

METROHM LTD. CH-9101 Herisau (Switzerland)

KF Coulometer

737

Series 01 ...

PARAM

Parameters

Go to next inquiry with <PARAM> and <ENTER>.
 ▽ means selection with <SELECT>.

Initial values are printed in **bold face**.

| Display | Meaning | Input range |
|-------------------------------------|---|---|
| ID.REQ▽ SMPL.REQ▽ CONF.START▽ | Request of identifications after start of titration. Request of sample size after start of titration. Additional start confirmation, e.g. after transfer of sample size from a connected balance. | ID1, ID1 2, ALL, OFF ON, OFF ON, OFF |
| D.START | Start drift as a threshold. Above this value, the titration can not be started. | 0... 98 ug/min |
| EXTR. | Extraction time. Positive: Iodine is generated during the extraction time. Negative: Waiting time before titration. | -63999... 0 ...63999 s |
| STOP DRIFT▽ DRIFT DELAY TIME | Selection of type of stop drift. With "AUTO", the drift value at the beginning of the titration is valid. Drift value for manual drift stop. Titration stops if the signal stays below the endpoint voltage during the delay time. | AUTO , MAN 0... 20 ...98 ug/min 0... 3 ...99 s |
| REPORT▽ | Type of report output at the end of the titration. | FULL, SHORT, OFF |

**CALC
DATA**

Calculations

Go to next inquiry with <CALC DATA> and <ENTER>.
 ▽ means selection with <SELECT>.

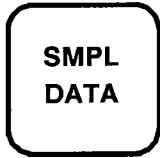
Initial values are printed in **bold face**.

| Display | Meaning | Input range |
|-----------------------------------|---|--|
| BLANK DRIFT CORR▽ DRIFT | Blank. Type of drift correction of the result. With "AUTO", the drift value at the beginning of the titration is valid. Drift value for manual drift correction. | 0 ...63999 ug AUTO , MAN 0 ...98 ug/min |

Calculation formula

$$\text{CONT-B} = \frac{\text{WATER} - \text{BLANK}}{\text{SMPL}} * F$$

F = 10³ for ppm
 10⁻¹ for %

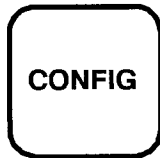


Sample Data

Go to next inquiry with
<SMPL DATA> and
<ENTER>.

Initial values are printed in
bold face.

| Display | Meaning | Input range |
|-------------------|---|-----------------------|
| SMPL | Sample size in mg. If sample size is = 0, content will not be calculated. | -63999...0...63999 mg |
| ID1 ID2 ID3 | } Sample identifications. | bis 8 ASCII Zeichen |



Configuration

Go to next inquiry with
<CONFIG> and
<ENTER>.
▽ means selection with <SELECT>.

Initial values are printed in
bold face.

| Display | Meaning | Input range |
|---------------------------|--|---|
| -- AUXILIARIES -- | Go to the inquiries of block "AUXILIARIES" with <ENTER>. | |
| MP.LIST▽ | Printout of a measuring point list "water vs. time" during the titration. | ON, OFF |
| TIME INTER. BALANCE▽ | Time interval for printout of measuring points. Selection of balance type. | 1...9999 s SARTORI, METTLER, AT- METT, AND, PRECISA |
| PERIPH.▽ BEEP LABEL | Peripheral instruments, at socket "Remote" Beeper. Device label to identify instrument for remote control. | OVEN , SAMPLER ON, OFF bis 8 ASCII Zeichen |
| PROGRAM | Program version. | keine Eingabe |
| -- RS SETTINGS -- | Go to the inquiries of block "RS SETTINGS" with <ENTER>. | |
| BAUD RATE▽ | Baud rate. | 9600 , 4800, 2400, 1200, 600, 300 |
| DATA BIT▽ | Data bit. | 7, 8 |
| STOP BIT▽ | Stop bit. | 1, 2 |
| PARITY▽ | Parity. | EVEN, ODD, NONE |
| HANDSHAKE▽ | Handshake. | HWS , HWF, SWLINE, SWCHAR, NONE |
| RS CONTROL▽ | Receiving commands via RS; "OFF" means no | ON , OFF |

737 KF Coulometer Instructions for use

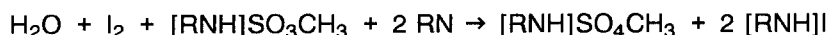
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1. Coulometric KF titration, what is it?

