

APPENDIX

INSTRUCTIONS FOR USE OF THE ELECTRODE ASSEMBLY TYPE E63

SECTION A1

GENERAL DESCRIPTION

A1.1 USES

The Electrode Assembly, type E63, is used with a Polariter, type PO or PO4, as a setup for conventional polarography.

A1.2 OPERATING PRINCIPLE

The equipment is shown in the drawing at the back of the instruction manual. The electrolysis cell consists of a calomel half-cell (type K500), the electrolysis vessel with the dropping mercury electrode, and an agar salt bridge that connects the calomel half-cell with the test solution. The electrolysis vessel proper is a test tube (25^ø x 75 mm), which is mounted around a rubber stopper with holes for agar bridge, capillary, gas inlet tube, and outlet for gas. The whole cell is mounted in a holder on an arm that faces downwards so that the cell can easily be immersed in a water thermostat.

The arm is mounted on a frame that carries an ascending tube with a scale and an adjustable index as well as an adjustable support for the mercury reservoir. By means of this arrangement it is easy to maintain the mercury pressure required. The entire system can be raised or lowered on the stand without changing the mercury level in relation to the tip of the capillary.

A needle valve is mounted on the frame for fine control of the flow of the inert gas. The gas inlet tube fits loosely into a short piece of tube in a hole in the stopper and is held in place by a clip on the arm in such a way that it can easily be lowered to let the gas bubble through the solution or raised so that the gas flows over the solution and forms a protective layer above it.

If a pool of mercury is desired as a non-polarizable electrode instead of the calomel half-cell, remove the latter and replace the bridge by a type P111 anode lead that forms the connection to the mercury pool.

SECTION A2 MOUNTINGS

A 2.1 SPECIFICATIONS

	type
1 holder for Hg ascending pipe	D101
1 electrode holder	D102
1 support for mercury reservoir	D103
2 stops	D105
1 index of Hg level scale	D106
1 clip for gas tube	D107
1 clip for anode lead	D108
1 needle valve for inert gas	D109
1 stand	D110
1 mercury reservoir	V400
1 Hg ascending pipe	B601
1 length of plastic tubing for mercury	B701
1 length of plastic tubing for mercury	B702
1 length of rubber tubing for inert gas	B705
2 glass tubes for inert gas	B600
2 Hg capillaries	B400
1 electrolysis vessel	V500
1 rubber stopper with 3 holes	B801
3 KCl-agar salt bridges	B500
1 calomel half-cell	K500
1 cathode lead	L301

Extra accessories:

platinum electrode	P111
titration vessel (with KCl-agar plug)	V600
titration vessel (with NH_4NO_3 -agar plug)	V601
calomel inner electrode for V600 vessel	K510

A 2.2 MOUNTINGS

A 2.11 Mounting and replacing the capillary

- (1) Place the mercury reservoir in its lowest position.
- (2) Close the B702 tubing near the capillary with a screw compressor with swivel jaw.
- (3) Remove the capillary from its holder so that the B801 rubber stopper is free.
- (4) Pull the capillary out of the B702 tubing.
- (5) Replace the capillary in the rubber stopper.
- (6) Put on the tubing.
- (7) Mount the capillary in its holder.
- (8) Open the screw compressor and make sure that no air bubbles are trapped in the B702 tubing.

A 2.12 The reference system

- (1) Place the calomel electrode in the electrode holder, D102.
- (2) Fix the thumb screw around the plastic-covered part of the electrode.
- (3) Refill the electrode to an adequate level with saturated KCl solution.
- (4) Immerse the free end (the short one) of the salt bridge in the side tube of the calomel cell.
- (5) Fix the electrode setup in the holder of the capillary.

