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

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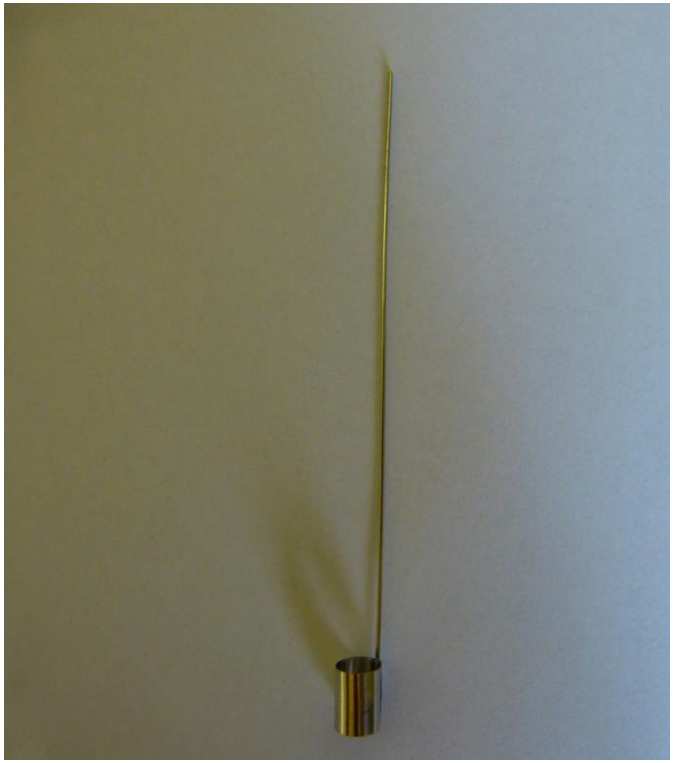
Alpha-Electro-Deposition-System

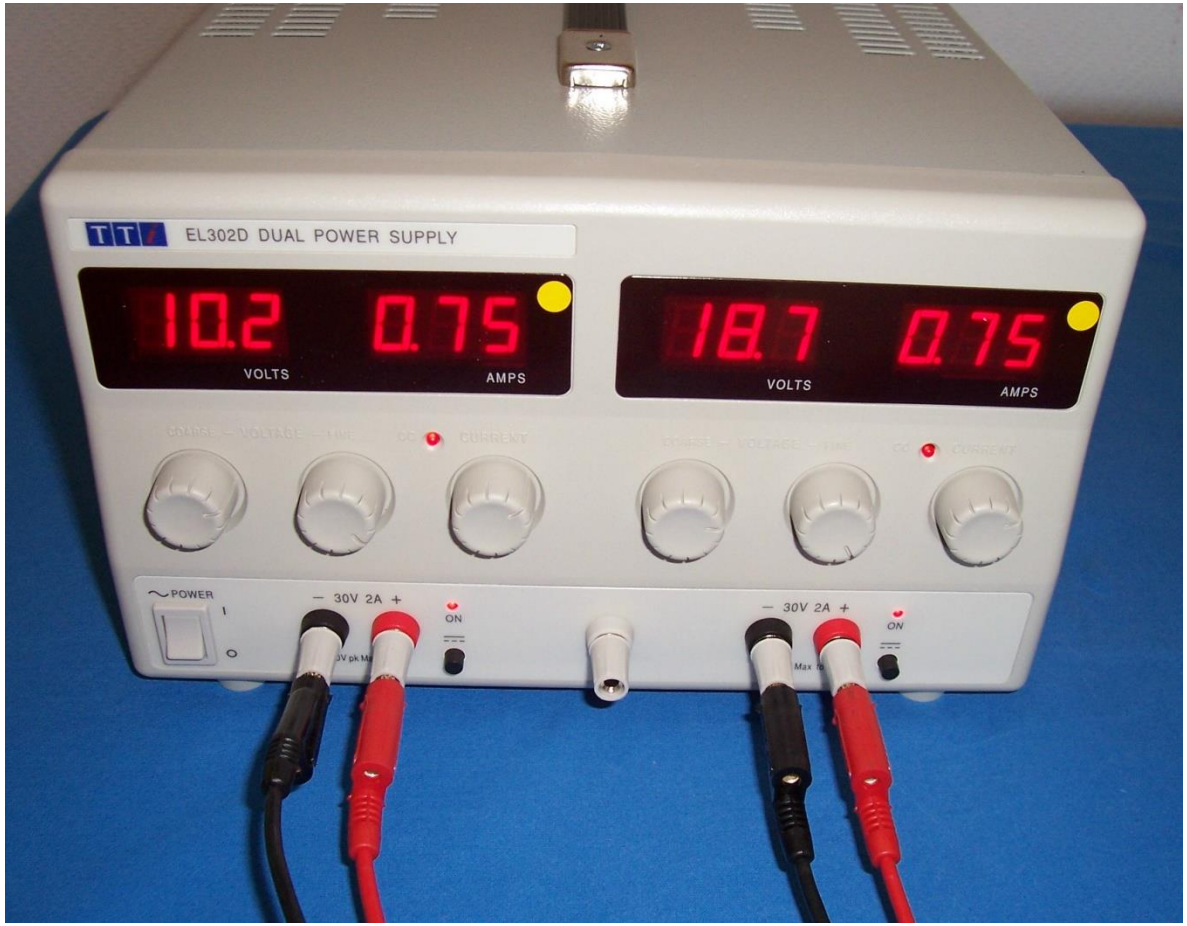
Standard-Systems Specifications:

- 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 housing and exhaust connection
- Set and Display of Voltage, up to 30 Volt
- Set and Display of constant-current, up to 2,0 Amp. (optional 5 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
- Requested ground-space: appr. 500 x 400 mm

Electrodeposition System N (L)
Standard cylinder-form Pt-Electrode
Modified spiral-coil-form Pt-Electrode



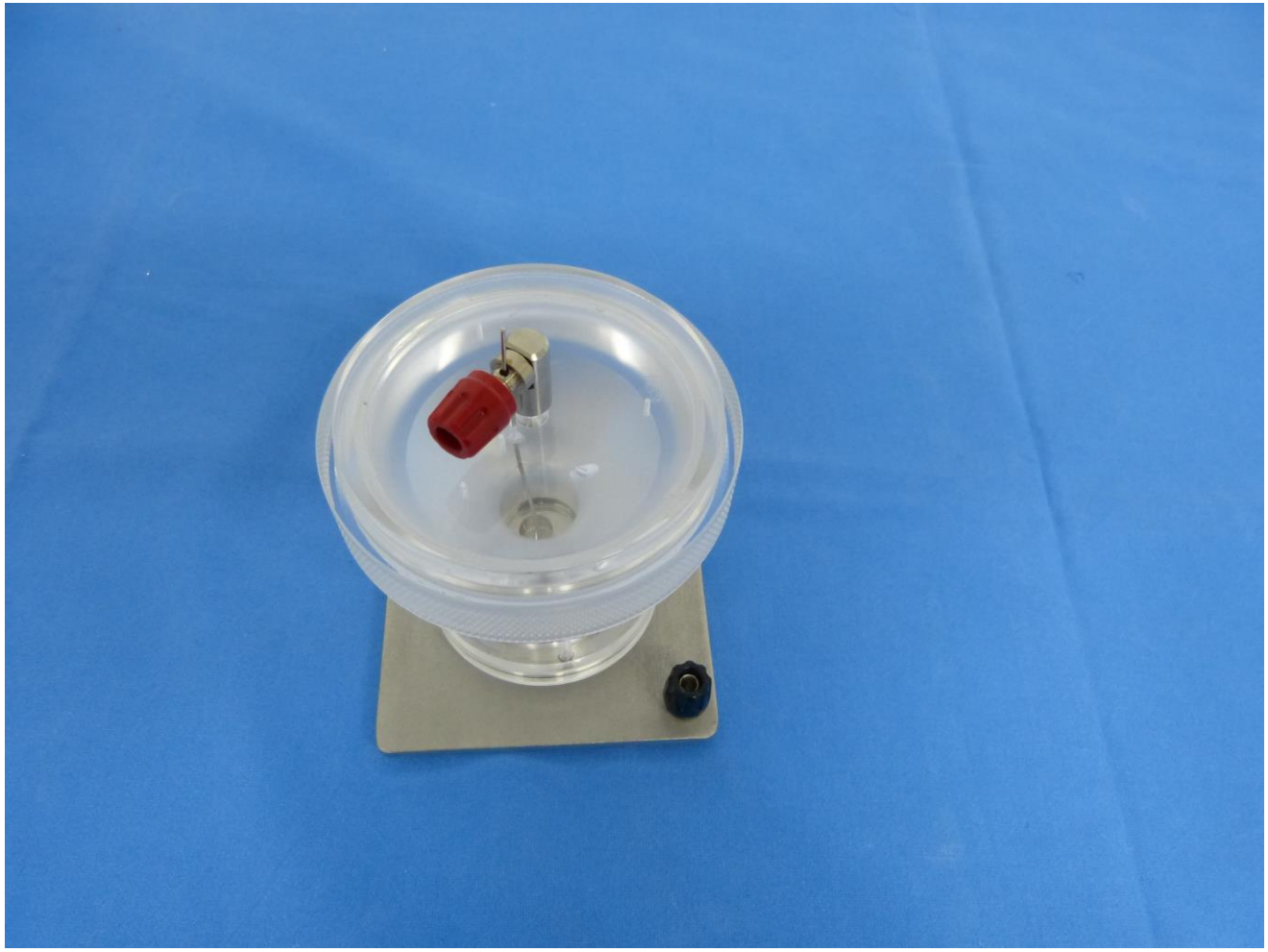
Electrodeposition System
Dual-Channel constant current Power-Supply



