

**Instruction Manual
for**

**Electrode Assembly
Type E 65**

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Electrode Assembly Type E 65

Section A. General Description

1 USES

The Electrode Assembly, type E65, is used with the Polariter, type PO4, and the Drop Life Timer, type DLT1, for polarography with controlled drop life - e.g. Rapid Polarography and Tast Polarography. However, the polarographic setup is still suitable for conventional polarographic analyses as described in the instruction manuals for the Polariter, type PO4, and the Electrode Assembly, type E64.

By means of the Kit for Drop Life Control, type D453, it is possible to convert an Electrode Assembly of the type E64 into the above mentioned Electrode Assembly, type E65.

2 THE HAMMER OF THE ELECTRODE ASSEMBLY

The Electrode Assembly, type E65, is set up in the same way as the Electrode Assembly, type E64, but in this case the Dropping Mercury Electrode is a specially formed, internally siliconized capillary, type B405. This capillary is placed inside a steel tube seated on the electrode head, D4561, and above this the hammer itself, type D4562, is mounted, (The electrode head, type D4531, as used with the Electrode Assembly, type E64, is thus replaced by the above mentioned type).

The hammer consists of a steel cylinder inside which a coil is mounted. A cylindrical armature, located within this coil,

is drawn up against a stop screw at the top of the hammer by the magnetic force of a permanent magnet.

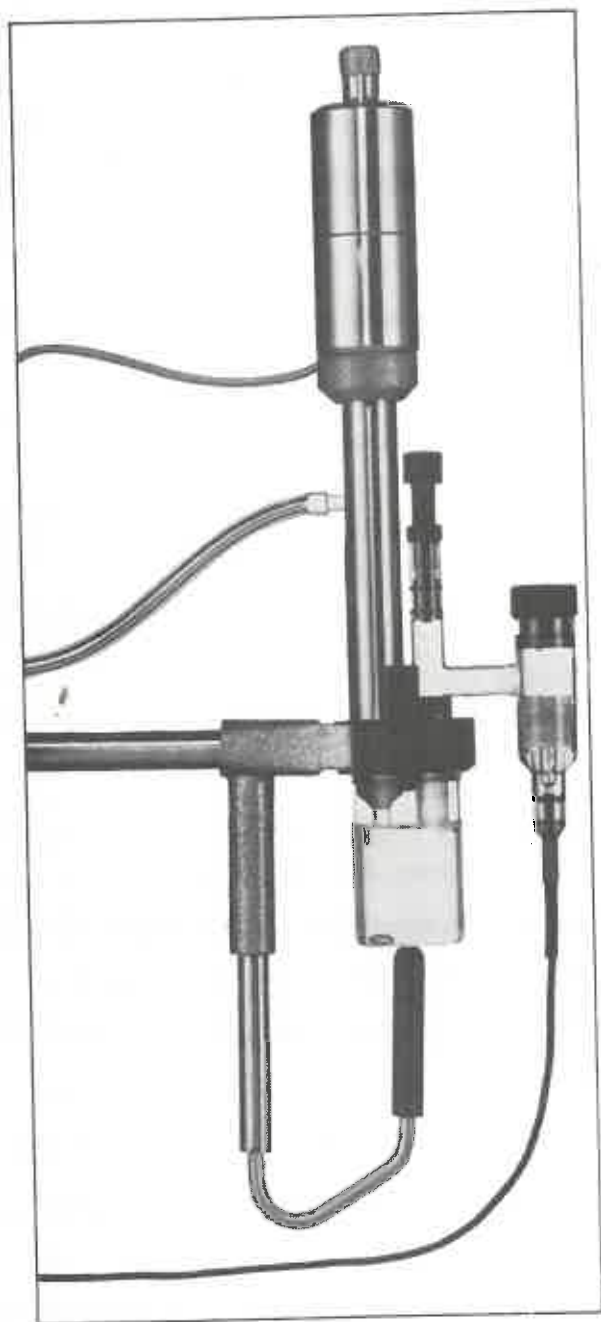


Fig.1. Photo of the Electrode Assembly, type E65.

When a current pulse is transmitted through the coil, the armature will be jerked downwards so as to strike the glass capillary lengthwise, and the mer-

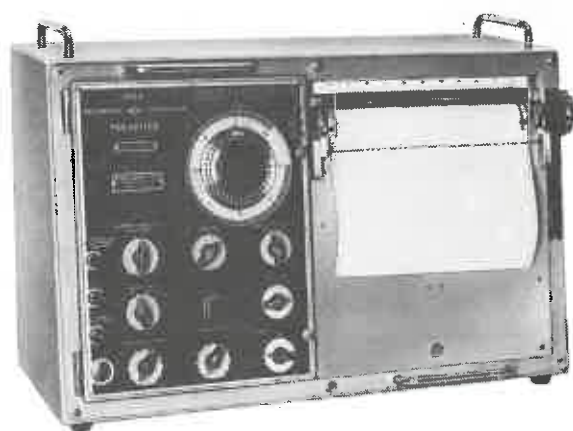


Fig.2. Photo of the Polariter, type PO4.

cury drop suspended at its tip is detached the instant the resultant downward thrust of the capillary is checked by a steel spring that is mounted inside the electrode head.

