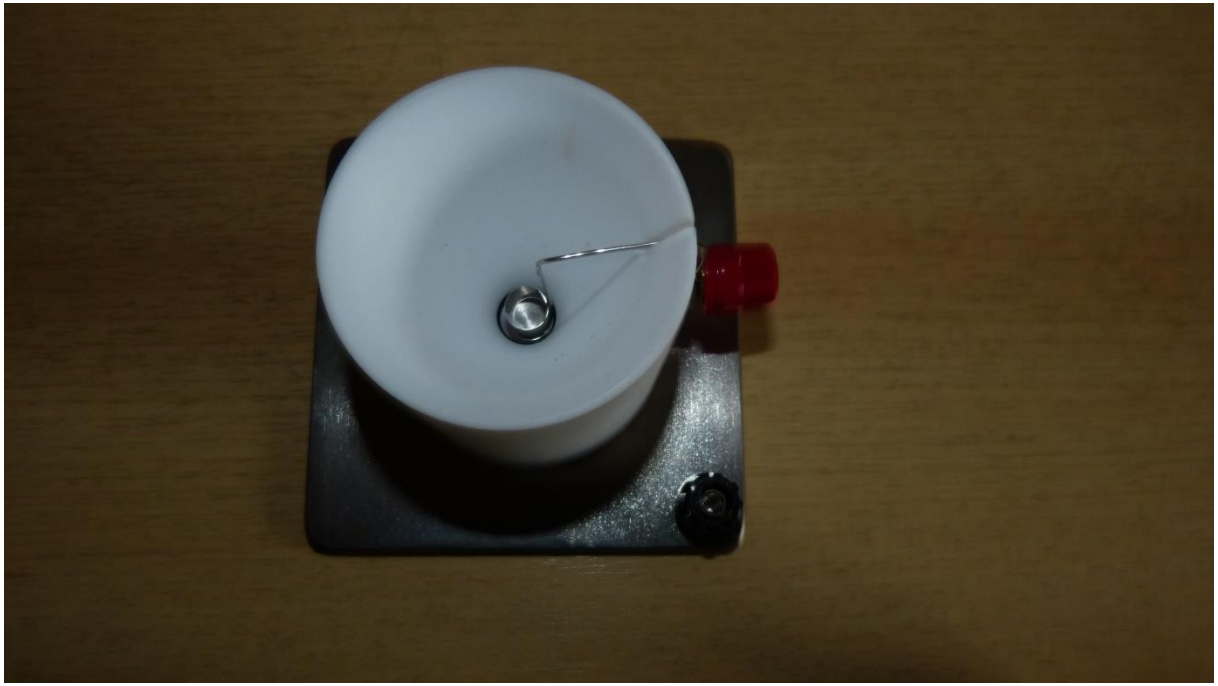
ALPHA-ELEC-2-S / 2-S (L)
2-Channel Electrodeposition System



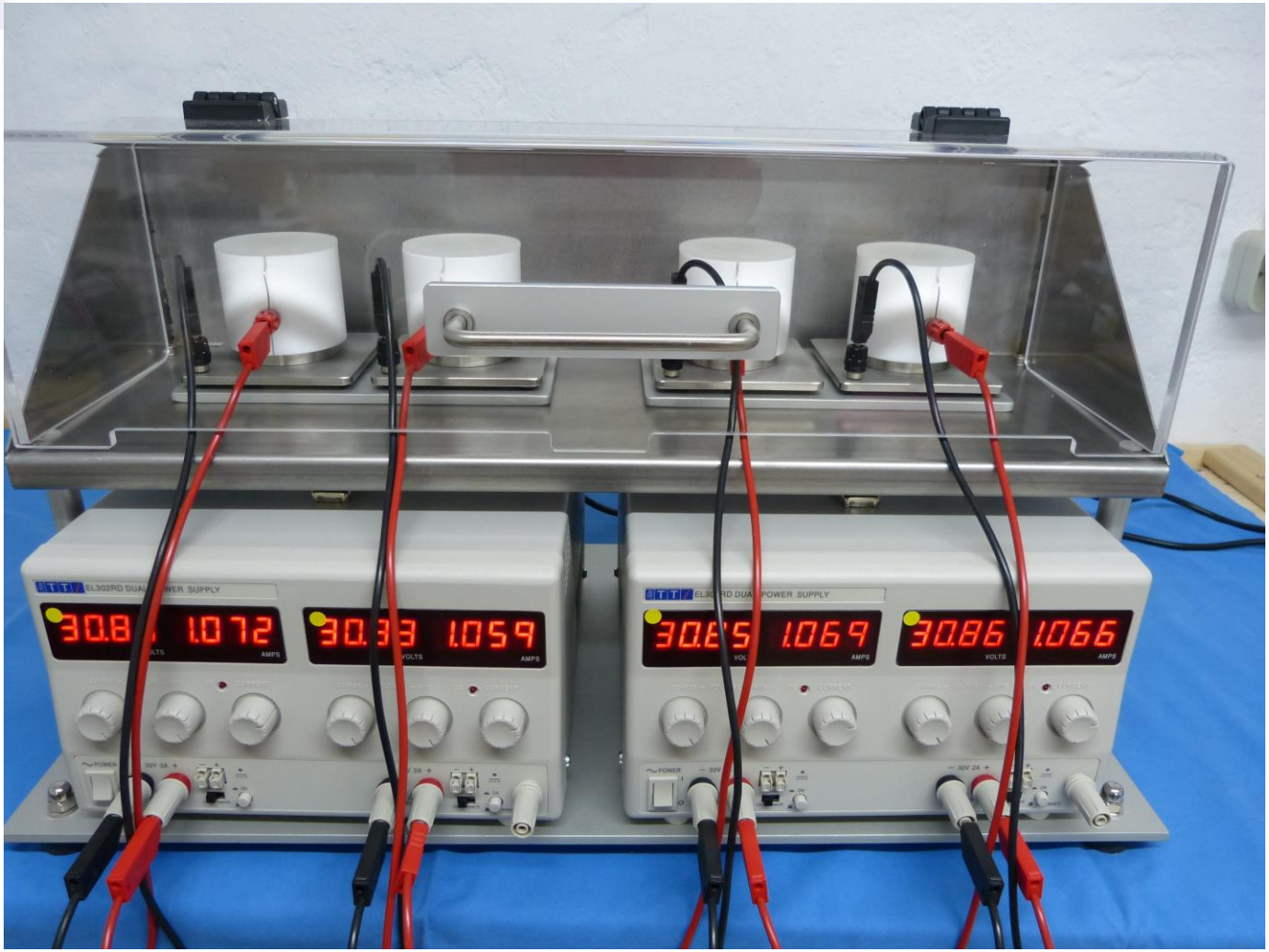
Electrodeposition-Cells Type S with the prepared standard Pt-Electrode