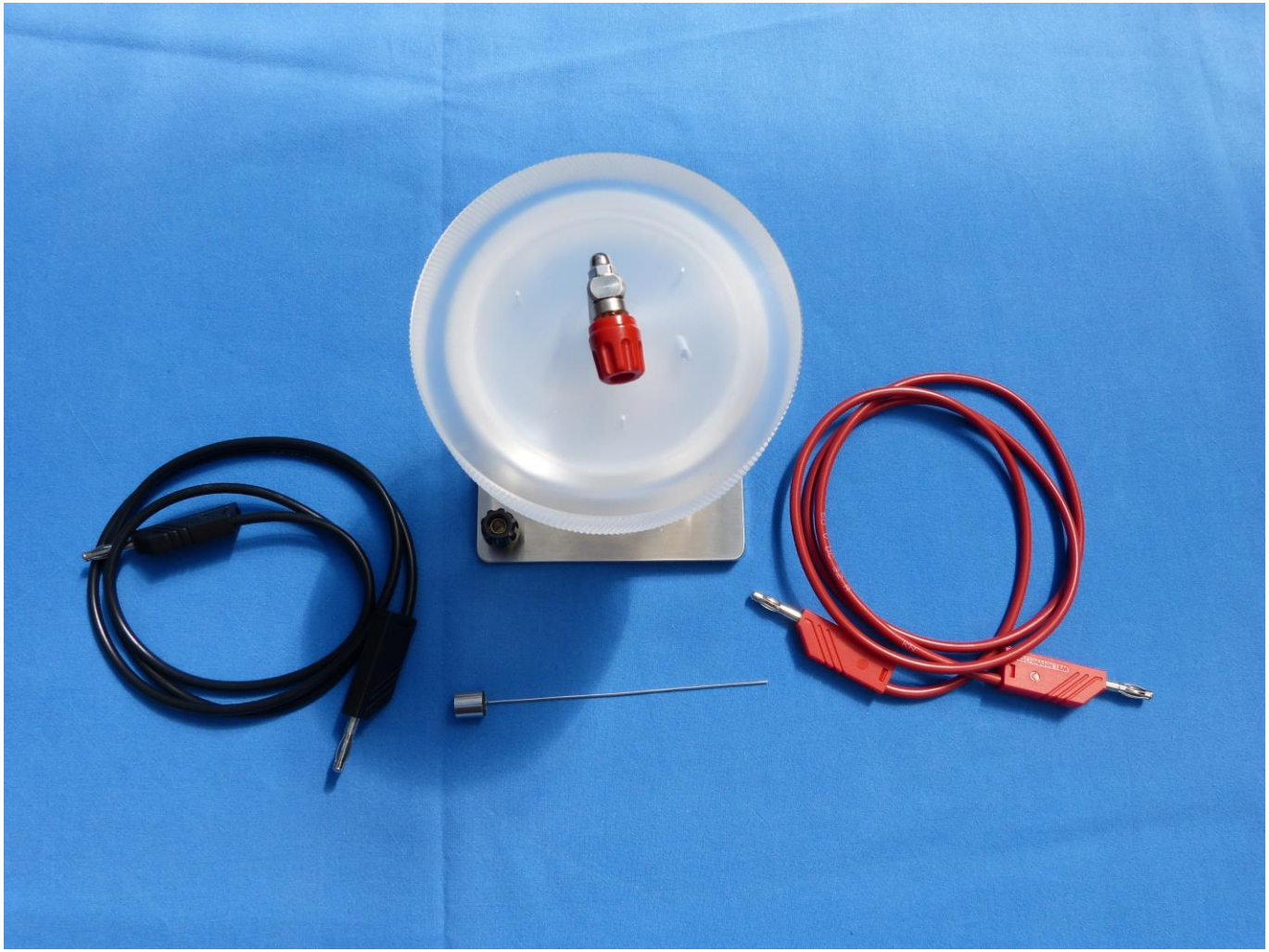
Electrodeposition-Cell Type N

Eledctrodeposition-Plexi-Glass Cells Type “N” with a removable one-way plastic-funnel to avoid any kind of memory-effect at the cell-body.
The electro-deposition-cells are made for the use of stainless-steel-planchets, standard diameter 25 mm, x 0,5 mm or others on request.
The deposition-spot is as standard with 12 mm diameter,
Or for type “N-L” with 22 mm diameter
Standard dual- channel constant current power supply, output up to 2000 mA in ea. channel

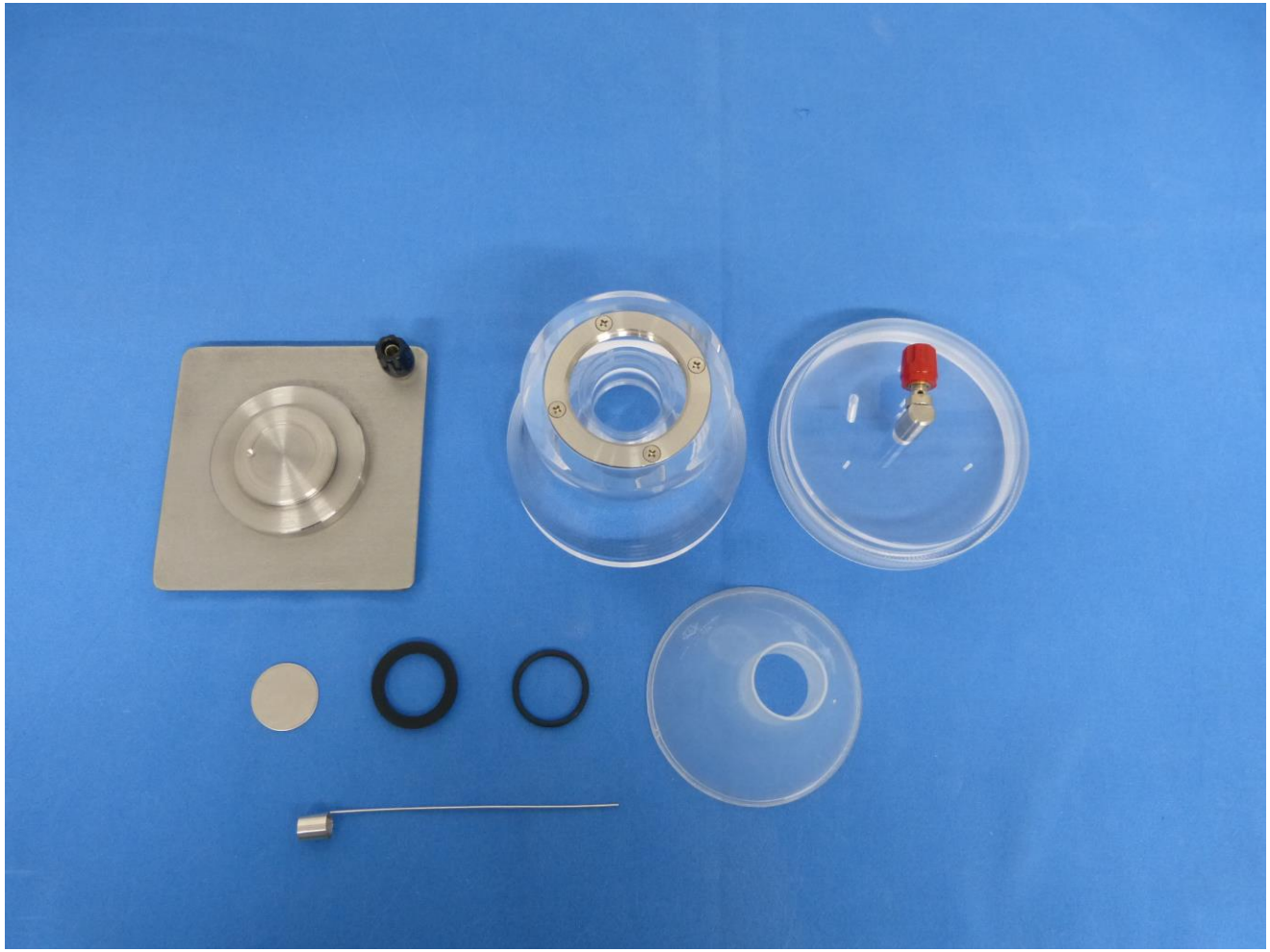
Electrodeposition-Cell Type N-L Full Plexi-Glass – Version



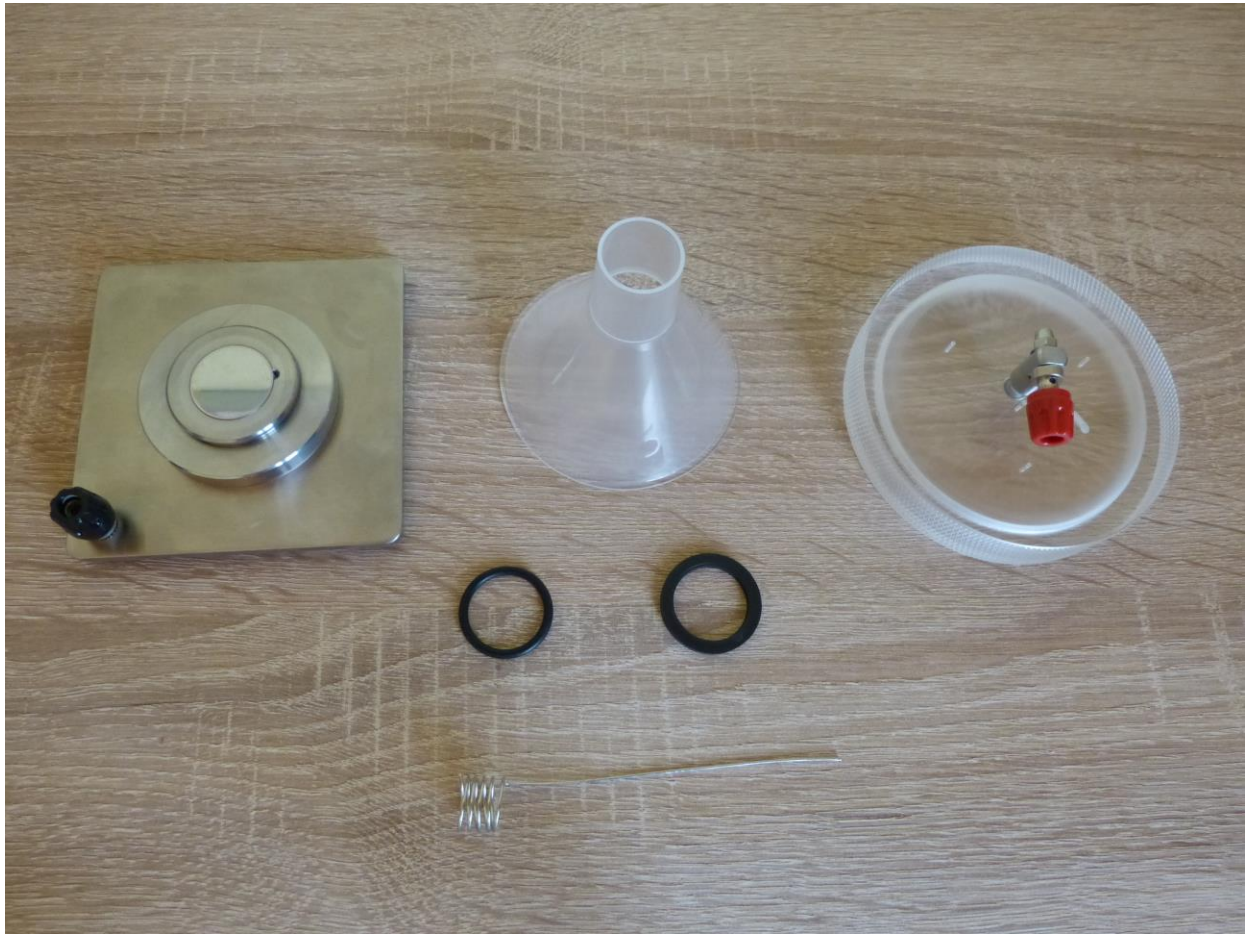
Electrodeposition Cell Type N (L)
Full Plexi-Glass – Version