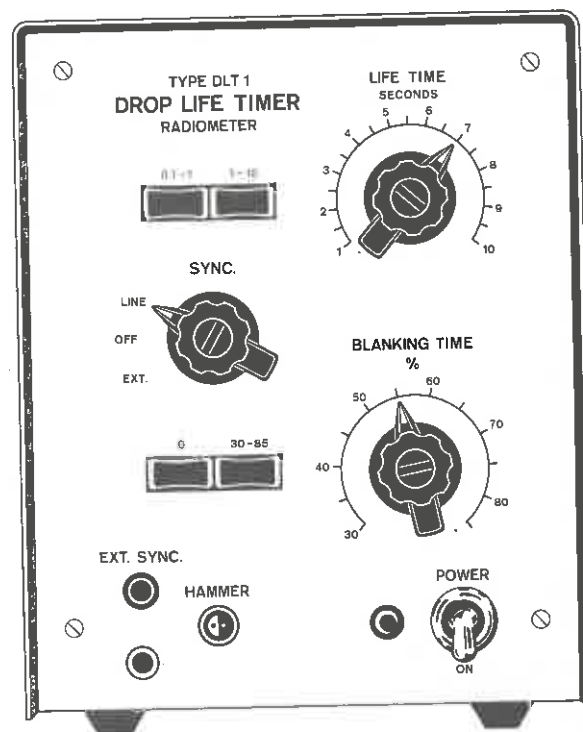


Fig.3. Photo of the Drop Life Timer, type DLT1.

Section B. Specifications

1 SPECIFICATIONS

2 Hg Capillaries		type B405
1 Stopper		- B757
1 Length of Plastic Tubing		- B7181/650
1 Stand		- D3100/110
1 Support Rod		- D4563
1 Base		- D3202
1 Clamp		- D3302
1 Gas Inlet Tube		- D4330
2 Spring Holders		- D4514
1 Electrode Head		- D4561
1 Spring	code 876-600	
2 Rubber Seal Rings	code 834-501	
1 Hammer		- D4562
1 Adapter for Metallic Electrodes		- D4532
1 Reservoir Holder		- D4533
1 Stopper with Platinum Electrode		- D4534
1 Hg Valve		- D4538
1 Rule with Clamps		- D4539
1 Calomel Electrode		- K501
1 Bottle 100 ml saturated KCl Solution		- S4004
1 Reservoir		- V402

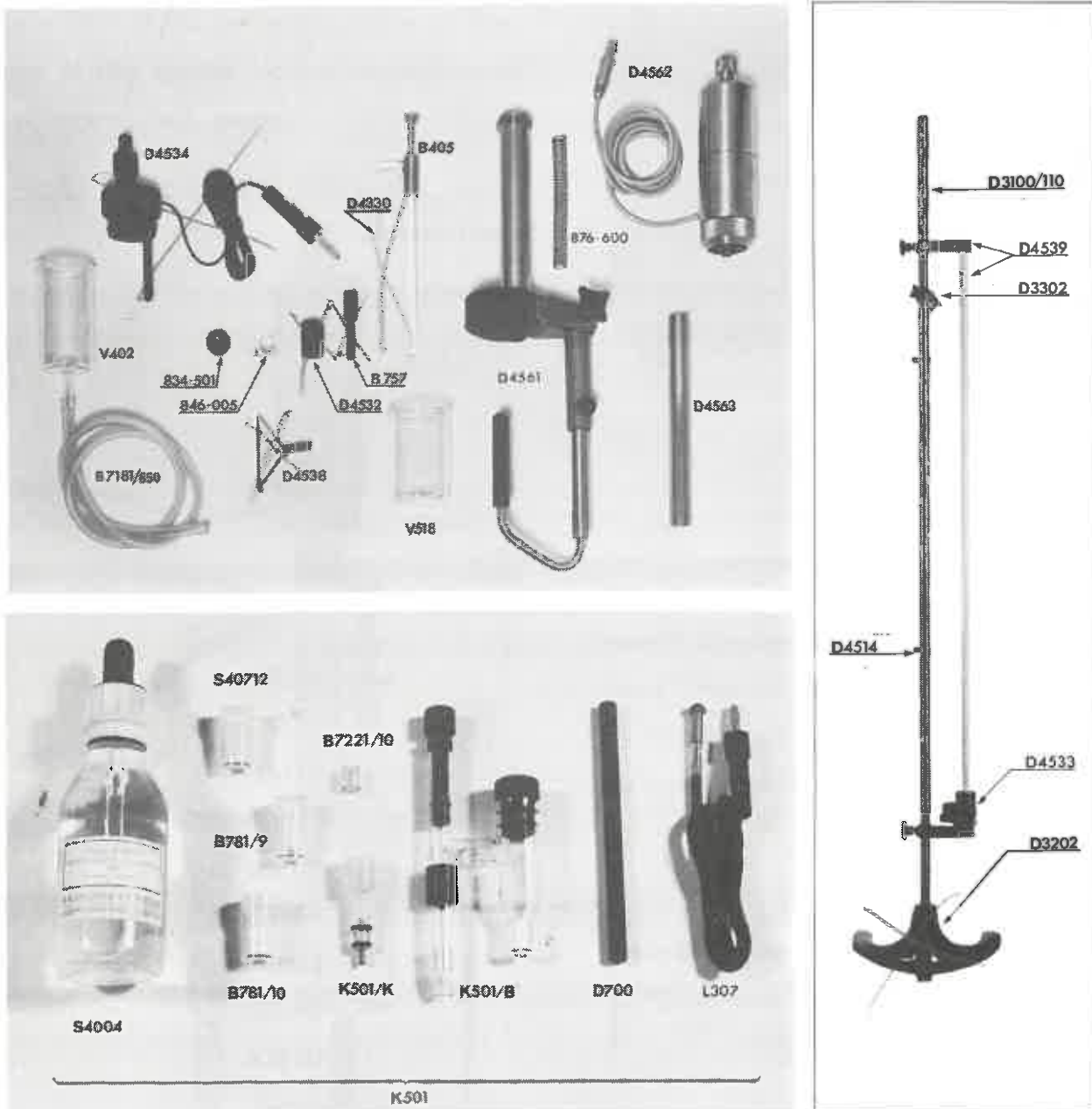


Fig.4. Designation of parts used in the Electrode Assembly, type E65.