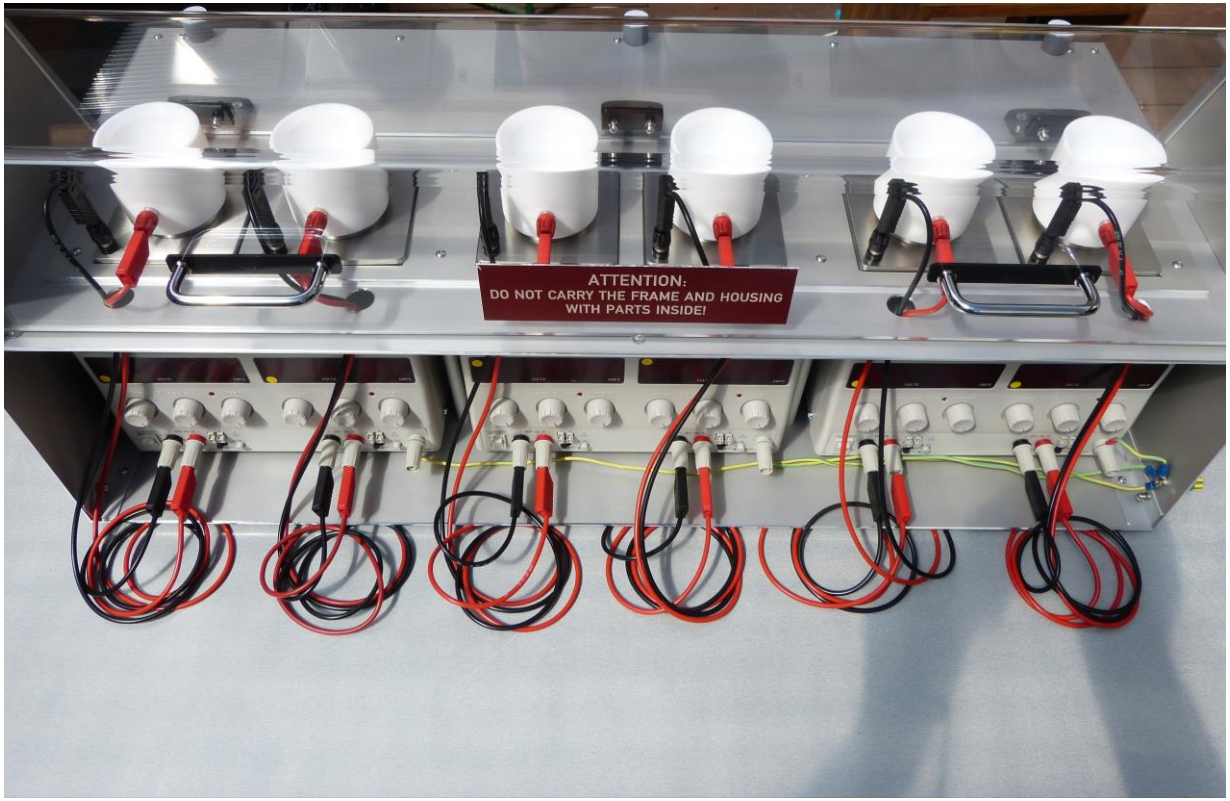
Electrodeposition-Cells Type S with the inserted Pt-Electrode