SECTION A3

INSTRUCTIONS FOR USE

A 3.1 MEASUREMENT

- (1) Mount the electrode setup as shown at the back of the instrument manual. Follow the above instructions.
- (2) Refill the mercury reservoir, if necessary. Use pure mercury (distilled twice).
- (3) Connect the inlet of the needle valve to the reduction valve of a nitrogen cylinder, and the outlet of the needle valve to the B600 glass tube.
- (4) Pour about 10 ml of the solution to be analyzed into the vessel, type V500, and press the vessel onto the rubber stopper.
- (5) If the sample is to be deoxygenated, bubble with nitrogen gas for 10-15 minutes. Regulate the gas flow with the needle valve.
- (6) After the bubbling, raise the B600 tube for inert gas and keep the gas flowing over the surface of the sample.
- (7) Use the cathode lead to connect the CATHODE binding post of the Polariter with the terminal bore of the capillary electrode, below the mercury ascending tube.
- (8) Connect the lead of the calomel cell to the ANODE binding post of the Polariter.
- (9) Raise the mercury reservoir until the drop frequency is suitable.
- (10) Record the polarogram.

A 3.2 AFTER USE

- (1) Place the reservoir in its lowest position and close the B702 tubing with a screw compressor.
- (2) Remove the sample.
- (3) Rinse the ends of the capillary, the salt bridge, and the gas inlet tube with distilled water, to remove any drops of the sample.
- (4) Mount a vessel of unsaturated KCl solution in place of the sample. (Unsaturated solution is less likely to clog the capillary.)

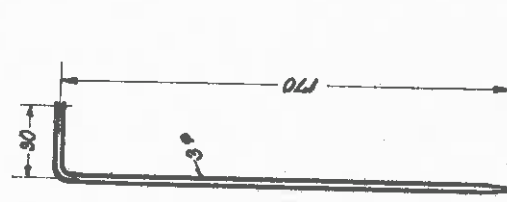
A 3.3 MAINTENANCE AND HANDLING

- (1) Always keep the level of the saturated KCl solution in the calomel cell higher than the end of the salt bridge.
- (2) Never let the other end of the salt bridge contact the sample for a long time when the apparatus is not in use (see item A 3.2.).
- (3) Bear in mind that mercury is poisonous. Inhalation of mercury vapors, even at room temperature, may lead to poisoning. All traces of spilled mercury should be carefully removed. Large

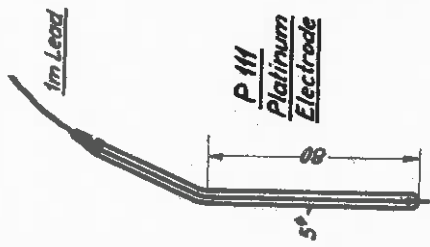
drops can be picked up by a suction flask with a short capillary as an inlet tube. A piece of amalgamated zinc or tin solder will be useful for picking up small inaccessible drops.

It is recommended that the Electrode Assembly be placed on an enamel or stainless steel tray with high edges.

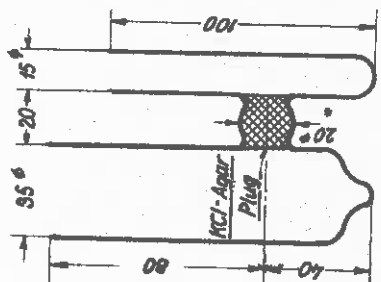
Persons wearing gold and silver jewelry and watches should be careful not to bring them in contact with mercury, because gold and silver will amalgamate with mercury.