3 Vessels

- V518

1 Fastener code 846-005

1 Instruction Manual for E65

2. ACCESSORIES

Humidifier	type D4530/V505
Vessel, thermostatted	- V519
Platinum Electrode	- P1312
Vessel, 50 ml	- V510
Rubber Cap used with V510	- B764
Vessel, 10 - 15 ml	- V521
Thermostat jacket for vessel, type V521	- V526

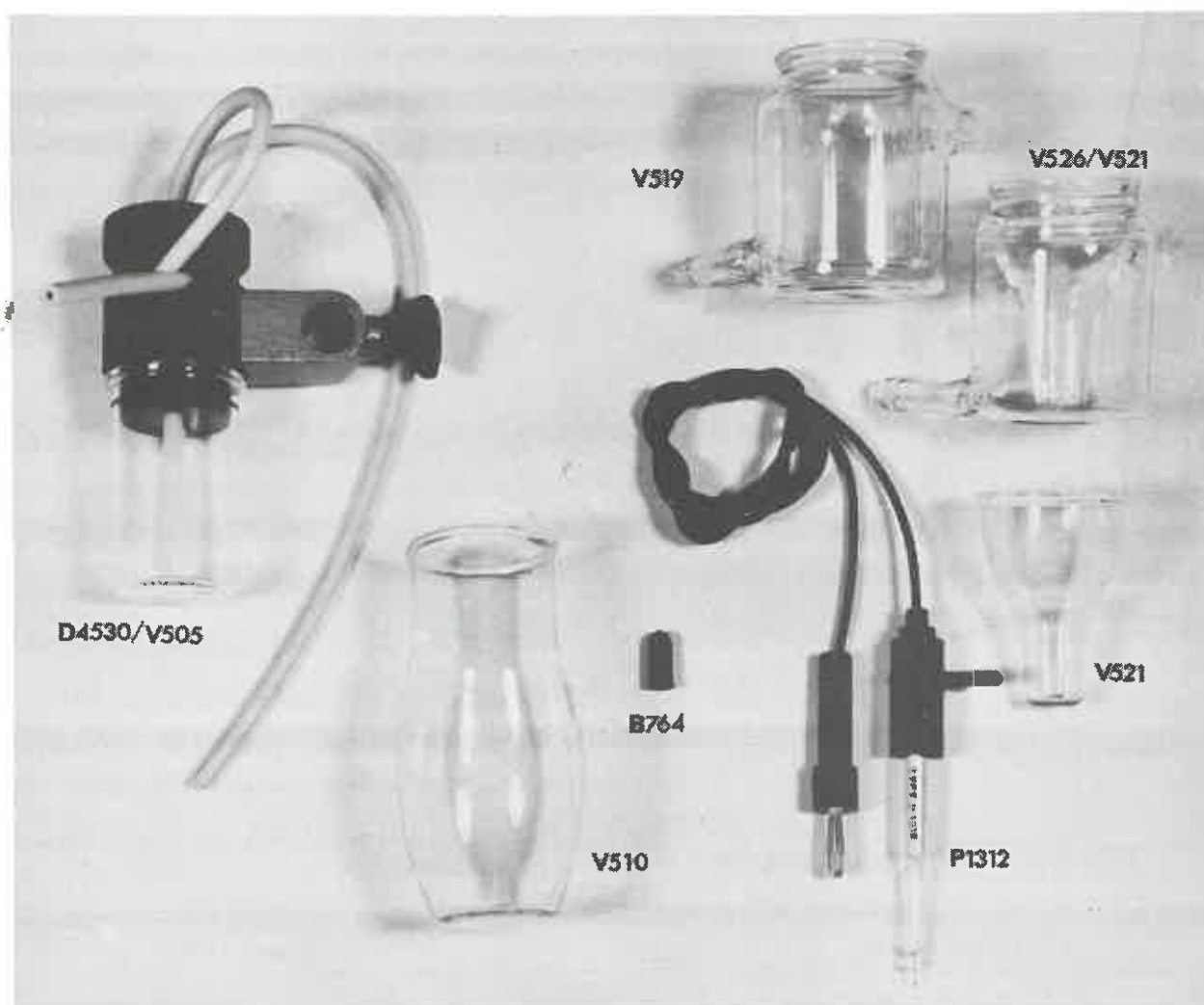


Fig.5. Accessories for the Electrode Assembly, type E65.