ALPHA-ELEC-4-S
2 x 2 Channel in a frame and housing

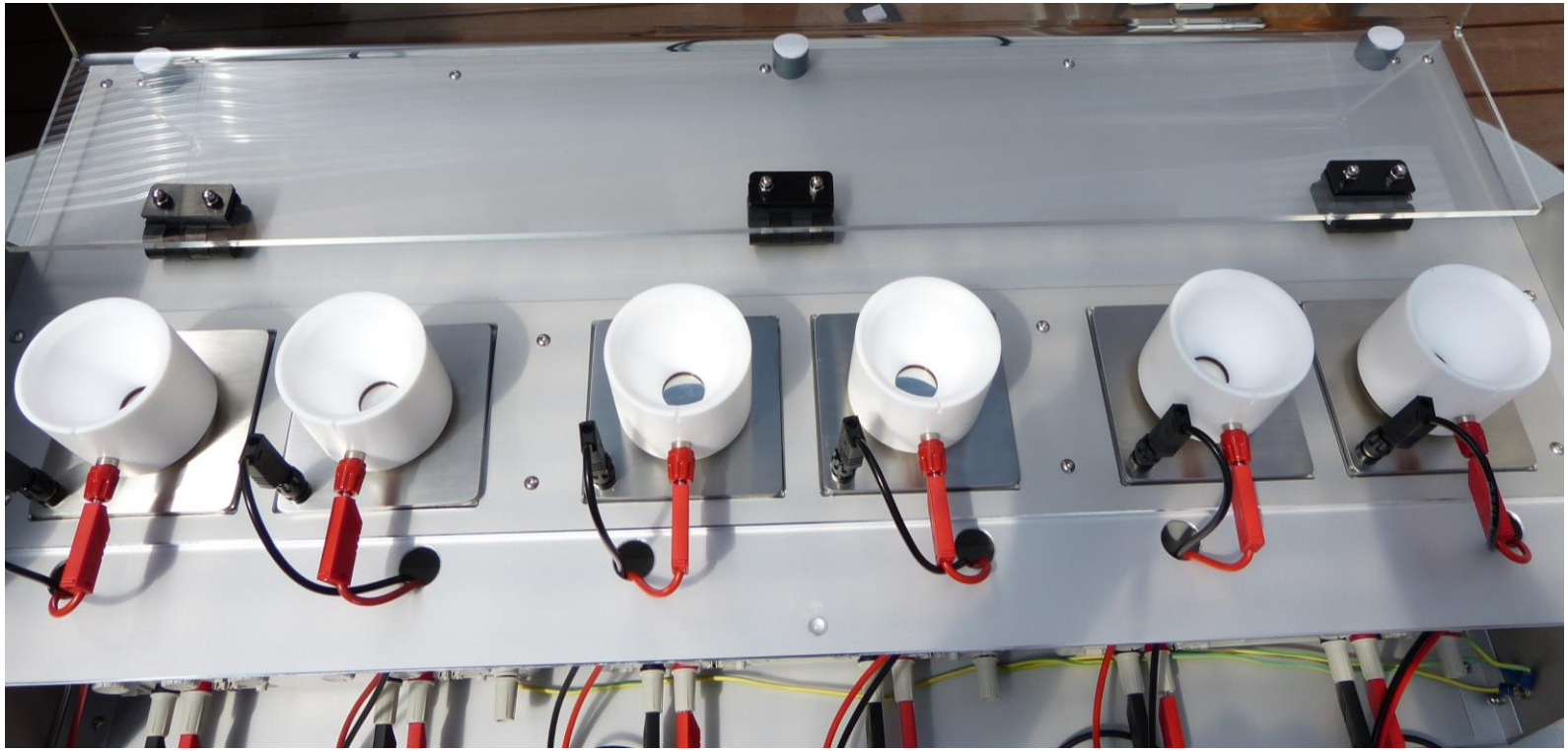


ALPHA-ELEC-6-S-L
3 x 2 Channel in a frame and housing

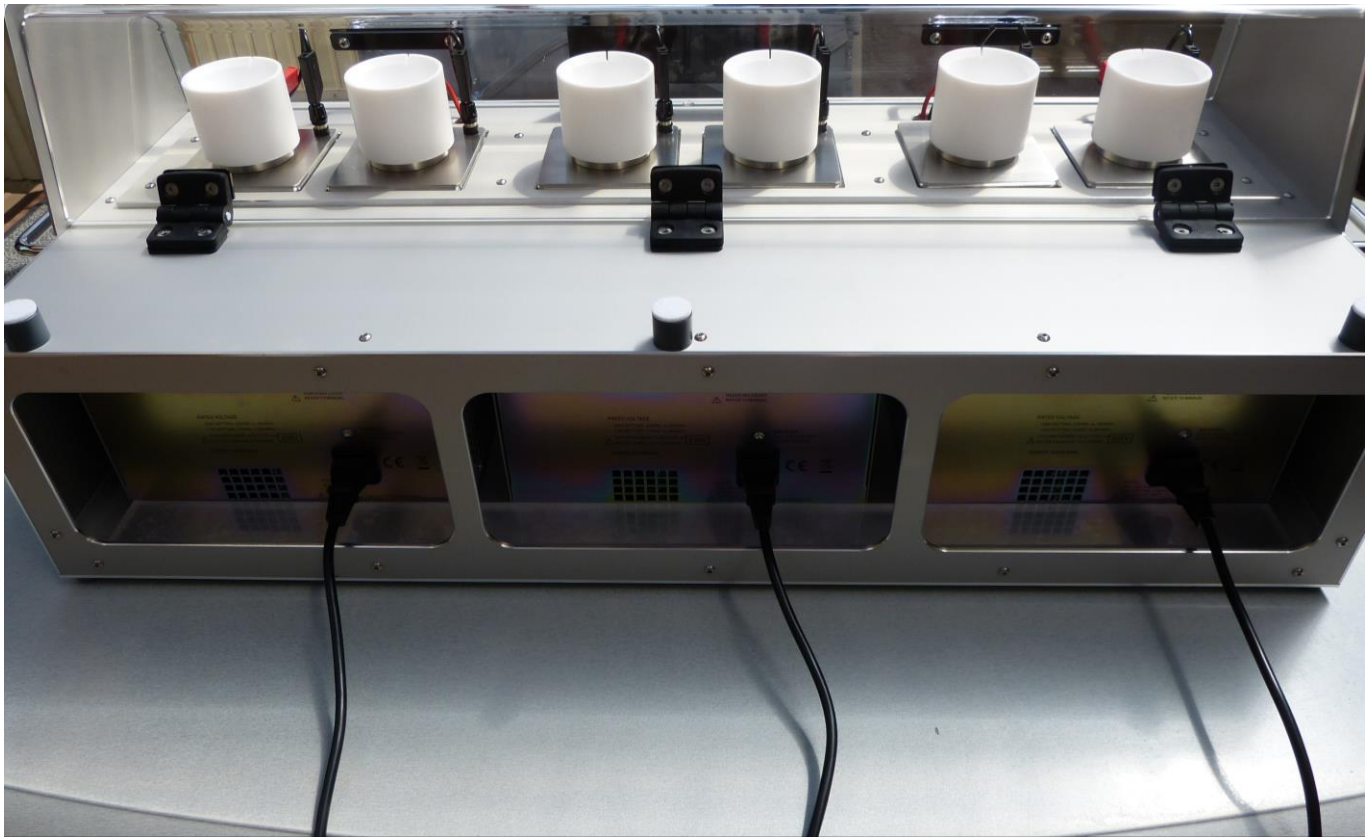


Tracerlab Electrodeposition-System