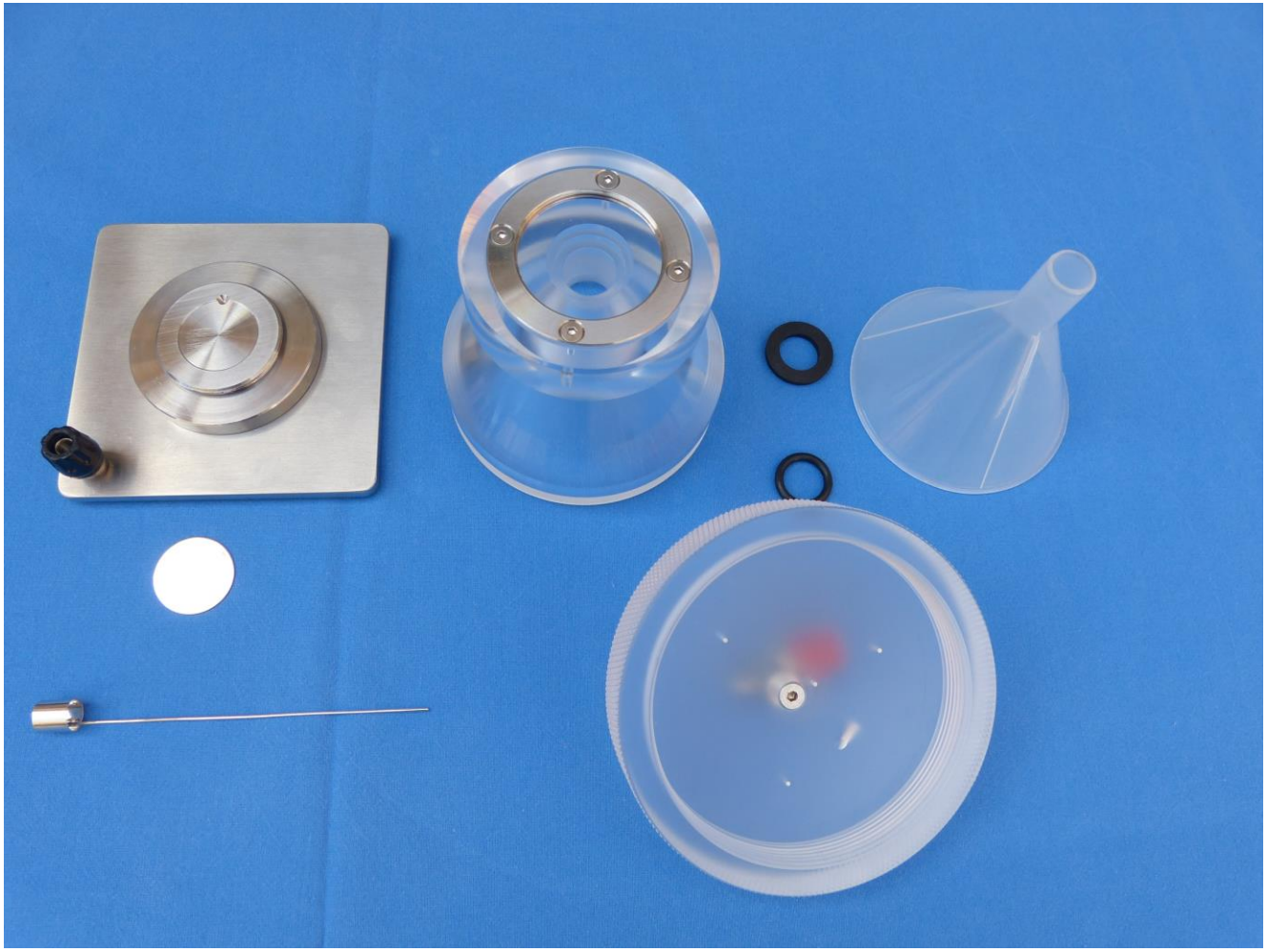
Electrodeposition Cell Type N-L
Individual parts of the cell N-L version with cylindrical-formed-Pt-Electrode



Electrodeposition Cell Type N-L
Individual parts of the cell N-L version with spiral-wire-formed-Pt-Electrode



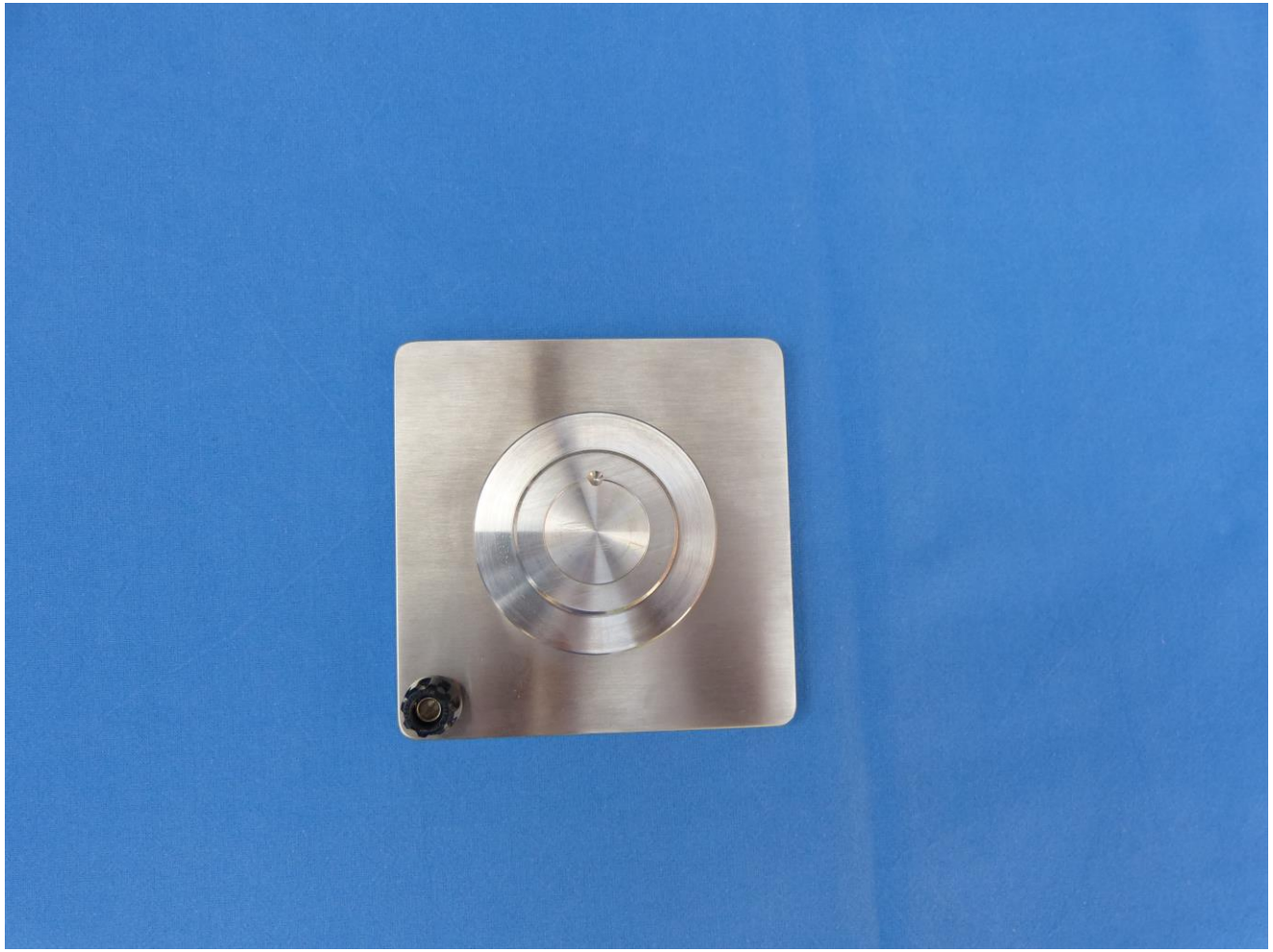
Electrodeposition Cell Type N (L)
Individual parts of the cell N version



Electrodeposition Cell Type N (L)