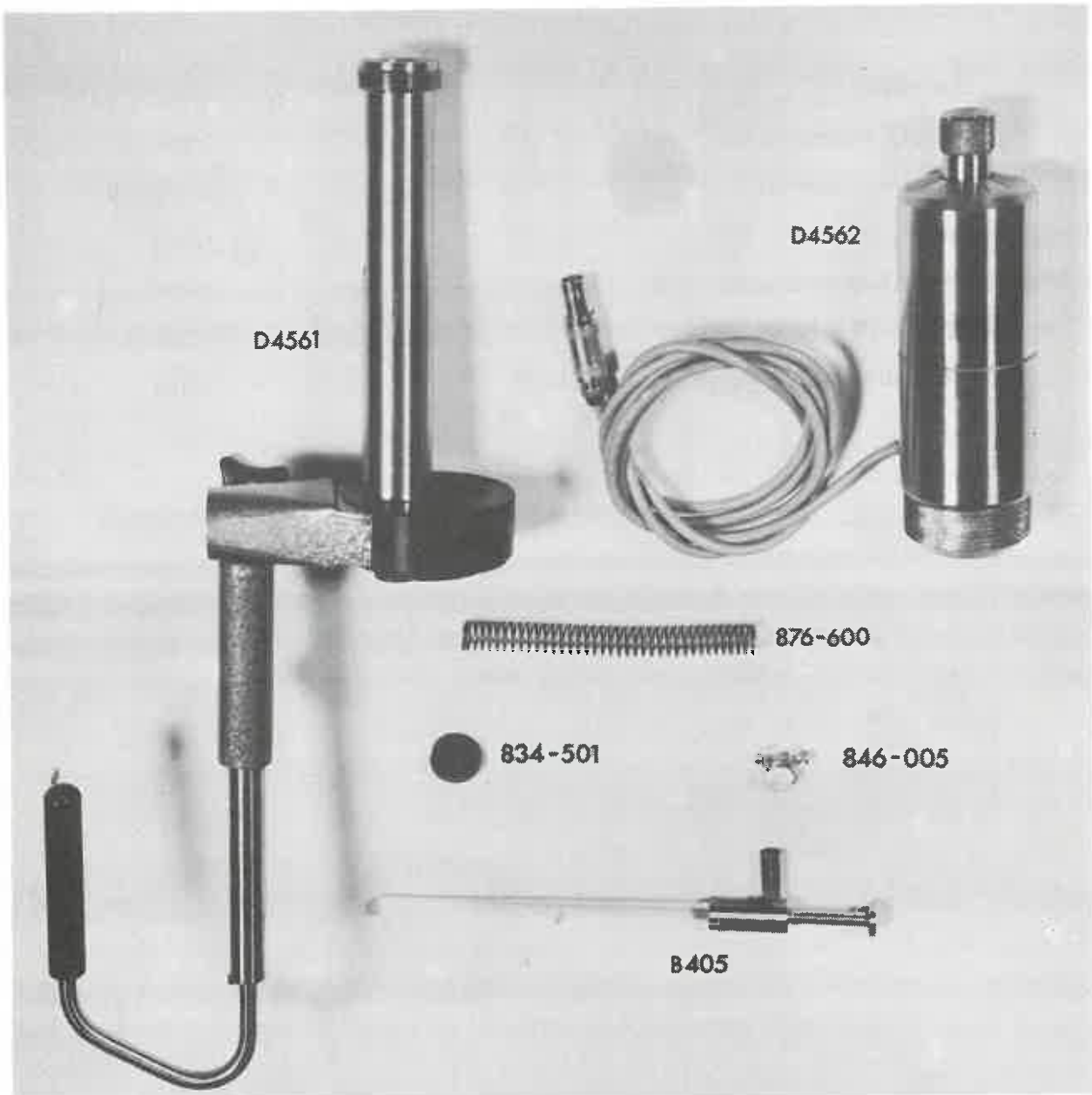


Fig.6. Parts included in the Kit for Drop Life Control, type D453.

3. SPECIFICATIONS FOR D453

2 Hg Capillaries		Type	B405
1 Electrode Head		-	D4561
1 Fastener	code 846-005		
1 Spring	code 876-600		
2 Rubber Seal Rings	code 834-501		
1 Hammer		-	D4562
1 Instruction Manual for E65			

Section C. Mounting

1. TO MOUNT THE LABORATORY STAND WITH AN ELECTRODE HEAD

- (a) Mount the stand, D3100/110 in the hole in the wide arm of the C-shaped base D3202. (See Fig.7).
- (b) Remove the upper clamp from the rule D4539 by unscrewing the top screw of the rule.
- (c) Place the reservoir holder, D4533, on the perspex tube of the rule with the nut downwards.
- (d) Fasten the reservoir holder to the perspex tube by tightening the nut.
- (e) Remount the clamp on the rule.
- (f) Mount the rule on the stand. Proceed as follows:
First place the lower clamp of the rule on the laboratory stand, next the clamp, D3302, and finally the upper clamp of the rule.
- (g) Fasten the rule to the laboratory stand so that the lower clamp is about 5 cm above the base, and so that the rule is placed to the left of the laboratory stand.
- (h) Fasten the clamp, D3302, at the "5 cm" mark of the rule with the free hole to the right.
- (i) Mount the support rod D4563 (or D3000/125) on the stand by means of the clamp D3302.
- (j) Mount the support rod D4563 on the stand by means of the clamp D3302.

In cases where the setup is supported by a base without rubber feet, it should be recommended to place the base on a rubber mat (abt. 5 mm thick) in order to dampen the vibrations caused by the hammer strokes.