correct ground-connections in between each power-supply
and Frame / Housing, to arrange a correct potential-exchange

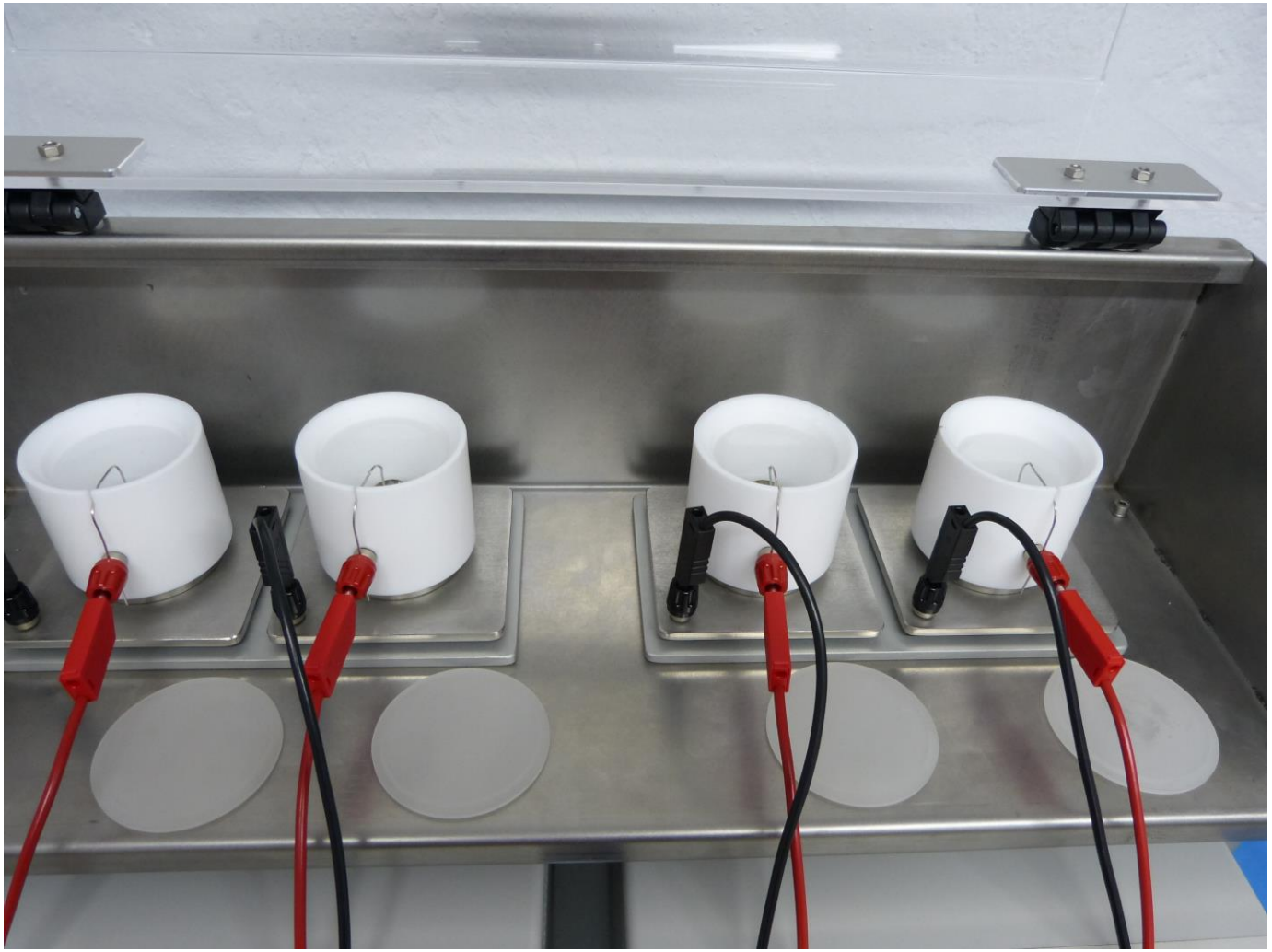
Top-View of ALPHA-ELEC-6-S-L
3 x 2 Channel in a frame and housing