The coulometric Karl Fischer titration is a version of the classical water determination method developed by Karl Fischer. The traditional method utilises a methanolic solution of iodine, sulphur dioxide and a base as buffer. Several reactions run in the titration of a water-containing sample and can be summarised by the following overall equation:



According to the above equation, I_2 reacts quantitatively with H_2O . This chemical relation forms the basis of the water determination.

The classical Karl Fischer method has undergone constant development in the past years. This further development has involved not only refinement and automation of the reagent dispensing, but also improvement of the end point indication and the reagents. Despite the progress made, the classical, volumetric Karl Fischer method suffers from the disadvantage that the reagents are not completely stable resulting in the need to redetermine the titer at intervals.

In the coulometric Karl Fischer titration, the iodine needed is generated directly in the electrolyte by electrochemical means ("electronic burette"). The rigorously quantitative relation between the electric charge and the amount of iodine generated is used for highly precise dispensing of the iodine. As the **coulometric Karl Fischer method** is an absolute determination, **no titer need be determined**. It is necessary only to ensure that the reaction which generates the iodine runs with 100% current efficiency. With the reagents available today, this is always the case.

The end point is indicated voltametrically by applying an alternating current of constant strength to a double Pt electrode. This results in a voltage difference between the Pt wires of the indicator electrode which is drastically lowered in the presence of minimal quantities of free iodine. This fact is used to determine the end point of the titration.

The following books provide an excellent overview of the KF titrations:

- E. Scholz, Karl Fischer Titration, Springer-Verlag, Berlin, 1984.
- G. Wieland, Karl Fischer Titration, GIT Verlag, Darmstadt, 1985

The 737 KF Coulometer is an automatic instrument for the exact determination of small amounts of water in the range 10 μg ... 10 mg. In the normal case, the default parameters need not be changed so that the instrument is immediately ready for measurement after setting up the measuring cell.

2. Handling the measuring cell

2.1 Measuring cell without diaphragm

2.1.1 Setting up the measuring cell

1. Fasten titration cell in holder to the stand rod with the clamping ring.
 2. Place stirring bar in titration cell.
 3. Cut ground joint sleeves to correct lengths at the narrower end and fit to all ground-glass joints.
 4. Insert indicator electrode (5) in titration cell.
 5. Screw electrode cable with the smaller plug (6.2104.020) to the indicator electrode and attach to the "Ind.El." socket at the rear of the KF Coulometer.
 6. Insert generator electrode (2) in titration cell.
 7. Fill drying tube (9) with molecular sieves and insert in the generator electrode.
 8. Screw electrode cable with the larger plug (6.2104.120) to the generator electrode and attach to the "Gen.El." socket at the rear of the of the KF Coulometer.
- Warning:** The two electrode cables must not be mixed up! Mark the cables and the corresponding electrode socket to avoid errors.
9. Insert a septum in screw cap (8) and screw this onto the glass fitting.
 10. Fill cell with reagent up to the bottom edge of the article number (ca. 80-90 mL).
 11. Close the last ground-glass opening with ground-glass stopper (7) or continue with point 12.