2. TO MOUNT THE Hg CAPILLARY AND THE MERCURY RESERVOIR

- (1) Mount the plastic tubing B7181/650

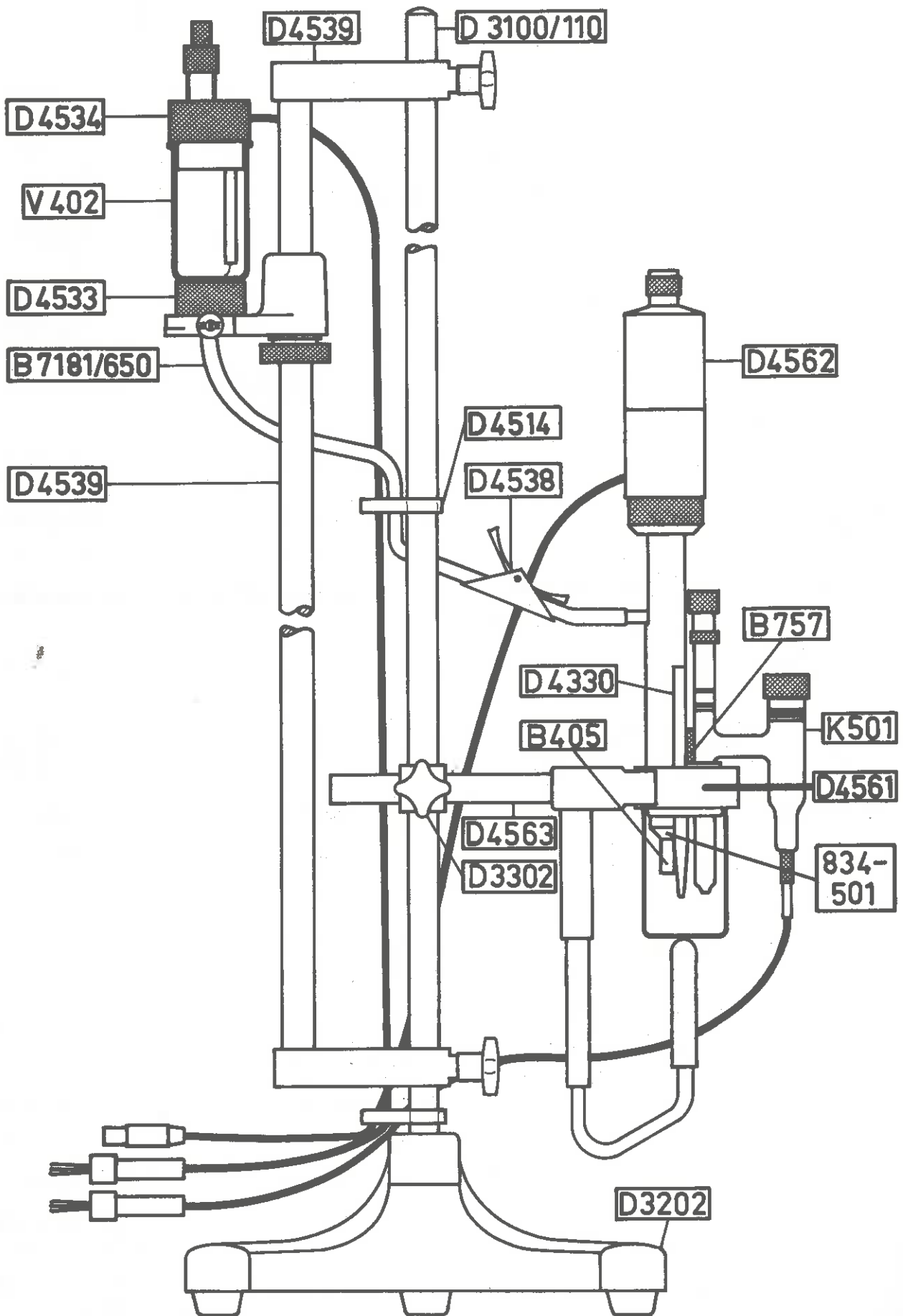


Fig. 7. Drawing of the Electrode Assembly, type E65.

on the reservoir V402. The plastic tubing must cover the entire length of the glass connecting branch.

- (2) Run the free end of the plastic tubing through the hole in the black seat of the reservoir holder D4533.
- (3) Press the reservoir V402 into the reservoir holder.
- (4) Place the Hg-valve, D4538, on the plastic tubing, and after that the fastener, code 846-005.
- (5) Mount the rubber seal cone (code 834-501) on the underside of the electrode head so that it will encircle the tip of the Hg capillary.
- (6) Place the spring (code 876-600) inside the steel cylinder of the electrode head.
- (7) Attach the plastic tubing from the mercury reservoir to the short branch of the Hg capillary and tighten the fastener to prevent the end of the tubing from sliding off the capillary.
- (8) Slip the Hg capillary through the electrode head and the spring so that its tip protrudes from the rubber seal ring.
- (9) Screw the hammer D4562 onto the steel cylinder of the electrode head.
- (10) Mount a vessel, V518, below the electrode head.

3. TO FILL THE SYSTEM

- (1) Fill the reservoir $\frac{3}{4}$ with mercury (distilled twice).
- (2) Move the plastic tubing a little so that the tubing is filled completely (the Hg-valve must be open). Now mercury will drop from the capillary.
- (3) Close the Hg-valve (the mercury drops will go on falling for a short time).
- (4) Remove the small stopper from the stopper with the platinum electrode, D4534.
This is very important.
- (5) Grease the O-ring of the stopper carrying the platinum electrode.
- (6) Mount this unit on top of the reservoir. (If the small stopper has not been removed, mercury will be pressed up through a small hole in the unit).
- (7) Now screw the syringe on top of the unit downwards as far as possible.
- (8) Replace the small stopper.
- (9) Screw the syringe upwards until an air bubble is seen coming through the mercury in the reservoir.

(This air bubble indicates that the constant pressure unit mounted in the reservoir is in operation).

At any movement of the reservoir or the reservoir holder the small stopper

must be removed and the operations mentioned in (7) - (9) above be repeated.

(10) Readjust the level of the electrode head so that the tip of the Hg capillary is on a level with the 0-mark of the scale.

(11) Connect the cable from the hammer to the HAMMER jack on the Drop Life Timer, type DLT1.

4. TO MOUNT THE CALOMEL ELECTRODE

- (1) Prepare a calomel electrode, K501, as mentioned in section E, page E2.
- (2) Place the salt bridge of the electrode in the large hole of the electrode head, D4561, with the calomel cell facing right.

5. TO MOUNT THE STOPPER, B757, AND THE GAS INLET TUBE, D4330

The stopper, B757, is generally placed in one of the small holes of the electrode head, D4561.

If an inert gas flow is required, proceed as follows:

- (1) Remove the stopper B757 to allow the gas to escape.
- (2) Place the gas inlet tube in one of the small holes in the electrode head.
- (3) Connect the gas inlet tube to a source with inert gas.

If a humidifier, D4530, is used, follow the instructions in section E, page E1.

Section D. Instructions for use

1. MEASURING

- (1) Mount the electrode setup as shown in the drawing (Fig. 7). Follow the above instructions.
- (2) Make sure that the mercury reservoir is filled with mercury and that it is ready for operation. (Screw the syringe on top of the reservoir a little upwards - an air bubble should pass through the mercury).
- (3) Make sure that the calomel electrode is ready for operation (completely filled with saturated KCl solution) and renew the liquid junction in the salt bridge by pressing a few drops of KCl solution out of it.
- (4) Pour about 20 ml of the solution to be analyzed into the vessel, V518, and place it below the electrode head.
- (5) Bubble with nitrogen gas for 10-15 minutes, if the sample is to be deoxygenated.

Note: If the humidifier, D4530, is used, fill its container with the same electrolyte solution as that used for the sample and adjust the gas flow by rotating the black nut on top of the humidifier.