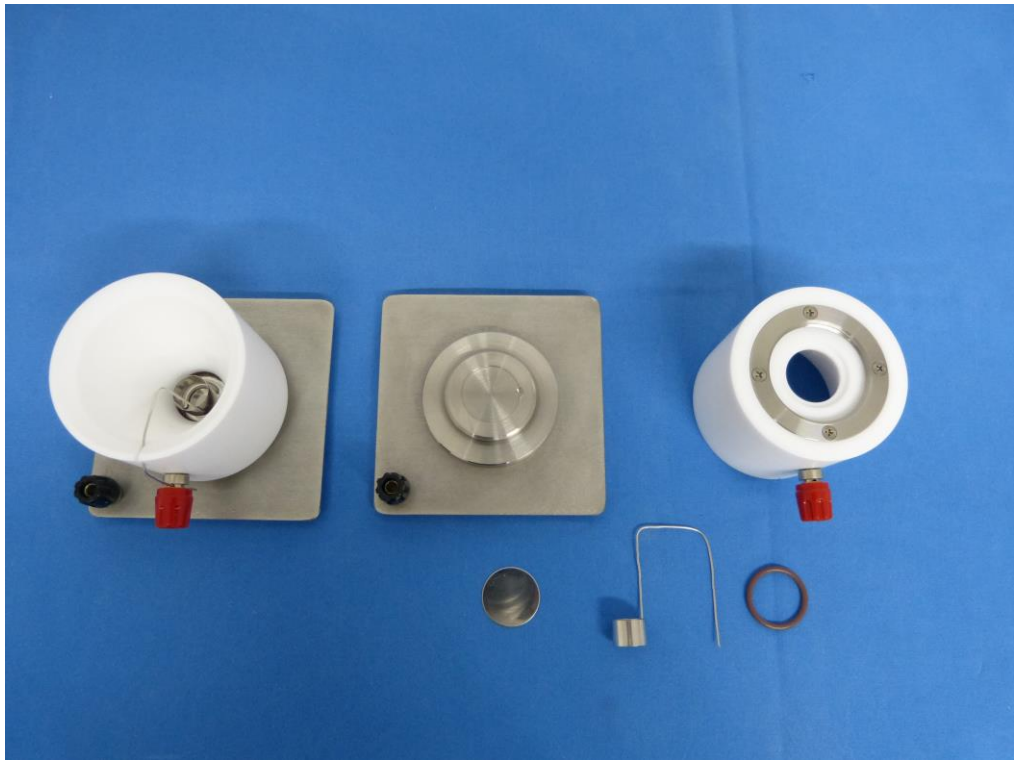
Rear View of ALPHA-ELEC-6-S-L AC-Connection of power-supplies