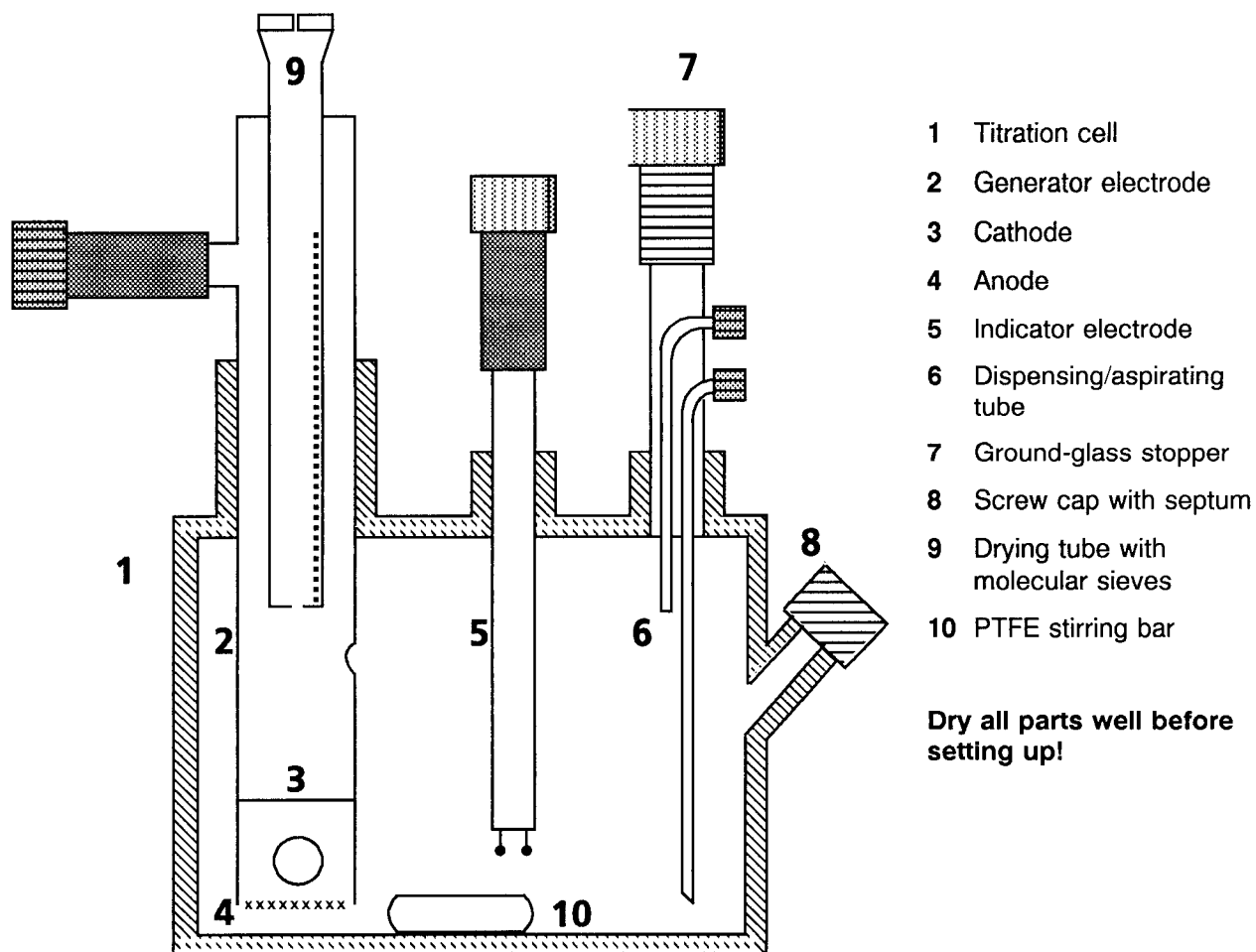


Fig. 2-1: Assembly of the measuring cell without diaphragm

Setting up the measuring cell together with the 703 Ti Stand

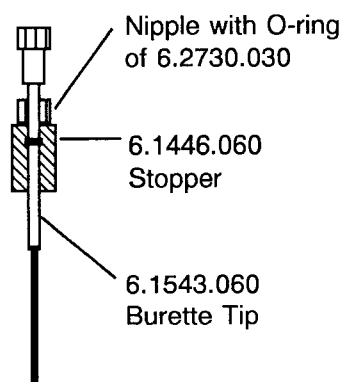
The dispensing/aspirating tube (6) is used in place of the ground-glass stopper (7). Proceed as follows:

12. Mount ground-glass stopper (7) on dispensing/aspirating tube (6) and insert this in the titration cell.
13. Screw on aspirating and dispensing tubing. (The aspirating tubing is attached to the long tube, bottom connection.)
14. If the pump is temporarily not connected, the two stoppers with thread M8 (6.1446.080) are used to close the tubing fittings of the dispensing/aspirating tube.