- (6) After bubbling, raise the gas inlet tube so that the gas keeps flowing over the surface of the sample.
- (7) Connect the lead from the calomel electrode to the binding post CAL-OMEL ELECTRODE of the type PO4 Polariter.
- (8) Connect the lead from the mercury reservoir to the binding post DROPPING Hg ELECTRODE of the type PO4 Polariter.
- (9) Open the Hg valve
- (10) Make sure that the mercury drops adequately from the Hg capillary.

- (11) Record the polarogram.

2 AFTER USE

- (1) Remove the sample
- (2) Press a few drops of KCl solution out of the calomel electrode.
- (3) Rinse the ends of the capillary, the salt bridge, and the inlet tube with distilled water, to remove any trace of the sample.
- (4) Wipe off droplets of water on the Hg-capillary with a piece of filter paper.
- (5) Mount an empty vessel in place of the sample.
- (6) Close Hg-valve. After a short time the mercury will stop dropping from the capillary.

3 MAINTENANCE AND HANDLING

- (1) Always keep the calomel electrode filled with saturated KCl solution (crystals of KCl visible).
- (2) Never move the reservoir or reservoir holder downwards without removing the small stopper at the top of the reservoir.
- (3) Place the reservoir a little above the "0" mark of the rule when the ELECTRODE ASSEMBLY is not in use at night or for protracted periods.
- (4) Carefully remove all traces of spilled mercury. (Bear in mind that mercury is poisonous, and inhalation of

mercury vapours - even at room temperature - may lead to poisoning).

Large drops can be picked up with 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.

- (5) The bore of the Hg capillaries, type B405, is siliconized, but the silicone layer will last for a limited time only when the capillaries are used in a strong alkaline electrolyte and in some organic solvents.

Procedure for Resiliconizing the Capillaries

The B405 capillaries can be resiliconized in the following manner:

- (a) Draw a 2% solution of hydrofluoric acid through the capillary for 10 seconds by applying suction.
- (b) Draw half-concentrated hydrochloric acid through the capillary for about 10 seconds.
- (c) Draw distilled water through for about 10 seconds.

- (d) Draw air through the capillary until all traces of water are removed (about 1/2 minute).
- (e) Now draw a siliconizing fluid (2% solution of dimethyldichlorosilane in carbon tetrachloride) through the capillary for 5 times approx. 2 seconds, briefly sucking air into the capillary between operations.
- (f) Finally draw air through the capillary until the bore appears to be quite dry.
- (g) Afterwards dry the capillary at 150°C for at least half an hour.
- (h) Attach the capillary to the tube leading from the mercury reservoir V402, and allow mercury to drip from the capillary tip.
- (i) With mercury still dripping from the capillary, immerse the tip for approx. 10 seconds in a 2% solution of hydrofluoric acid.
- (j) Next - with mercury still dripping - immerse the tip for approx. 10 seconds in half-concentrated hydrochloric acid.
- (k) Swill the capillary tip with distilled water.
- (l) Check the flow of mercury.

Irregular Formation of Droplets

The formation of irregular droplets can be

caused either by a defective Hg capillary or by a faultily adjusted hammer mechanism.

A defective capillary is as a rule revealed by frequent and unreproducible jumps in the registered polarographic current, although their occurrence also may be attributable to many other causes. On the other hand, defects in the hammer mechanism proper are usually revealed in two ways:

- (a) Irregular formation of droplets in the neighbourhood of the electro-capillary maximum (approx. -560 mV in $\text{NH}_3 \text{ NH}_4\text{Cl}$ buffer) is most frequently the result of incorrectly adjusted impact force. The screw at the top of the hammer is used for readjusting.
- (b) Periodical variations in the polarographic current. Provided that the SYNC-switch on the Drop Life Timer, DLT1, is not set to position LINE, the cause of such may be interference between the period length of the line frequency and the drop life. However, if these are synchronized, the cause must be sought for in a periodical displacement of the movable parts in the electrode head — i.e. either the Hg capillary itself or the spring.

Section E. Specific instructions

1 HOW TO MOUNT AND USE THE HUMIDIFIER, D4530

- (1) Mount the humidifier at the free end of the support rod, D4563.
- (2) Connect the silicone rubber tubing from the gas outlet tube of the humidifier to the gas inlet tube, D4330, in the electrode head, D4561.
- (3) Connect the free end of silicone rubber tubing from the gas flow regulator to a source of inert gas.
- (4) Half fill the vessel in the humidifier with the electrolyte solution used for the sample.
- (5) Adjust the gas flow to a suitable speed by rotating the black nut on top of the humidifier.