Electrodeposition-Cells with optional plexi-glass-covers

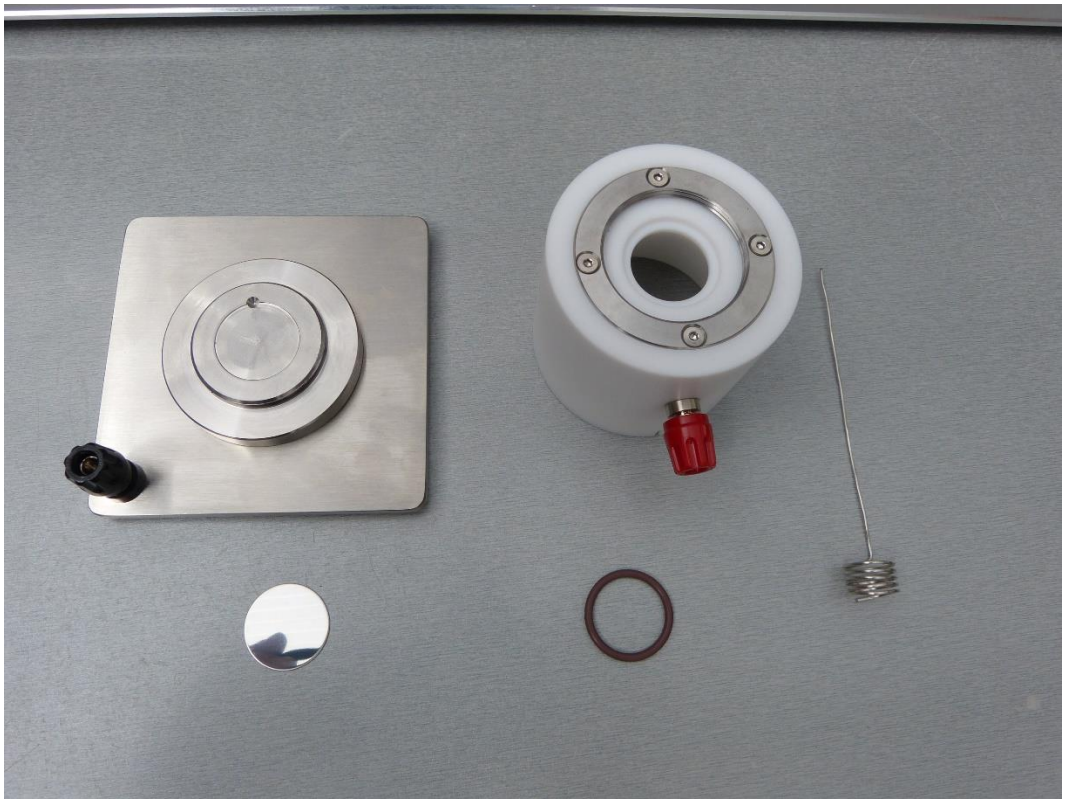


Individual parts of PTFE Electrodeposition-Cells Type-S (L)



Tracerlab Electrodeposition-Cell-S (L)
stainless-steel groundplate to insert the stainless-steel-planchet, Viton-O-Ring, PTFE-Cell-Body and standard cylindrical formed Pt-Electrode

Individual parts of PTFE Electrodeposition-Cells Type S-L-CSC

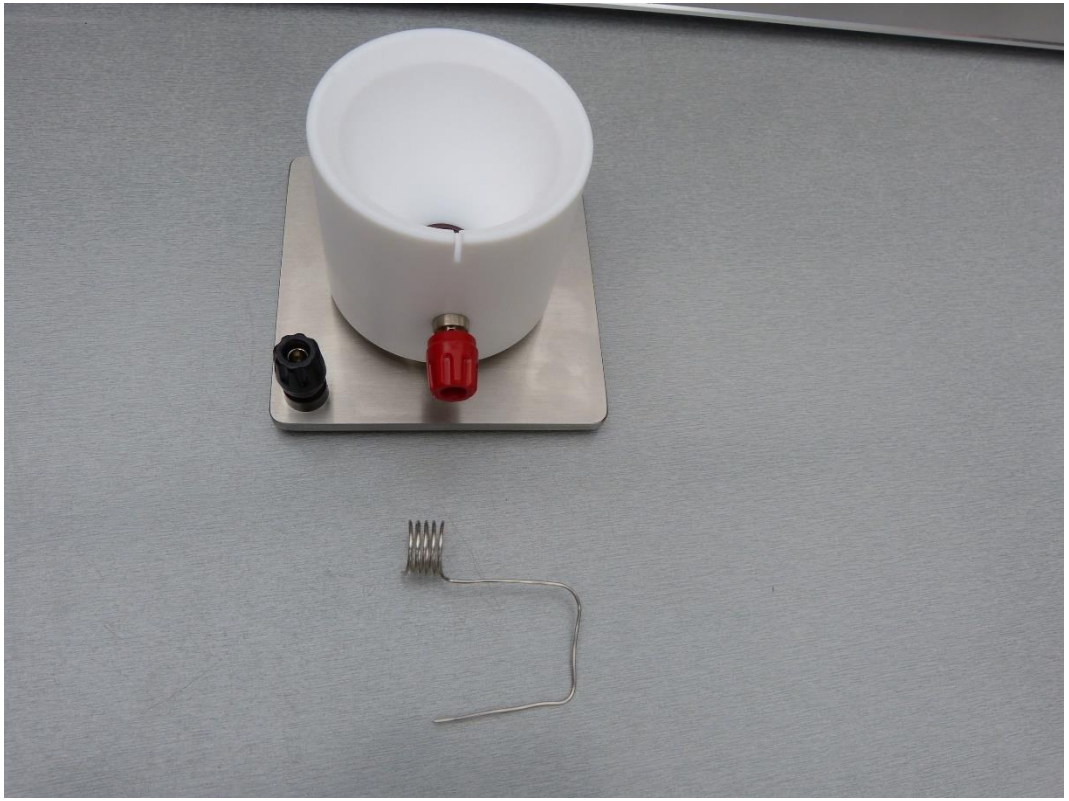


Tracerlab Electrodeposition-Cell „S-L“ - version CSC
stainless-steel groundplate to insert the stainless-steel-planchet,
Viton-O-Ring, PTFE-Cell-Body and modified Pt-Electrode

Individual parts of PTFE Electrodeposition-Cells Type S-L-CSC

Tracerlab Electrodeposition-Cell „S-L“ - version CSC
with the modified spiral-wire-formed-Pt-Electrode,