If a drop remains in the tube following addition of electrolyte solution, this can be expelled by briefly pressing the dispensing button of the pump several times.

If the dispensing/aspirating tube is temporarily not inserted in the measuring cell, it can be stored in the 6.1228.000 Holder.

Gas supply to the measuring cell, e.g. for work with KF Oven



1. Screw nipple with O-ring of 6.2730.030 into 6.1446.060 Stopper.
2. Insert 6.1543.060 Burette Tip from the accessories of the oven through the nipple
3. Screw 6.1805.070 Tubing to the burette tip and connect to outlet of oven.
4. Insert in the measuring cell in place of the dispensing/aspirating tube (6) or the ground-glass stopper (7).

Fig. 2-2: Gas supply, e.g. for work with the KF Oven

2.1.2 Cleaning the measuring cell

The electrolyte solution can usually be changed without the cell having to be cleaned. Cleaning is necessary only when the cell is highly contaminated. The platinum gauze of the generator electrode must not be damaged during cleaning.

Oil-containing contamination: Clean with solvent (e.g. hexane), then rinse with ethanol.

Salt-like deposits: Clean with water, then rinse with ethanol.

Generator electrode has black spots on Pt gauze: Clean with conc. nitric acid and with water, then rinse with ethanol. The electrolyte solution should be changed more frequently.

After cleaning, dry all parts thoroughly. A hair dryer can be used for this. If the parts need to be dried in a drying oven, ensure the temperature is not greater than 70°C (plastic parts!).

2.1.3 Reagents

Use reagents which are specially intended for use with a measuring cell without diaphragm.

For the water determination in ketones and aldehydes, to date no special reagents are on the market for measuring cells without diaphragm. However, the anolyte of the ketone reagent for cells with diaphragm can frequently be used. This reagent should be changed every week.

2.2 Measuring cell with diaphragm

2.2.1 Setting up the measuring cell

Tighten screw fittings firmly, but without using force.

1. Place blue sealing ring (from 6.1454.000 Sealing Set; ordering number for separate order 6.1454.030) on underside of titration cell top half.
2. Fasten locking screw and titration cell top half to stand rod.
3. Place stirring bar in titration vessel, fit the retaining ring to the vessel. Press top and bottom halves together powerfully with the clamp.
4. Roll O-ring carefully over the platinum gauze at the bottom end of the generator electrode. Ensure compliance with the distance of 1-2 mm between the diaphragm and the Pt gauze.
Care: Handle platinum gauze only when necessary and then proceed with extreme caution as the gauze can break off.
5. Screw in generator electrode (3). The supply cable should point outwards. Plug electrode cable into the "Gen.El." socket at the rear of the KF Coulometer.
6. Insert indicator electrode (4) in opening.
Screw on electrode cable (6.2104.020) and plug into the "Ind.El" socket at the rear of the KF Coulometer.
7. Add anolyte solution up to the lower edge of the article number.
8. Close the two remaining openings with a screw nipple and septum (5).
9. Add catholyte to the interior of the generator electrode (funnel) until the level is ca. 2...3 mm below the anolyte (ca. 5 mL). Siphon off any excess catholyte, e.g. with a pipette.
10. Fill drying tube with molecular sieves, fit it with a ground joint sleeve (cut joint sleeves to correct lengths at narrower end) and insert in the generator electrode.

Gas supply to measuring cell, e.g. for work with the KF oven

Use 6.1617.010 Gas Inlet Tube. Check that the O-ring is fitted to the gas inlet tube.

If temporarily not in use, close gas inlet tube with 6.1446.040 Dummy Stopper.

2.2.2 Cleaning the measuring cell

The electrolyte solution can usually be changed without the cell having to be cleaned. Cleaning is necessary only when the cell is highly contaminated. The platinum gauze of the generator electrode must not be damaged during cleaning.

If resinous deposits are observed on the diaphragm of the generator electrode, they can be removed with conc. HNO_3 : Suspend generator vertically from a stand rod, fill with conc. HNO_3 and allow to stand overnight. Rinse with dist. water, then with ethanol.