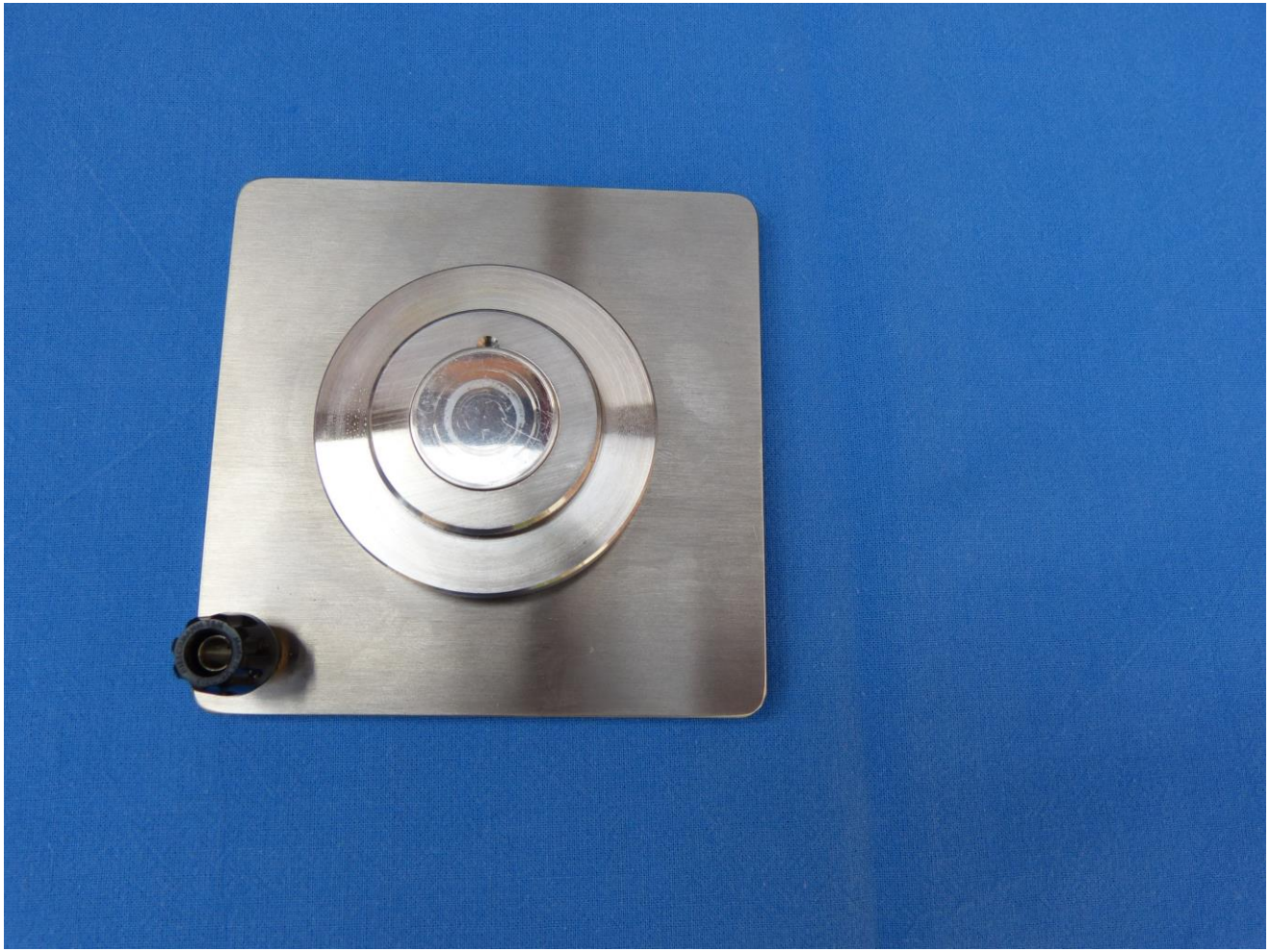
Handling-Procedure Step-by-Step

Preparation of the clean stainless-steel groundplate



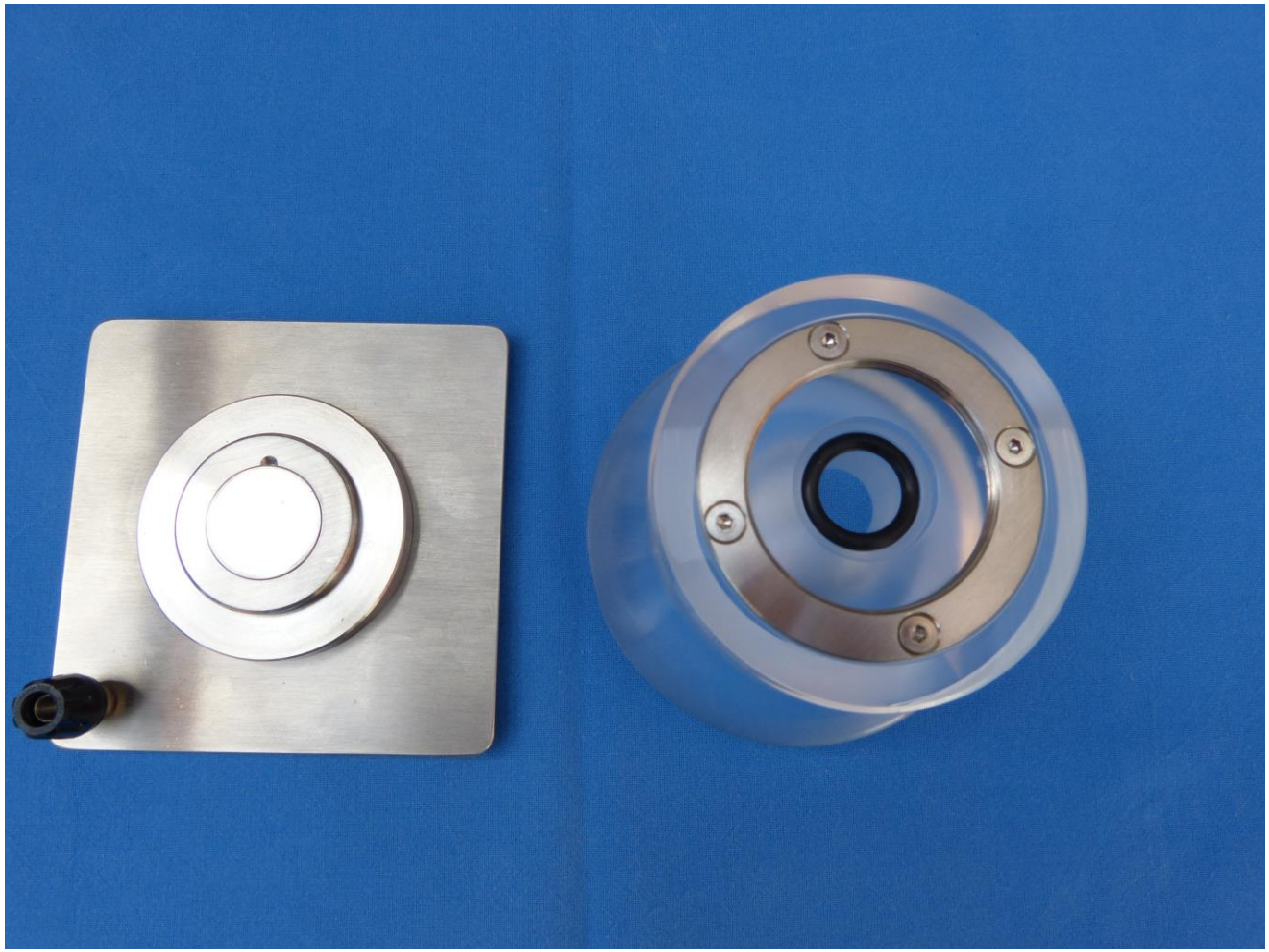
Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step
Insert the stainless-steel-planchet



Electrodeposition Cell Type N (L)

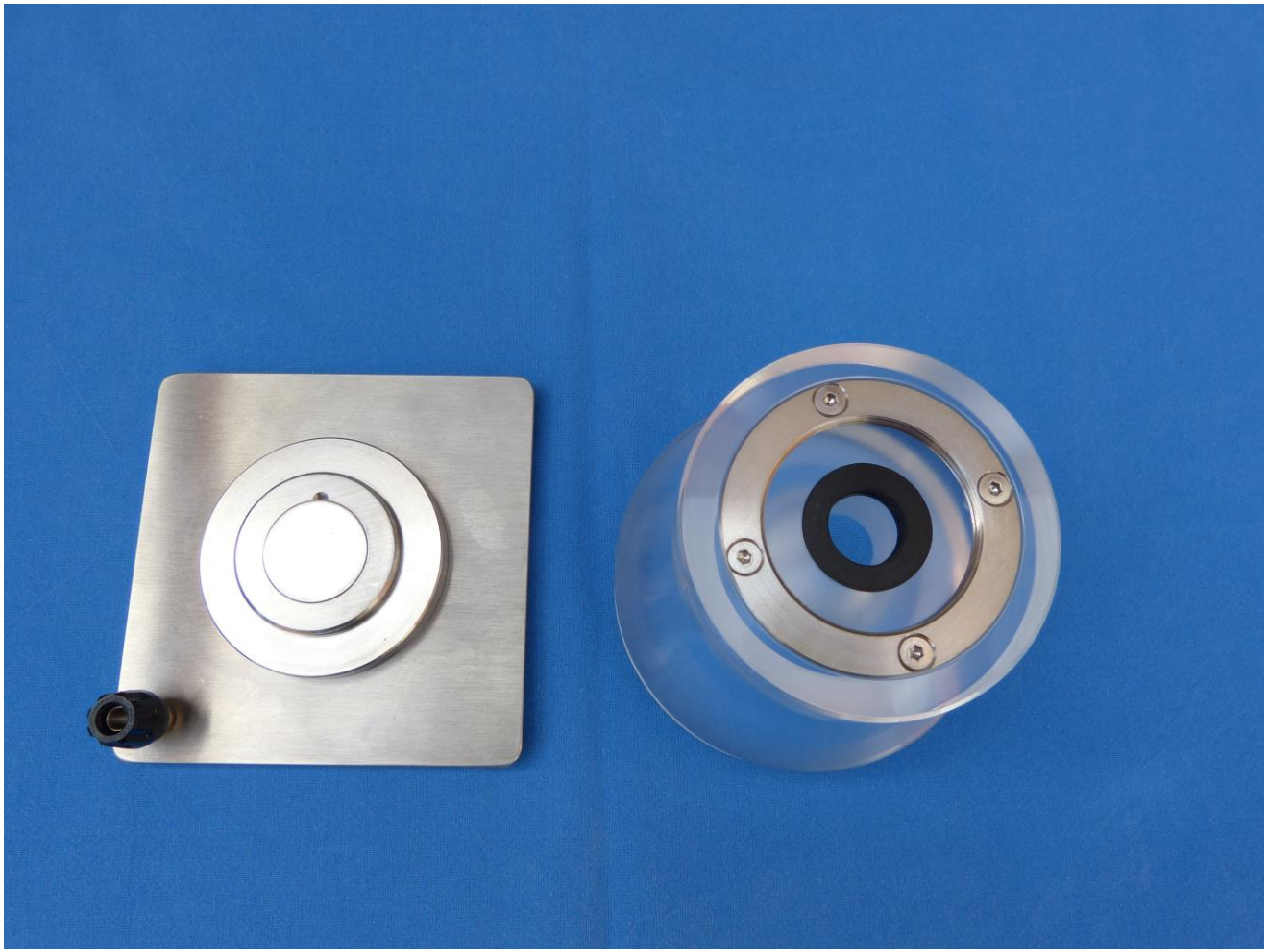
Handling-Procedure Step-by-Step
Insert the O-Ring into the cell-body



Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step

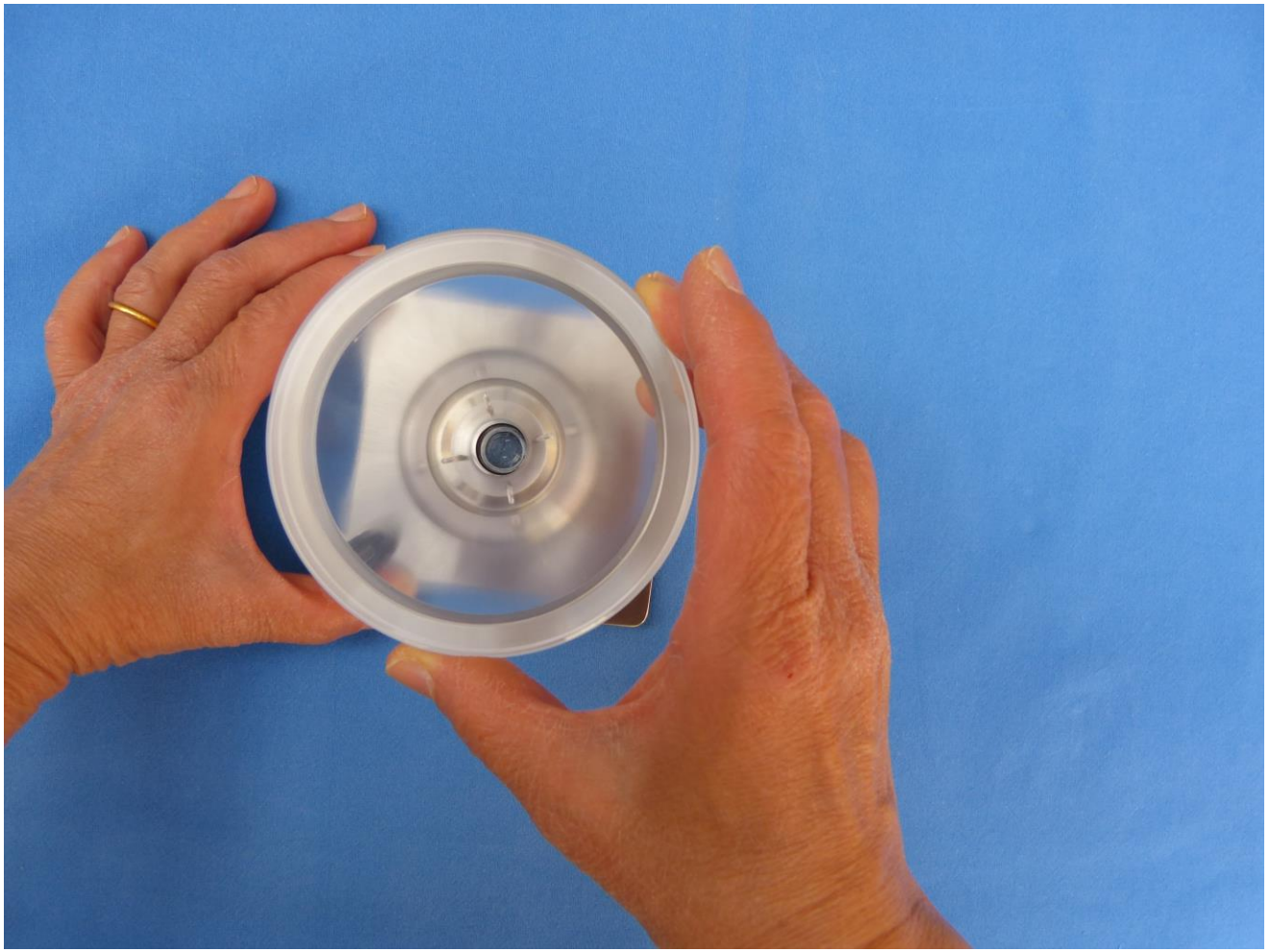
Insert the flat washer into the cell-body



Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step

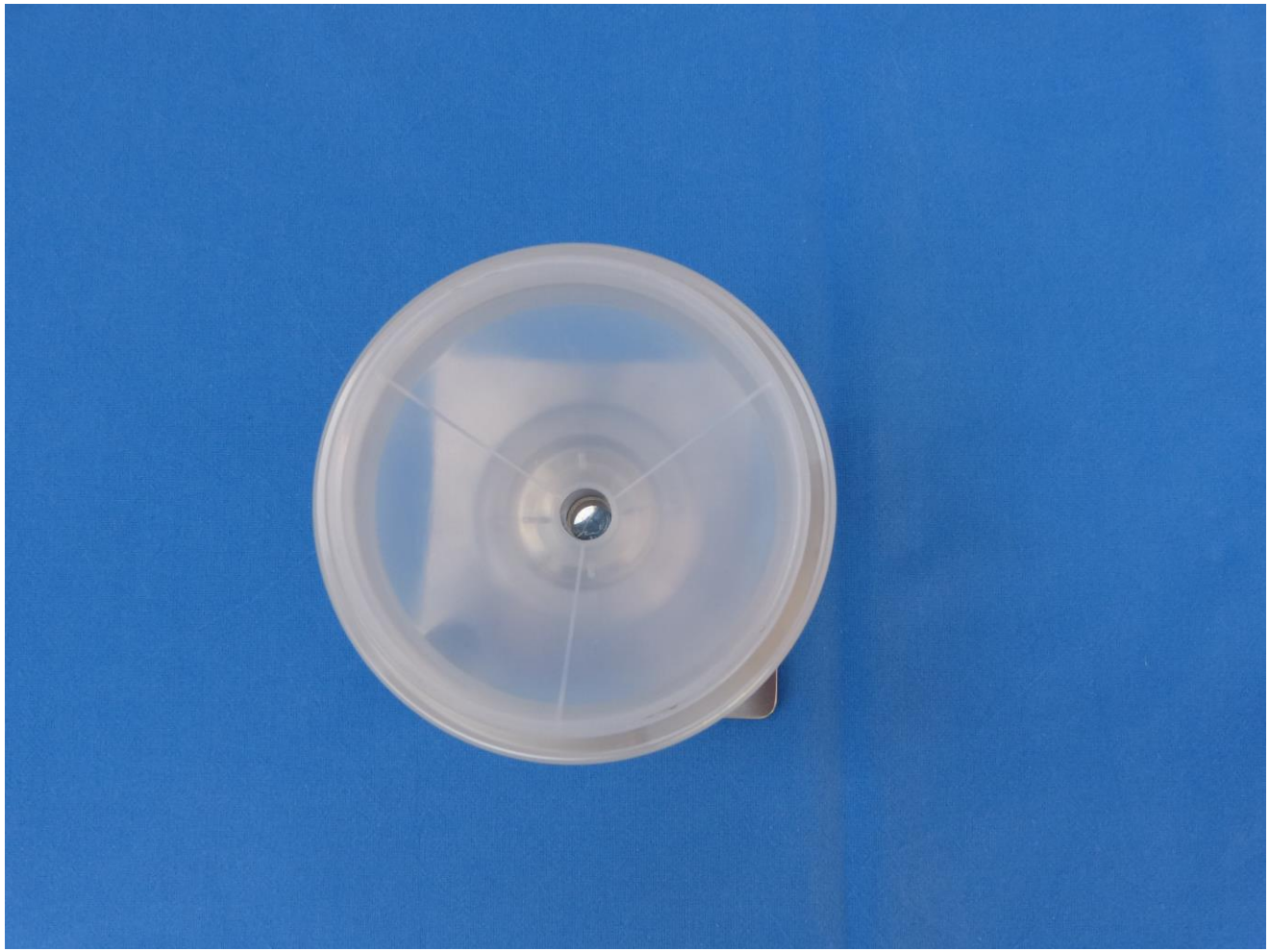
Screw the cell-body carefully on the groundplate with inserted planchet



Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step

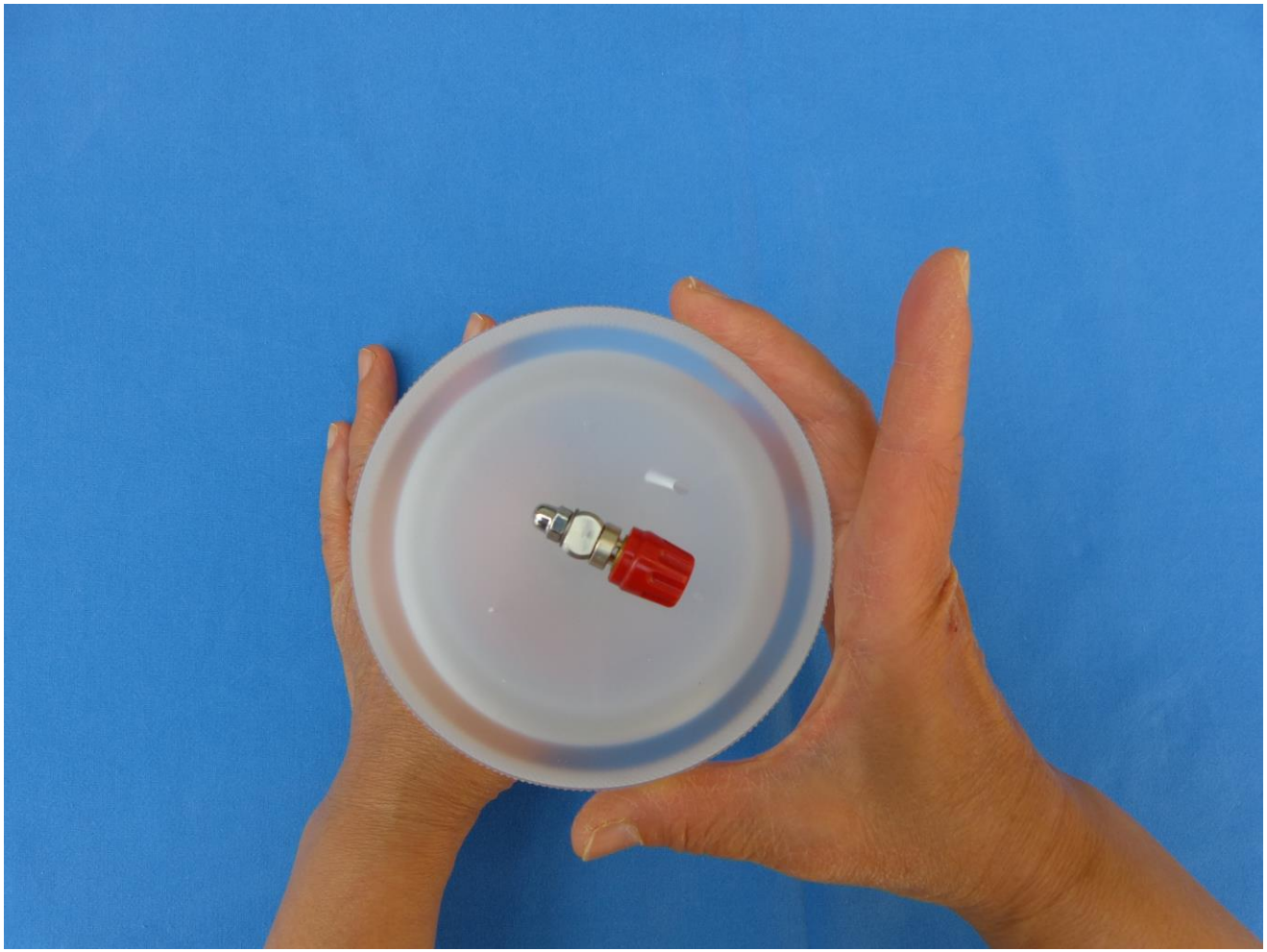
Insert the one-way plastic-funnel into the cell-body



Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step

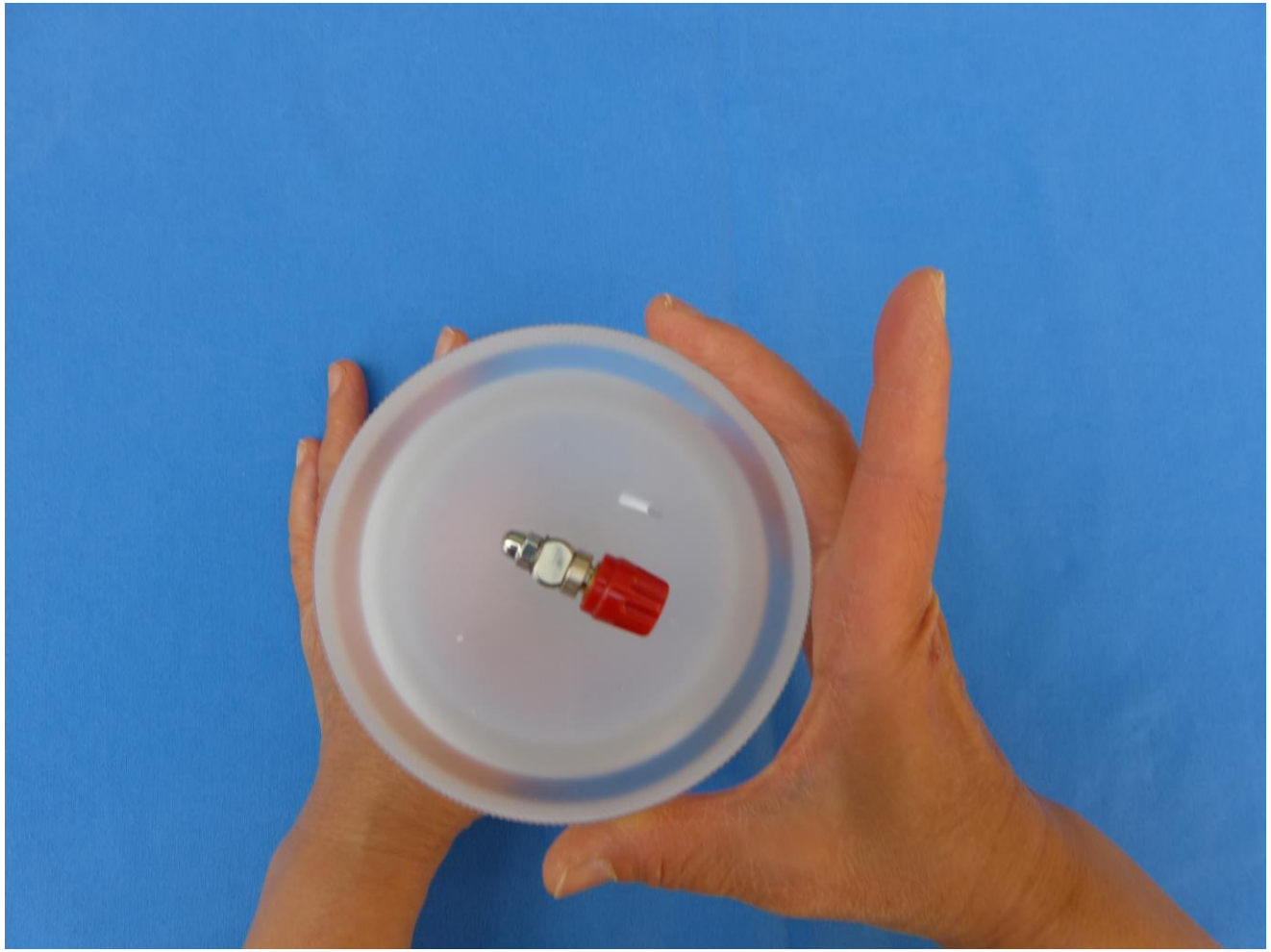
Screw the cell-cover very carefully (hand-tight) on the cell-body, this is to push-in the one-way-plastic funnel to the end-position



Electrodeposition Cell Type N-(L)