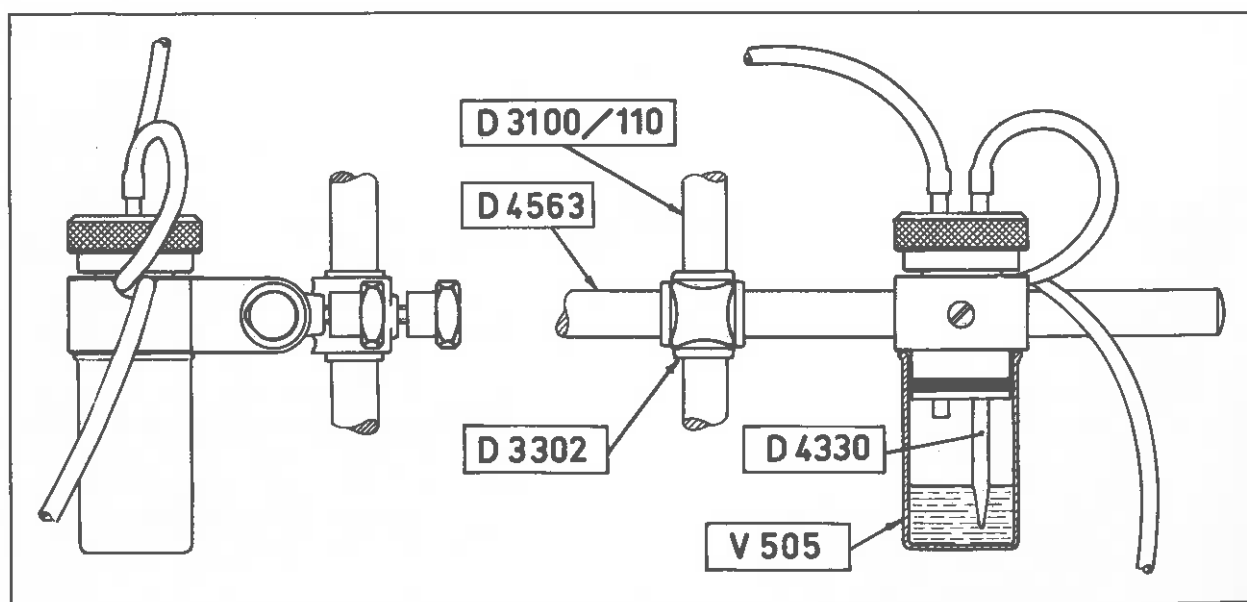


Fig.8. Humidifier, type D4530.

2 HOW TO MOUNT THE CALOMEL ELECTRODE, K501

- (1) Dismount the perspex container by unscrewing the perspex screw, and place some mercury in the well - enough to cover the platinum wire so that the mercury surface stands approximately 1 mm over the edge of the lower part of the container and fills the cross section of the wide part of the container.
- (2) Pour approximately a 1 mm thick layer of calomel paste on the mercury in the container and add a few drops of saturated KCl solution to form a smooth layer of calomel on the mercury surface.
- (3) Place a plate of filter paper B781/10 on the calomel layer and soak it with saturated potassium chloride solution.
- (4) Place the 3-bore perspex cone on the filter plate with the base in contact with it.
- (5) Fix the elements mounted in the calomel container by means of the 3-bore screw.

Note: Do not tighten the screw so much that the mercury and the calomel paste are pressed up along the sides of the container, but be sure that no air bubbles are present in the small bores.

- (6) Place the mounted container in the wide branch of the H-shaped glass part, and press it into position so that the 3 mm screw in the bottom protrudes from the narrow end of the glass part.
- (7) Mount the electrode cable on the protruding threaded pin with a pertinax ring between the glass and the nut of the electrode cable.

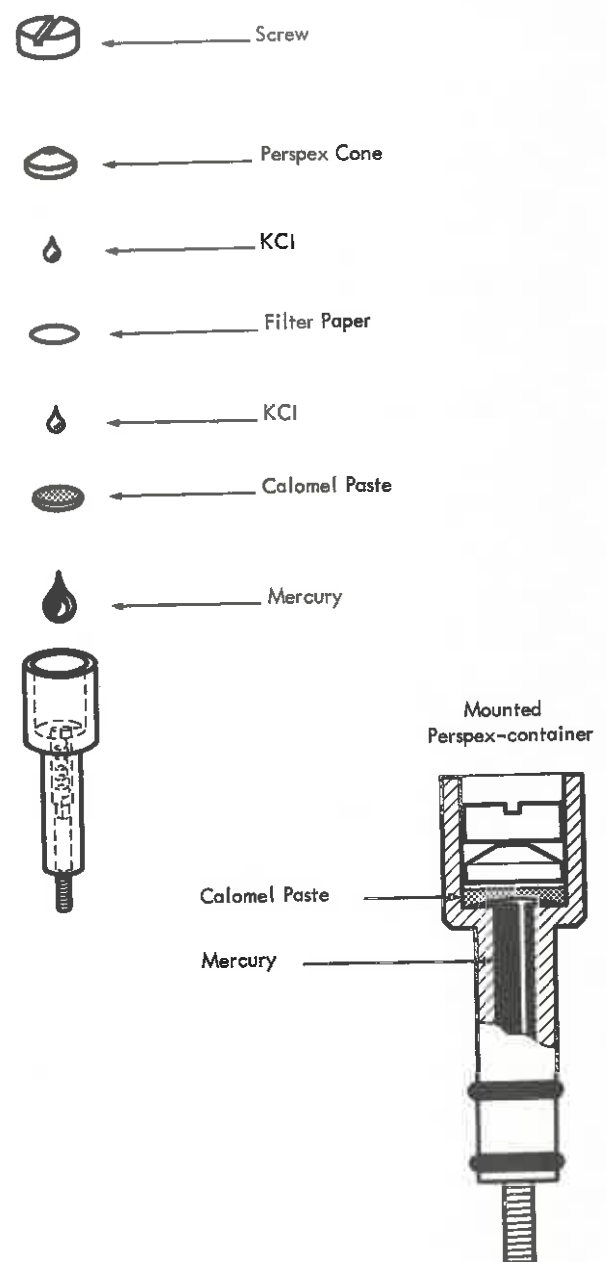


Fig.9. Preparing the Perspex Container of the K501, Calomel Electrode.

- (8) Fill the cell with saturated KCl solution.
- (9) Mount the big plastic stopper on the wide glass tube.
- (10) Tilt the calomel cell so that no air will be present in the side with the wide branch.
- (11) Screw off the cap of the small stopper.
- (12) Mount the plunger of the small stopper in the salt bridge side of the electrode and press till all the air has escaped.
- (13) Screw on the cap of the small stopper.
- (14) Press a few drops of KCl solution through the liquid-junction filter using the small stopper as a plunger.
- (15) Mount the electrode prepared in this way in the electrode assembly.

Refilling the calomel electrode

- (1) Screw off the cap of the small stopper.
- (2) Pull up the small plunger.
- (3) Refill with saturated KCl solution as much as required.
- (4) Follow the instructions above from (12) to (15).