Individual parts of PTFE Electrodeposition-Cells Type S-L-CSC



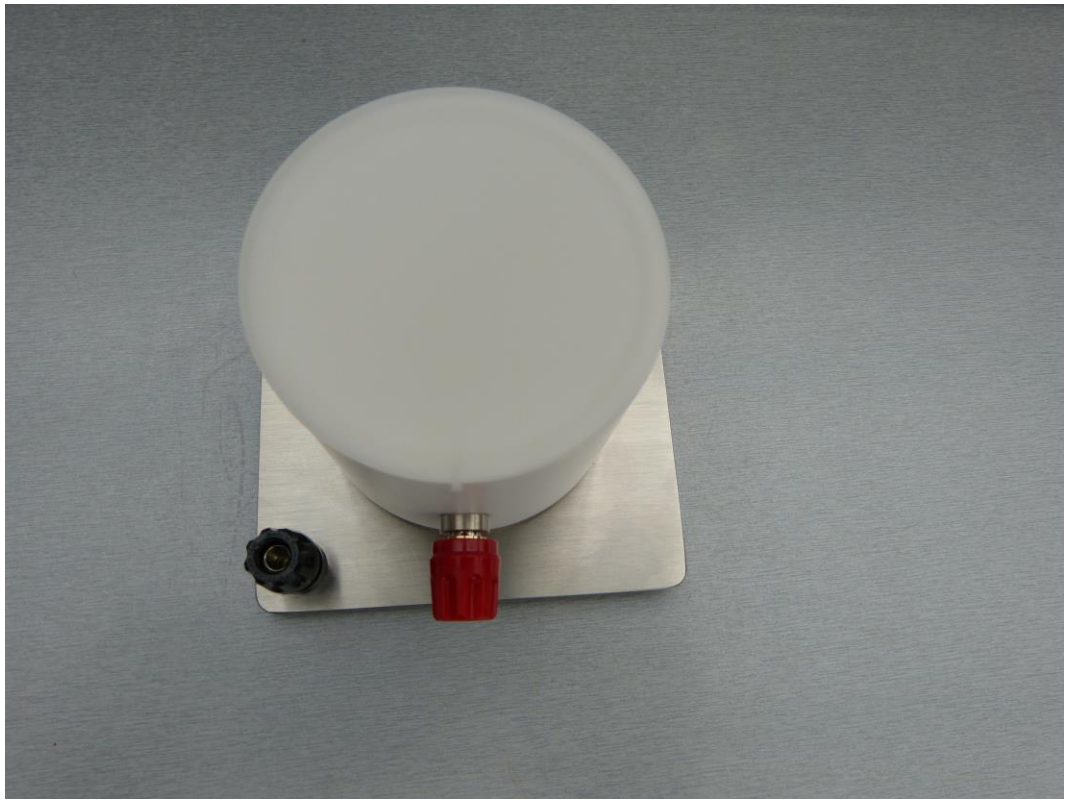
Tracerlab Electrodeposition-Cell „S-L“ - version CSC
with the modified spiral-wire-formed-Pt-Electrode,
ready to insert into the cell-body

Individual parts of PTFE Electrodeposition-Cells Type S-L-CSC

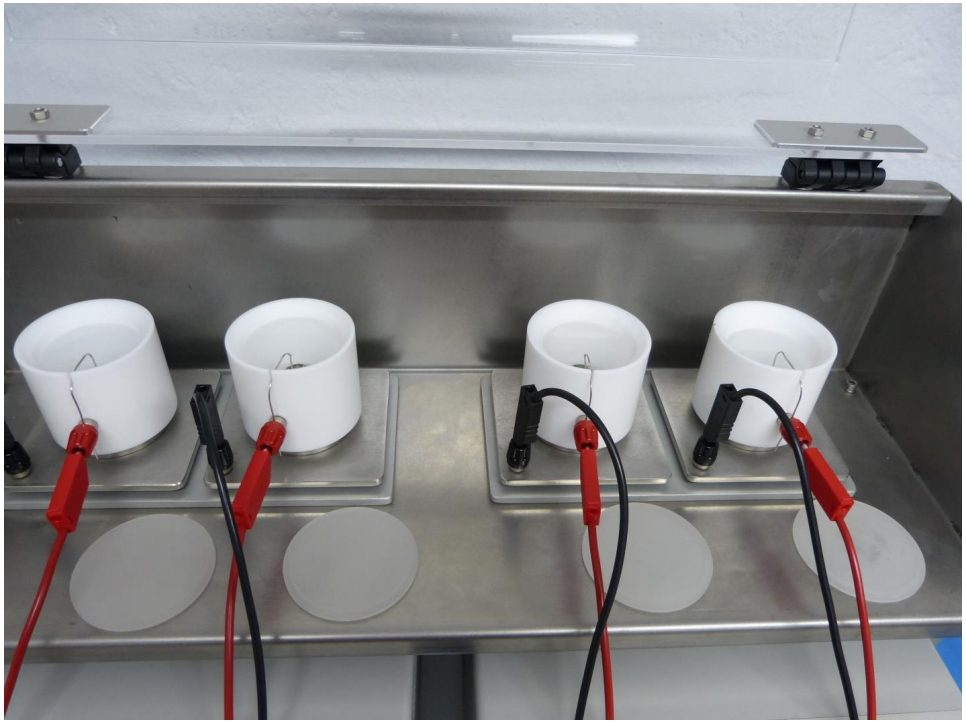
Tracerlab Electrodeposition-Cell „S-L“ - version CSC
with the modified spiral-wire-formed-Pt-Electrode,
ready to use with the inserted Pt-Electrode in the cell-body