Handling-Procedure Step-by-Step

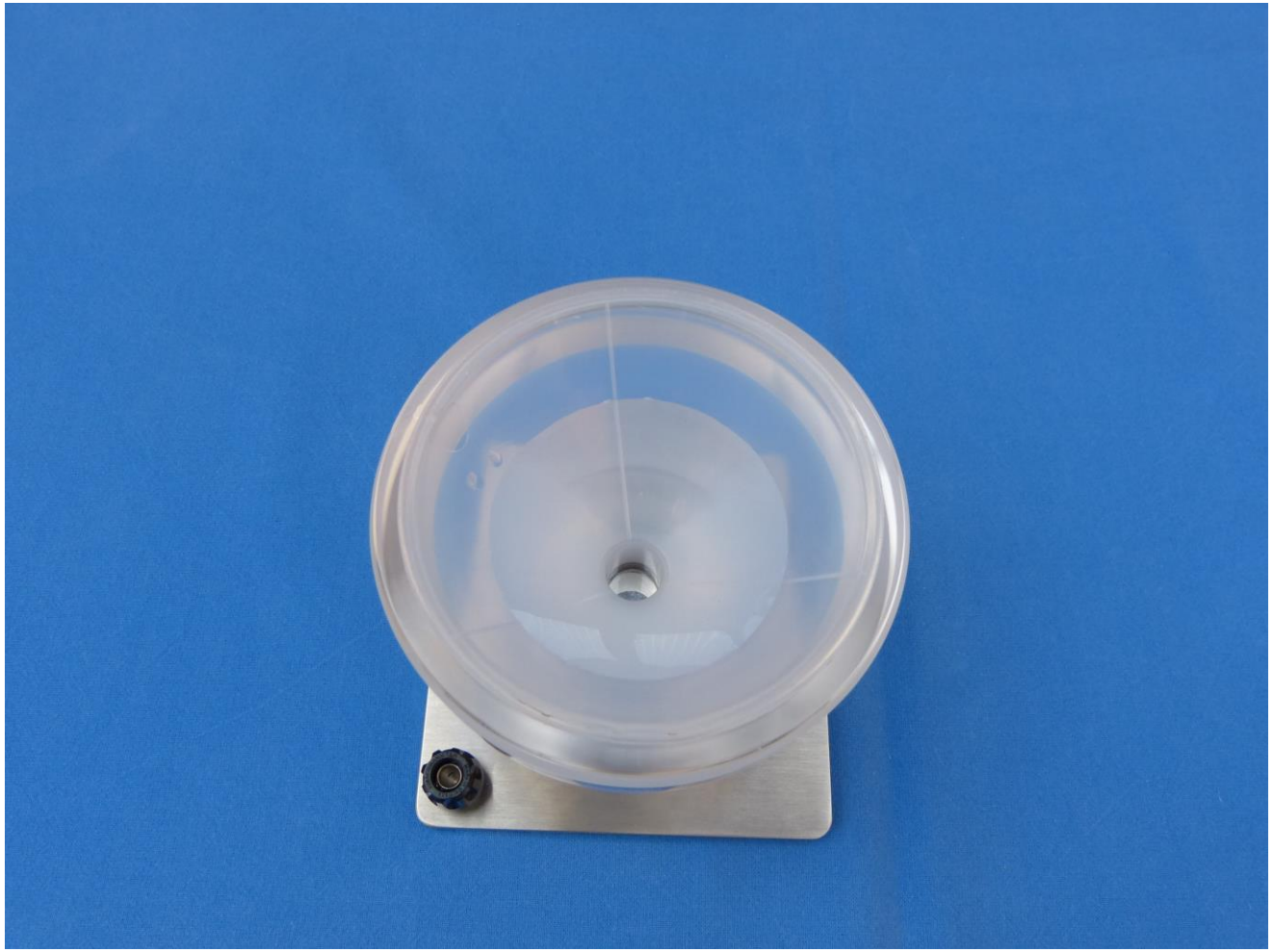
Remove the cell-cover very carefully from the cell-body



Electrodeposition Cell Type N (L)

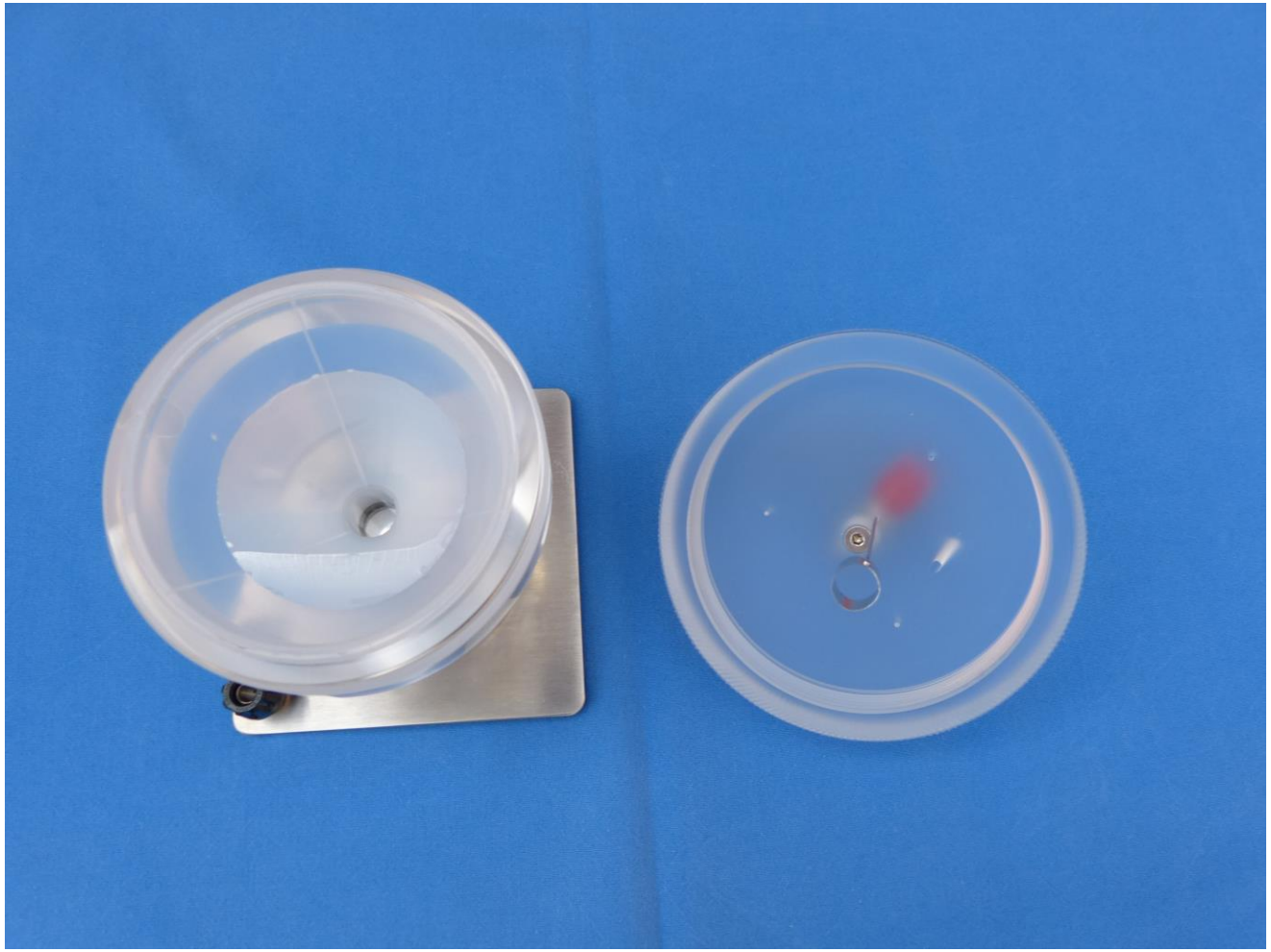
Handling-Procedure Step-by-Step

Fill-in the solution into the cell-body with the inserted one-way-plastic-funnel



Electrodeposition Cell Type N (L)

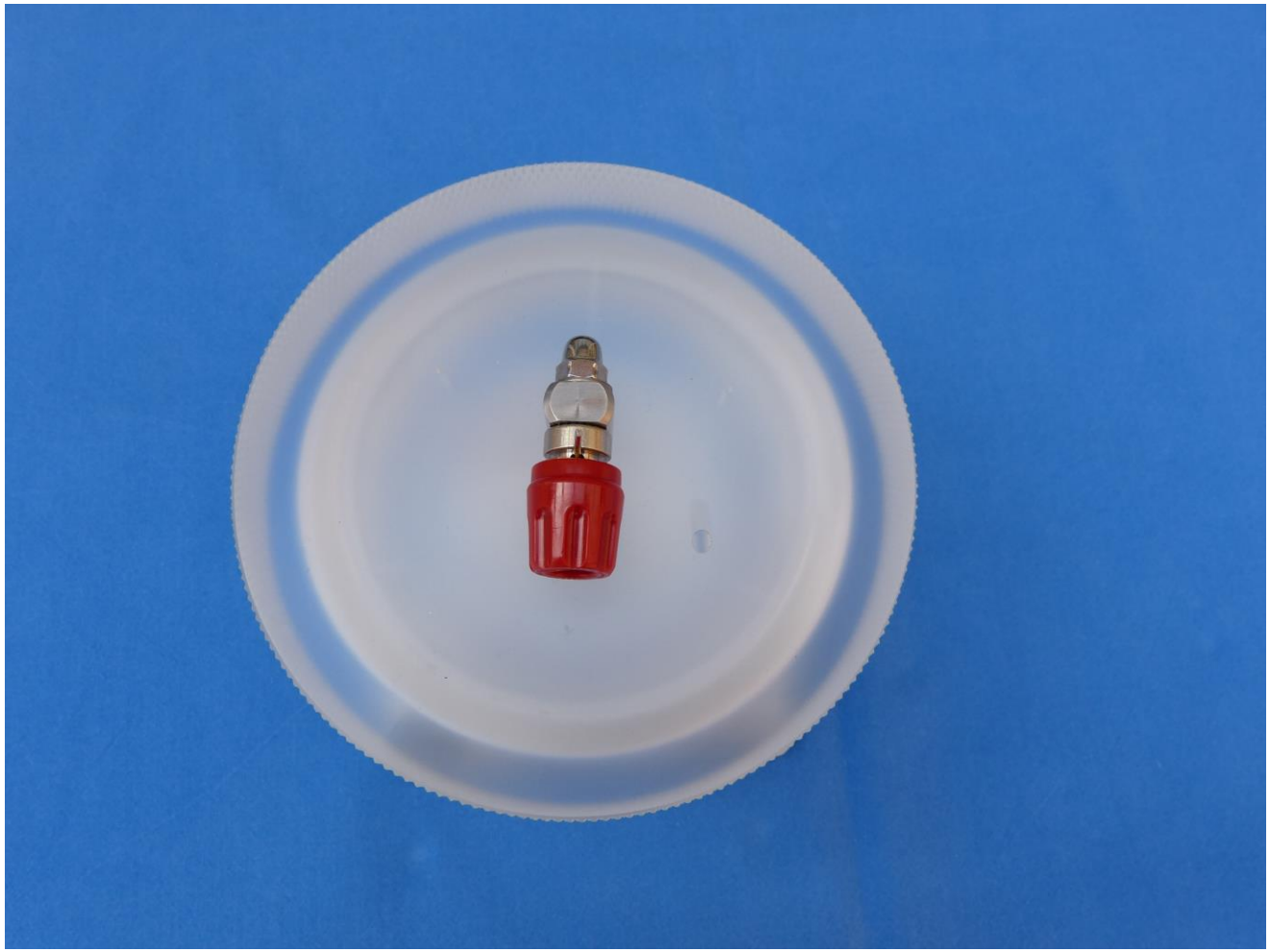
Handling-Procedure Step-by-Step
Insert and fix the Pt-Electrode into the cell-cover



Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step

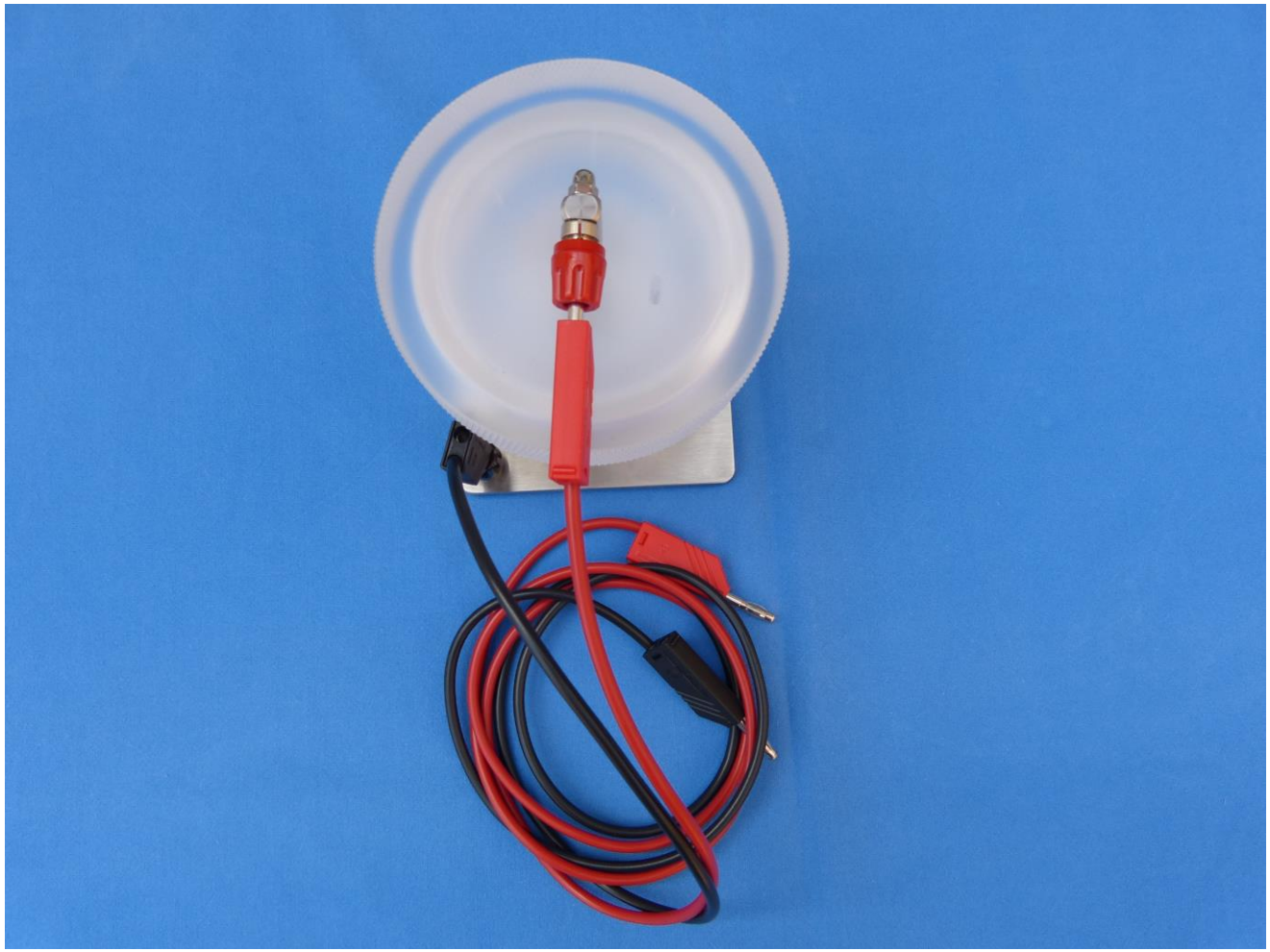
Screw the cell-cover very carefully with the inserted Pt-Electrode on the cell-body



Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step

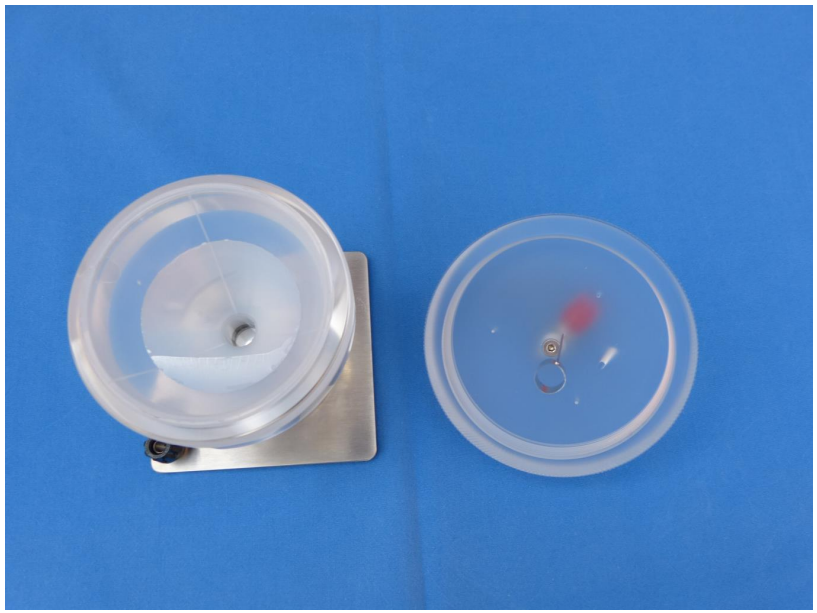
Conncet the cables to the power-supply and start the Electrodeposition-Procedure

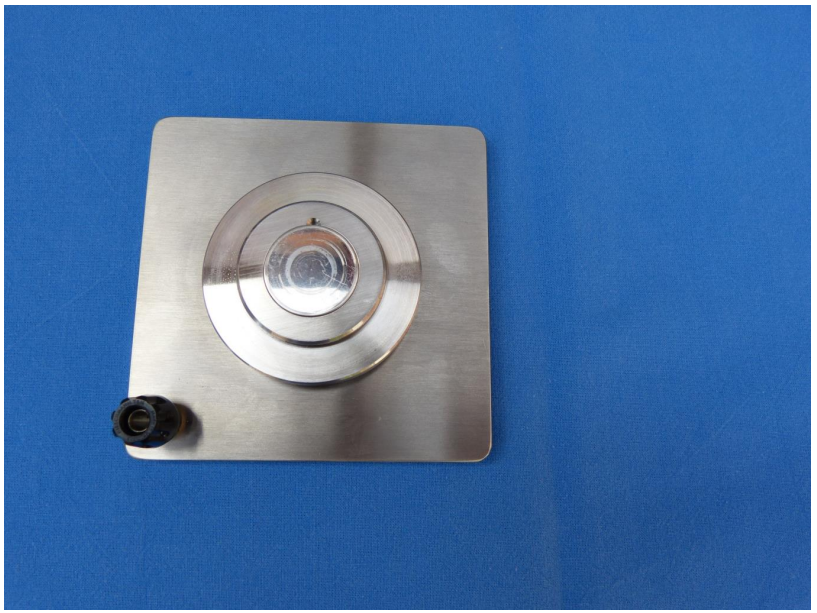


Electrodeposition Cell Type N (L)

Handling-Procedure Step-by-Step – finish the Electrodeposition-Procedure

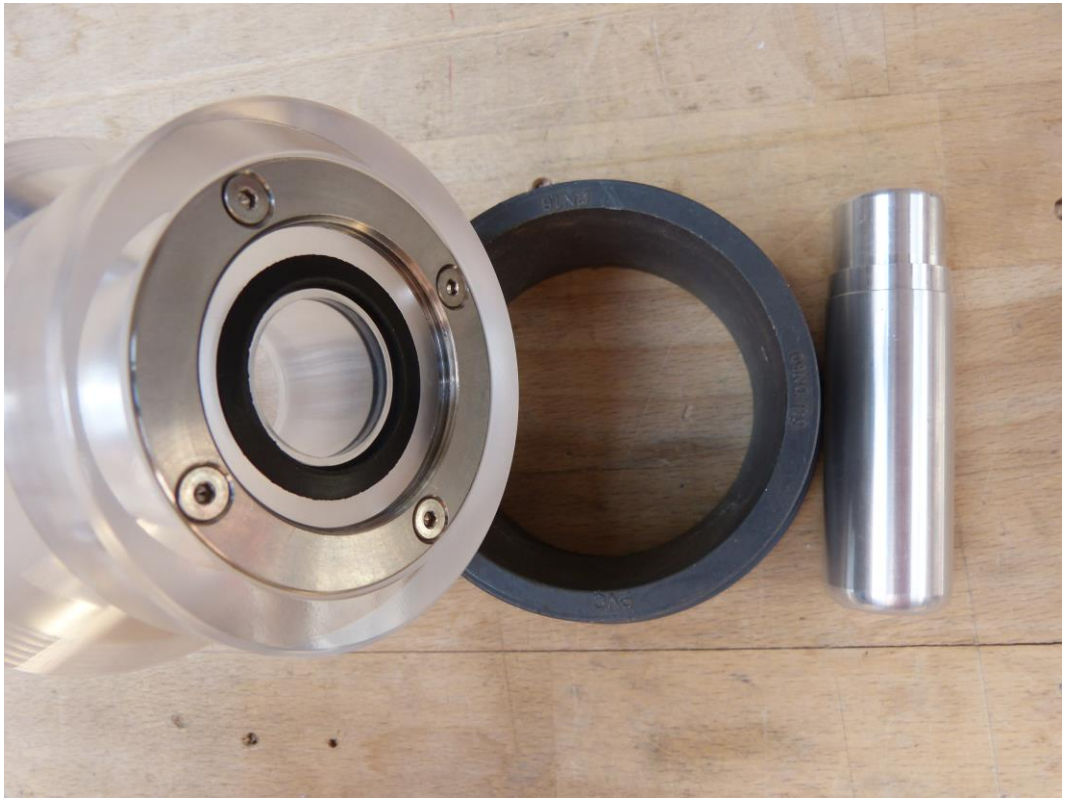
Remove very carefully the cell-cover from the cell-body
remove the remaining solution from the cell-body with inserted one-way-plastic-funnel
remove the planchet for the analysing-procedure
clean up the electroxdeposition-cell-parts for the next procedure





ALPHA-ELEC-N (L)

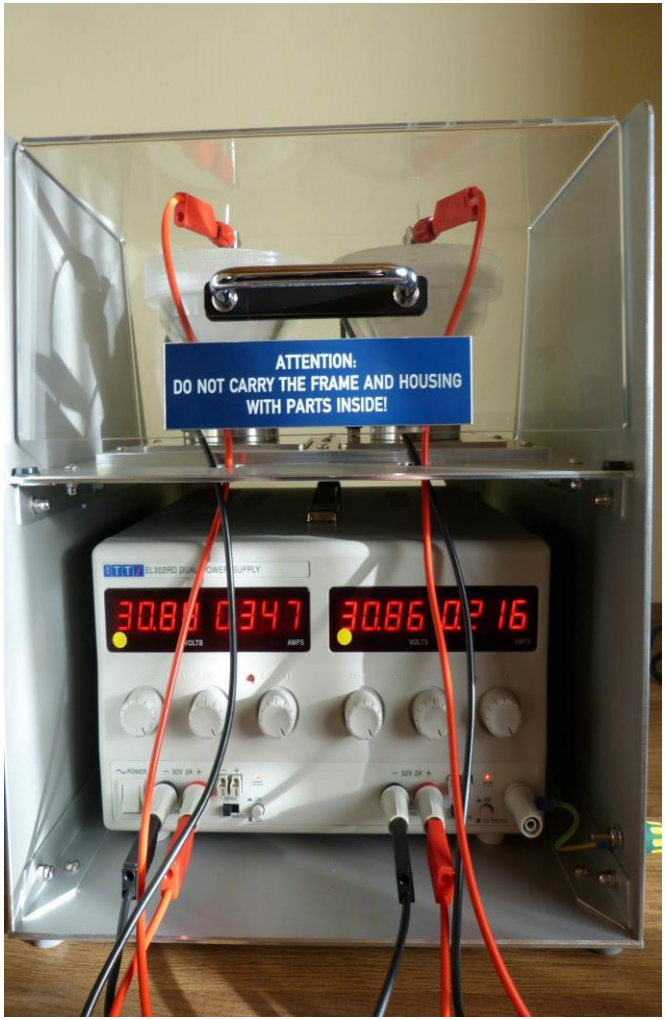
Tool to remove the one-way-plastic-funnel