Maintenance of the salt bridge

The saturated solution of potassium chloride in the salt bridge is separated from the sample in the vessel by some discs of filter paper. When changing to a new sample, expel a drop of the saturated solution of KCl through the filter at the tip:

- (1) Release the thumb nut, which acts as a stop for the plunger of the small stopper, half a turn.
- (2) Press the plunger down to the new stop.

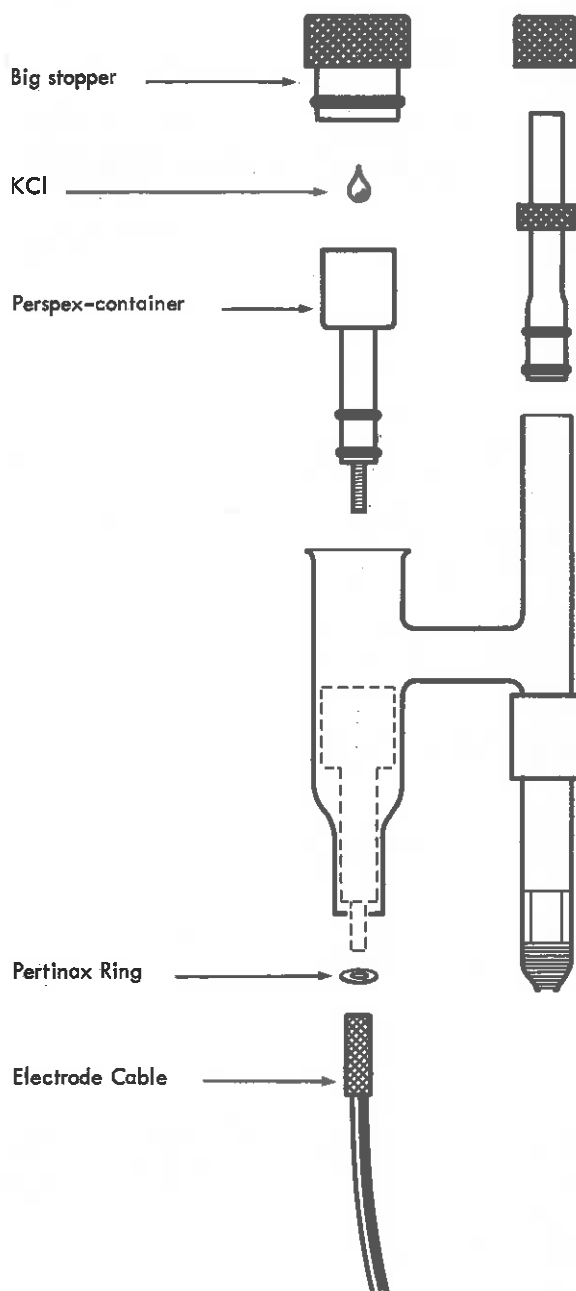


Fig.10. Preparing the Electrode, type K501

Note: If the filter paper at the tip of the salt bridge has been contaminated or otherwise damaged, it must be replaced:

- (1) Empty the salt bridge of the solution of potassium chloride.
- (2) Pour enough acetone into the branch with the filter in the tip to cover the piece of plastic tubing that keeps the filter in position.

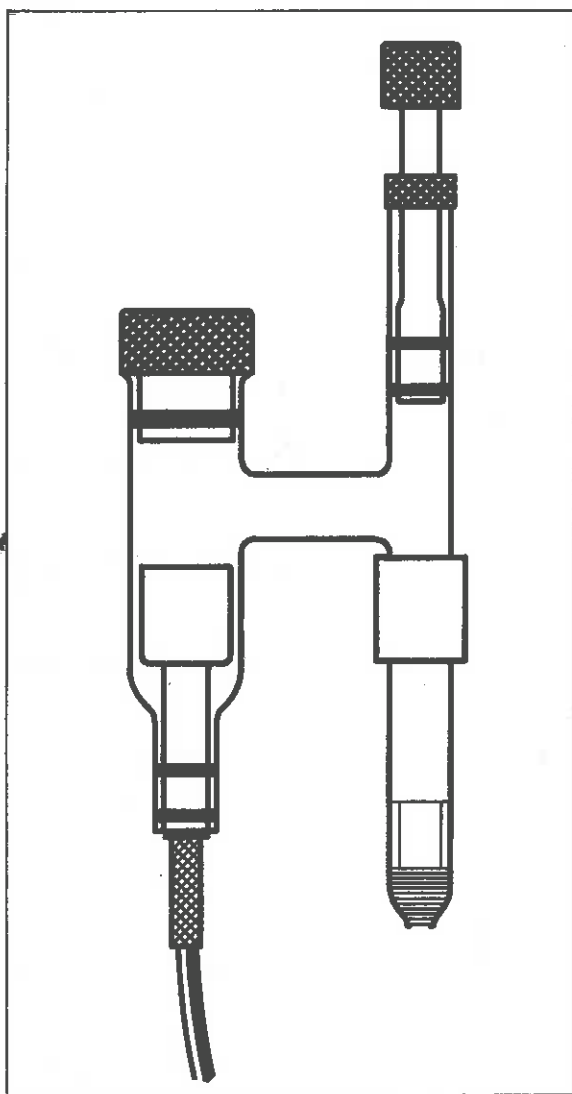


Fig.11. Mounted Electrode, type K501.

- (3) Now let the electrode stand overnight so that the piece of plastic tubing first can swell, the acetone then evaporate, and the piece of plastic tubing afterwards dry out again.
- (4) Push the plug of filter paper and the above piece of plastic tubing out of the lower end of the salt bridge.
- (5) Place a stack of six fresh discs of filter paper, type B781/9, in the tip of the salt bridge.
- (6) Press down another piece of plastic tubing. Use the tool, i.e. the plastic rod, to compress the filter paper in the tip of the salt bridge.
- (7) Refill the electrode with saturated solution of potassium chloride.
- (8) Complete the mounting of the electrode by following the instructions given in item 2 from (10) to (15).