Oil-containing contamination: Clean with solvent (e.g. hexane), then rinse with ethanol.

Salt-like deposits: Clean with water, then rinse with ethanol.

After cleaning, dry all parts thoroughly. A hair dryer can be used for this. If the parts need to be dried in a drying oven, ensure the temperature is not greater than 70°C (plastic parts!).

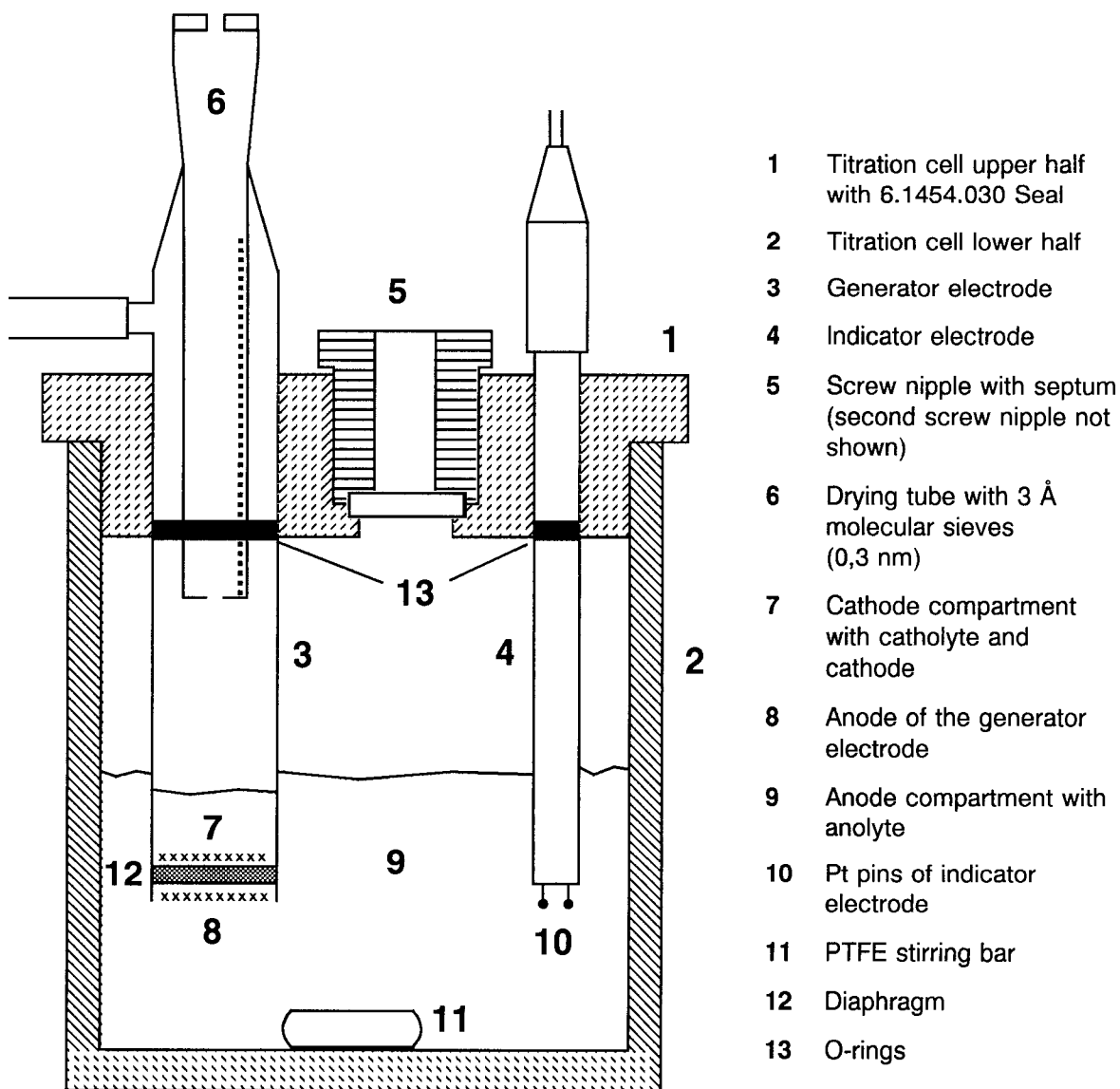


Fig. 2-3: Assembly of the measuring cell with diaphragm

2.2.3 Reagents

Reagents for the coulometric water determination with measuring cells with diaphragm comprise the anode solution (anolyte), added to the measuring cell (anode compartment), and the cathode solution (catholyte), which is added to the generator electrode (cathode compartment).