ALPHA-ELEC-N (L)

Plexi-Cell-Body with the inserted tool to remove the one-way-plastic-funnel

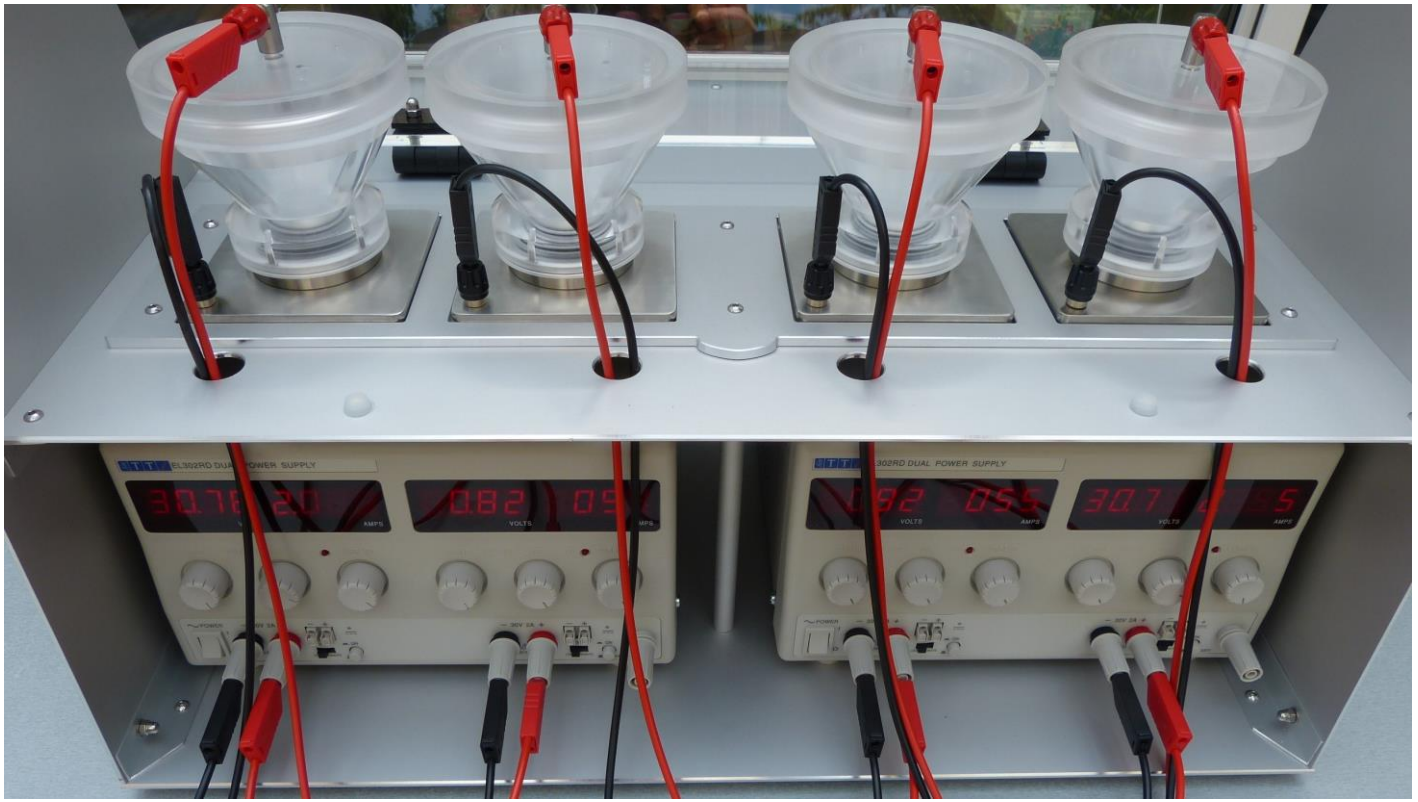
ALPHA-ELEC-2-N-(L)
2 Channel in a frame and housing



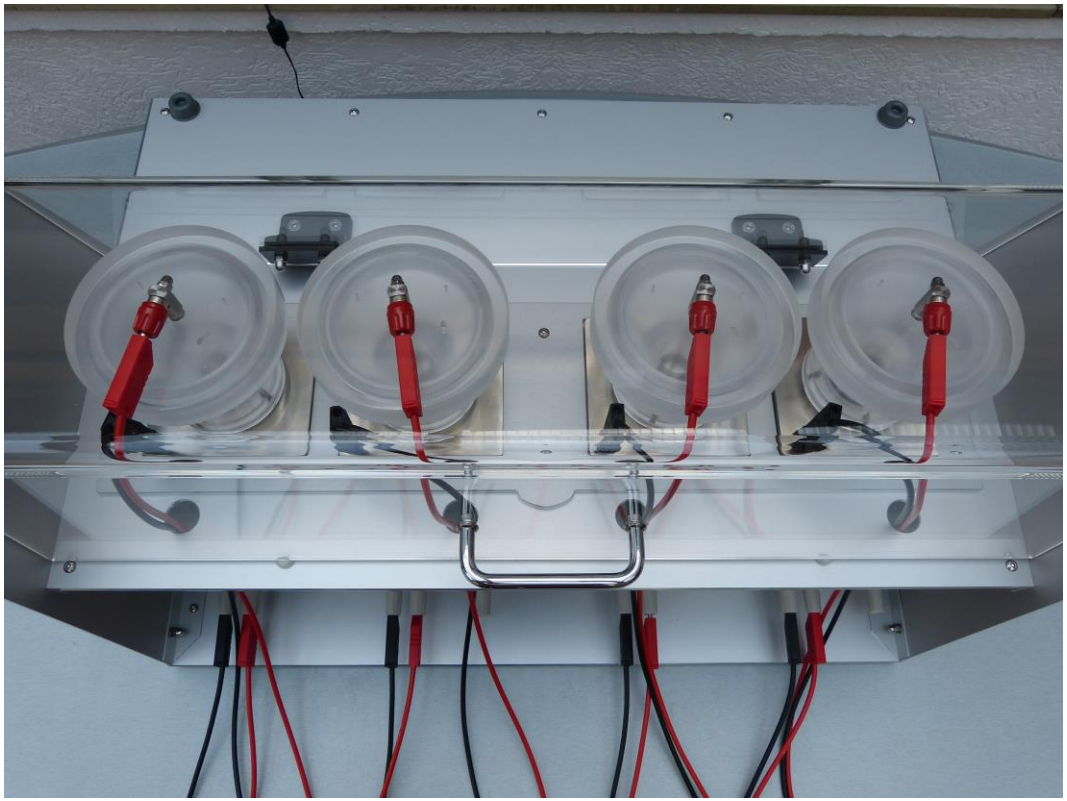
ALPHA-ELEC-2-N-(L) – 5 Amp.-Version 2 Channel in an operational frame

ALPHA-ELEC-2-N-(L) – 5 Amp.-Version 2 Channel in an operational frame

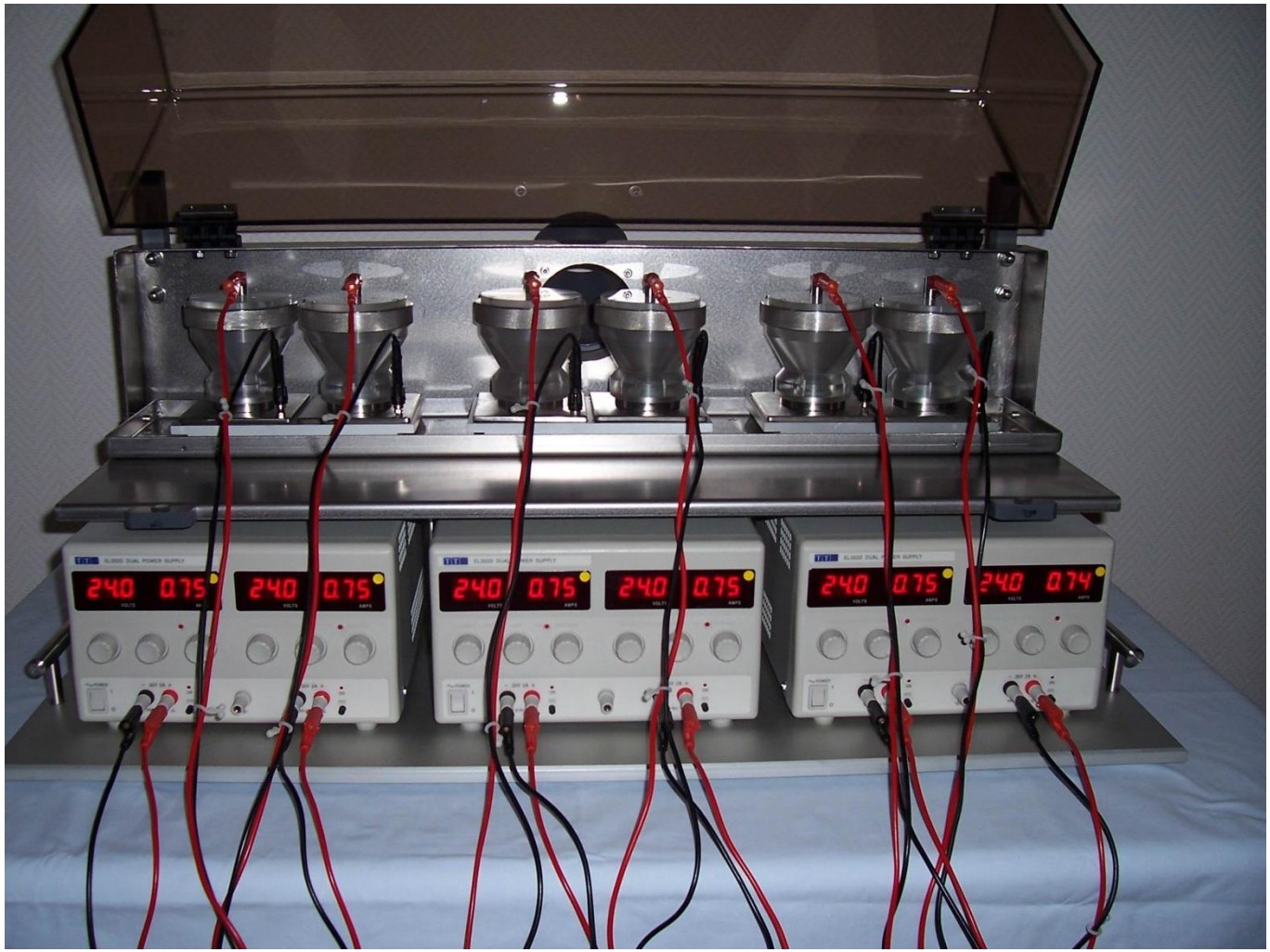
ALPHA-ELEC-4-N-(L)
2 x 2 Channel in a frame and housing



ALPHA-ELEC-4-N- (L)
2 x 2 Channel in a frame and housing



ALPHA-ELEC-6-N-(L)
3 x 2 Channel in a 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.