B 600
Tube for
Inert Gas

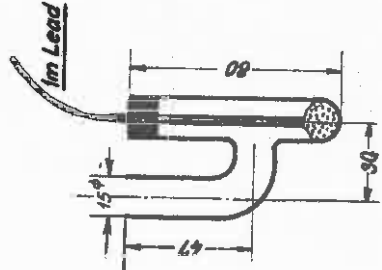


P 111
Platinum
Electrode



V 500
Standard Vessel

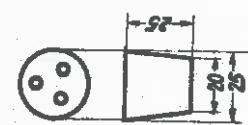
V 600
Titration Vessel



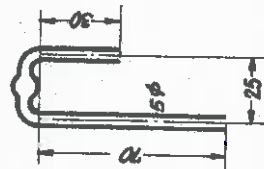
K 500
Calomel Half-Cell



K 510
Calomel
Inner Electrode



B 801
Rubber Stopper



B 500
MCI-Agar Saltbridge

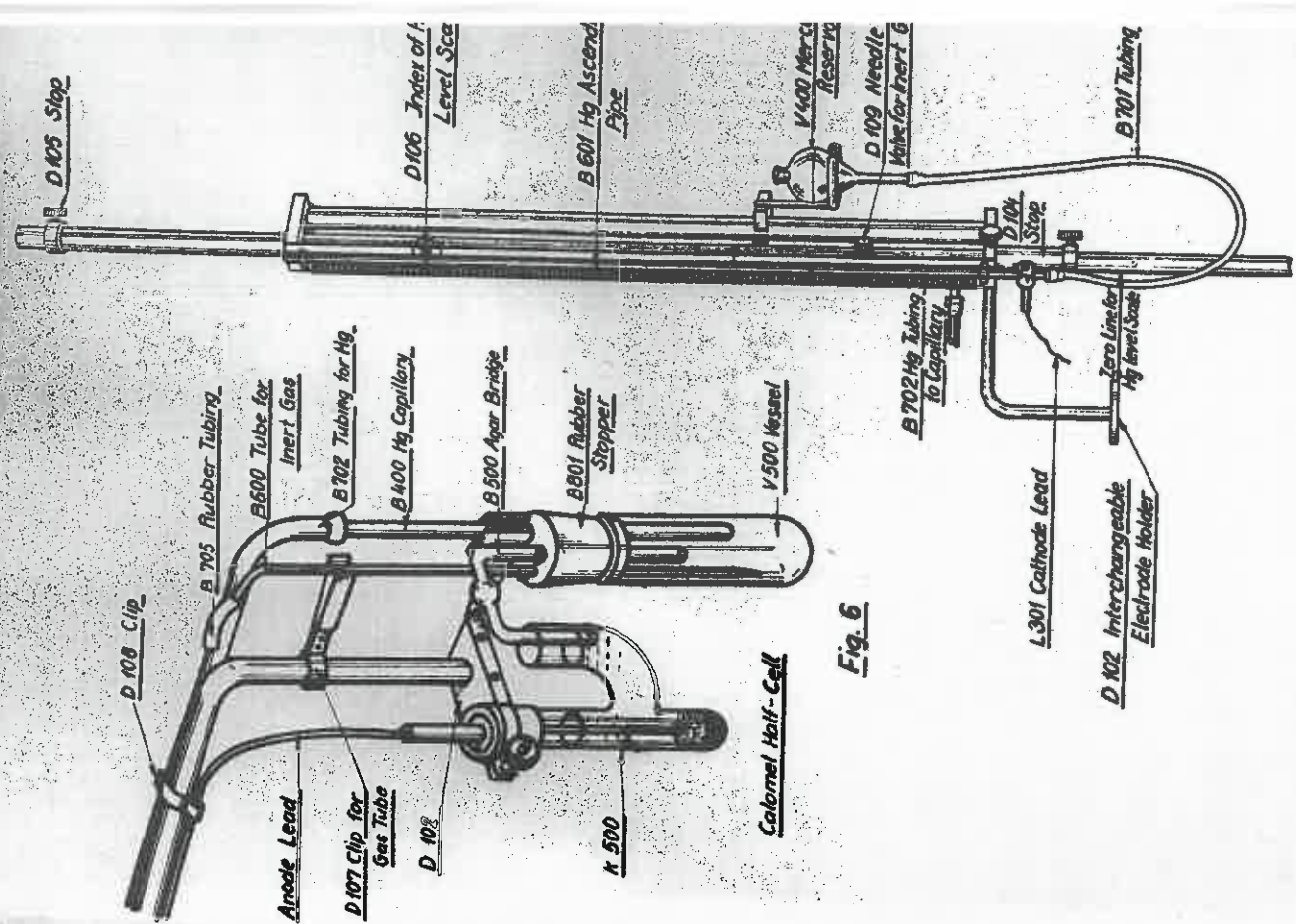


Fig. 6

Calomel Half-Cell

Fig. 7

Details of Electrode Assembly
Type E 63

Spare Parts for
Polarographic Cells