For the determination of water in aldehydes and ketones, special reagents are used, see documentation of the reagent producers. These reagents (anolyte and catholyte) should be changed every week.

2.3 Sample addition

General remarks

In what follows, you will find several tips for sample addition. However, a complete treatment of all problems in connection with the sample addition is not possible. A selection of METROHM Application Bulletins (available free of charge) is given below:

- No. 77: Karl Fischer water determinations
- No. 88: Bibliography concerning Karl Fischer water determinations
- No. 109: Karl Fischer water determinations with the KF Drying Oven
- No. 137: Karl Fischer water determinations with the KF Coulometer
- No. 142: Karl Fischer moisture determination in gases
- No. 145: The determination of low water contents in plastics
- No. 209: Coulometric water determinations following Karl Fischer in insulating oils, hydrocarbons and their products
- No. 236: Coulometric water determinations in brake fluids by the Karl Fischer method with automatic sample metering

2.3.1 Liquid samples

Syringes are used for the manual addition of liquid samples.

For automatic sample metering and addition, the 708 Sampling Unit can be used, see page 56.

A low sample volume should be selected to allow titration of the maximum number of samples in the same electrolyte solution. However, the amount of sample should also be kept as small as possible to shorten the analysis time. The amount of sample needed to ensure the result of the determination lies in the desired range can be seen from Fig. 2-4.