Electrodeposition-Cell Type S-L with optional plexi-cell-cover

Electrodeposition-Cell Type S-L with optional plexi-cell-cover



Electrodeposition-Cell Type S-L with optional plexi-cell-cover in an optional Frame- and Housing



Electro-Deposition methods overview

Parameter	Oxalate/HCl	$(NH_4)_2SO_4/H_2SO_4$	$DMSO/HNO_3$
Evaporation	1 ml HCl, 10 min	HNO_3/H_2SO_4, 0,5-5 h	1 ml HCl, 10 min
Deposition duration	120 min	90 min	10 min
Chemical efficiency	100 %	90 - 100 %	90 - 95 %
Standard deviation	± 1 %	± 10 %	± 5 %
Electrolyt sensitivity	Low	High	High
Equipment	Cl-outlet	Table	DMSO-Outlet
Voltage	15 - 20 Volts	15 - 20 Volts	300 Volts
FWHM	58 ± 3 keV	46 ± 5 keV	51 ± 9 keV

Electro-Deposition methods overview

Method overview:

Published methods for electrodeposition of actinides use different parameters p.e. chemical constitution of electrolyte, current intensity, deposition duration and electrolyte volume.
The table in previous page shows commonly used deposition methods.

Electro-Deposition methods overview

Cleaning methods:

Deposition cells as shipped from factory should undergo several electrolysis passes with pure chemicals called blind analysis.
The surface of the polished stainless steel may turn black the first times. After four or five passes this effect will disappear.
To remove all impurities from new stainless steel planchets they are cleaned with chromium sulfuric acid once. Between analysis the planchets are recommended to be stored under pure alcohol.
Before use they are cleaned with a fluffless cloth and acetone.

Electro-Deposition 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.

Electro-Deposition Procedure

1. The beaker or evaporation dish with the residue of the radiochemical separation is fumed with 500 mL each of HNO_3 (65 %) and H_2O_2 (30 %) in order to remove organic traces. The result should be a colourless residue. Alternatively, fuming with H_2SO_4 (98 %) and calcination with $NaHSO_4$ at 520 °C for 15 minutes can be used (1). The residue is dissolved in 4 mL $NaHSO_4$-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.

Electro-Deposition Procedure

2. The sample is transferred into the electrolytic cell and beaker is rinsed successively with 1 mL 1,5 M-$(NH_4)_2SO_4$-solution three times and with 1 mL deionized water twice.

3. 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 NH_4OH (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-NH_4OH, 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 NH_4OH (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 ≈ 2) has been poured out. Without that quenching the loss amounts to 5 % ... 30 %.

2. Residues of the electrolyte are removed with diluted NH_4OH 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.

Remarks:

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 %.

Remarks:

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:

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

Electrodeposition-System S (S-L CSC)
2 up to 16-channel version,
made of 1 up to 8 each dual channel power-supply

1. Step, setup the 230 Volt AC-Power-Connections for each dual-channel power-supply
2. Step, setup and arrange the ground-connections for each power-supply to the frame and housing

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Short operating instructions how to use TRACERLAB Alpha-Electrodeposition-Systems, standard with PTFE-Cell-Body,
or advanced Deposition-Cells with Plexi-Cell-Body.
The Electrodepositon-cells are available as type S or N with an active deposition-spot diameter of 12 mm,
or as Type S-L or N-L with an active deposition-spot diameter of 22 mm

1) Preparation of the deposition-cell:

- for cell S (S-L) install the Pt-electrode into the PTFE-Cell-Body (Standard-Cell) or for cell N (N-L) in the top-plate of plexi-cell-body (New Plexi-Cell-Body) and connect to the red anode-connector
- insert the well prepared and clean planchet in the middle of the stainless-steel-ground-plate
- prepare the clean PTFE-Cell-Body (Standard-Cell) by inserting the silicone-O-ring and srew the cell-body on the ground-plate.

For cell N (N-L) prepare the plexi-cell-body by inserting the O-ring and flat-disk and screw it on the stainless-steel-ground-plate, hand-tight,
than insert the plastic-funnel, push it down and screw the top of the plexi-cell to the plexi-cell-body and screw it tight together

- the Pt-electrode is to be placed in the middle of the Cell-Body and should have a distance to the planchet of appr. 1 – 2 mm.

2) fill into the cell the prepared liquid for deposition
3) prepare the 2-channel power supply
   - connect AC-line, 220 - 240 Volt, 50 cycles and switch ON power
   - follow the instruction of the manual, to operate the power-supply
4) connect the two cables for each cell
   black = cathode to the stainless-steel-ground-plate
   red = anode to the Pt-Electrode
5) switch on the power-output to start the electrodeposition process
   control the current and voltage at the digital-meters
6) end of electrodeposition, turn OFF power supply
   - Cell S (S-L) empty the PTFE cell-body
   - Cell N (N-L) screw off the top-plate of the plexi-cell-body
   - empty the plexi-cell-body and screw off the plexi-cell-body
   - remove the planchet from the stainless-steel-ground-plate
   - take the planchet for the measurement process at the alpha-spectrometer
   - for cell N (N-L), remove the plastic-funnel, O-ring and flat-disk
   - clean up the cell and electrode for next electrodeposition process