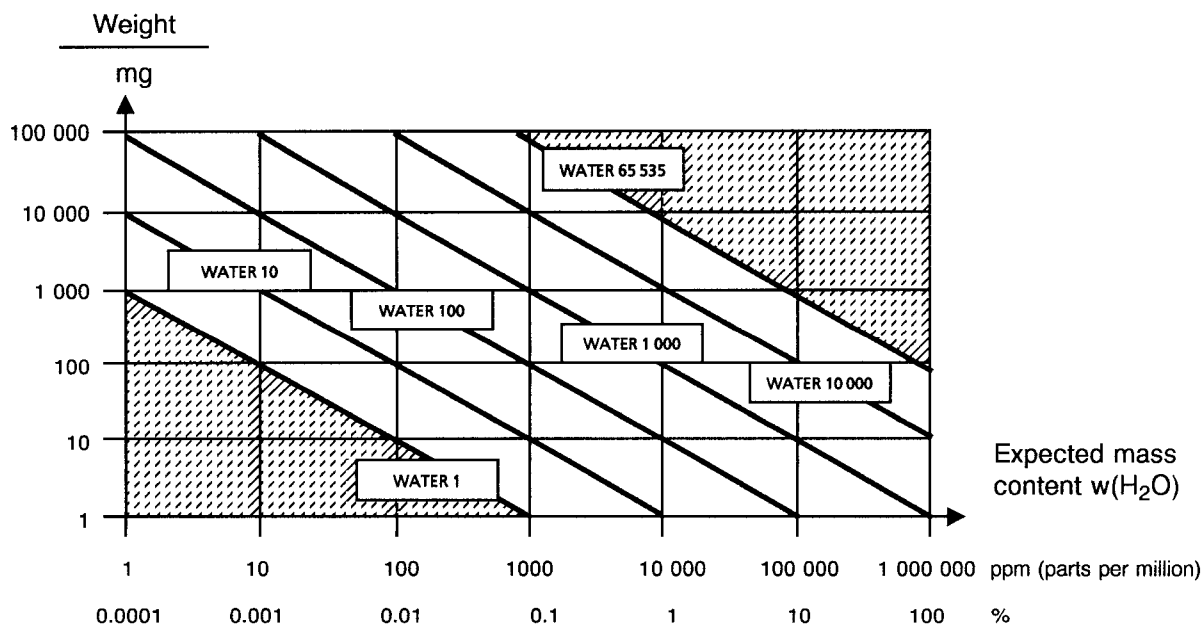


Fig. 2-4: Guide values for the sample size

Highly volatile or low-viscosity samples should be cooled before sampling to avoid losses during handling. On the other hand, the syringe must not be cooled directly as water of condensation can form. For the same reason, the entrainment of air into a syringe cooled by siphoning off an aliquot of cooled sample must be avoided.

Highly viscous samples can be thinned by warming, but the tip of the syringe must also be warmed. The same goal can also be achieved by dilution with a suitable solvent. In this case, the water content of the solvent must be determined and subtracted as a blank value.

Pastes and fats can be added to the measuring cell using a syringe without needle.

With **samples containing large amounts of water**, ensure that the needle is not inserted through the septum into the measuring cell before <START> is pressed as otherwise the drift and hence the result of the analysis could be falsified.

With **samples which contain only trace water**, it is essential to predry the syringe thoroughly. If possible, the tip should be rinsed with the sample solution by siphoning off and discarding solution several times.

2.3.2 Solid samples

If possible, solid samples are extracted with or dissolved in a suitable solvent and the resulting solution injected. A blank value correction for the solvent is necessary.

If no suitable solvent for a solid sample can be found or if the sample reacts with the Karl Fischer reagent, the drying oven should be used.

If solid samples have to be introduced directly, use the cell without diaphragm. Take care that

- the sample releases its humidity completely
- there is no side reaction of the sample with the KF solution
- the surface of the electrodes is not getting coated with sample in the course of your determinations (uncomplete KF reaction!)
- the Pt mesh of the generator electrode and the Pt wires of the indicator electrode are not damaged

2.4 Optimum working conditions

Drift

A constant drift in the range 4 ... 12 $\mu\text{g}/\text{min}$ is good. Lower values are, however, entirely possible. When work is started with a measuring cell which has been well predried, the background drift is attained within a few minutes. Measuring cells without diaphragm are usually in operational readiness quicker than measuring cells with diaphragm.

For precise determinations of water quantities less than 10 μg , it can be an advantage to condition the instrument overnight before use.

A permanently high drift can be caused by water-containing deposits at inaccessible positions in the cell. In such cases, shaking the measuring cell lowers the value. With measuring cells with diaphragm, never shake so hard that the catholyte and anolyte mix. If the drift remains high for a lengthy period after shaking the cell, the electrolyte solution should be changed.

If a filled measuring cell remains unconditioned for a relatively long time, it takes a certain time until the cell is dry. This time can be shortened by shaking the cell once or twice. It is advisable to precondition the measuring cell overnight after lengthy breaks in operation. When the instrument is in continuous use, it should not be switched off overnight.

If the drift is frequently negative, the end point voltage should be set lower with the potentiometer "EP voltage" at the rear of the Coulometer. If, however, the end point voltage is too low, the titration times increase.

Drift fluctuations when attached devices (printer, balance, PC) are switched on and off indicate earth loops. In this case, these devices should be attached to the Coulometer via an optocoupler.

Changing the electrolyte solution

The electrolyte solutions must be changed in the following cases:

- 1) If the titration cell can hold no more samples.
- 2) If the error message `/GEN_ELECTRODE /` appears during the determinations (see page 36). In the case of the measuring cell with diaphragm, it is often sufficient to change just the cathode solution.
- 3) If the drift is too high and no improvement can be achieved by shaking the cell (see above).
- 4) If a two-phase mixture forms in the titration cell.
- 5) Change the special reagents for the water determination in aldehydes and ketones every week.

The simplest way to remove exhausted electrolyte solutions from the cell is to siphon them off. The cell need not then be disassembled.

With severe contamination, the cell can be rinsed with a suitable solvent, which is also siphoned off.

To siphon off the electrolyte solutions, the siphoning device available as an option can be used, see page 55.

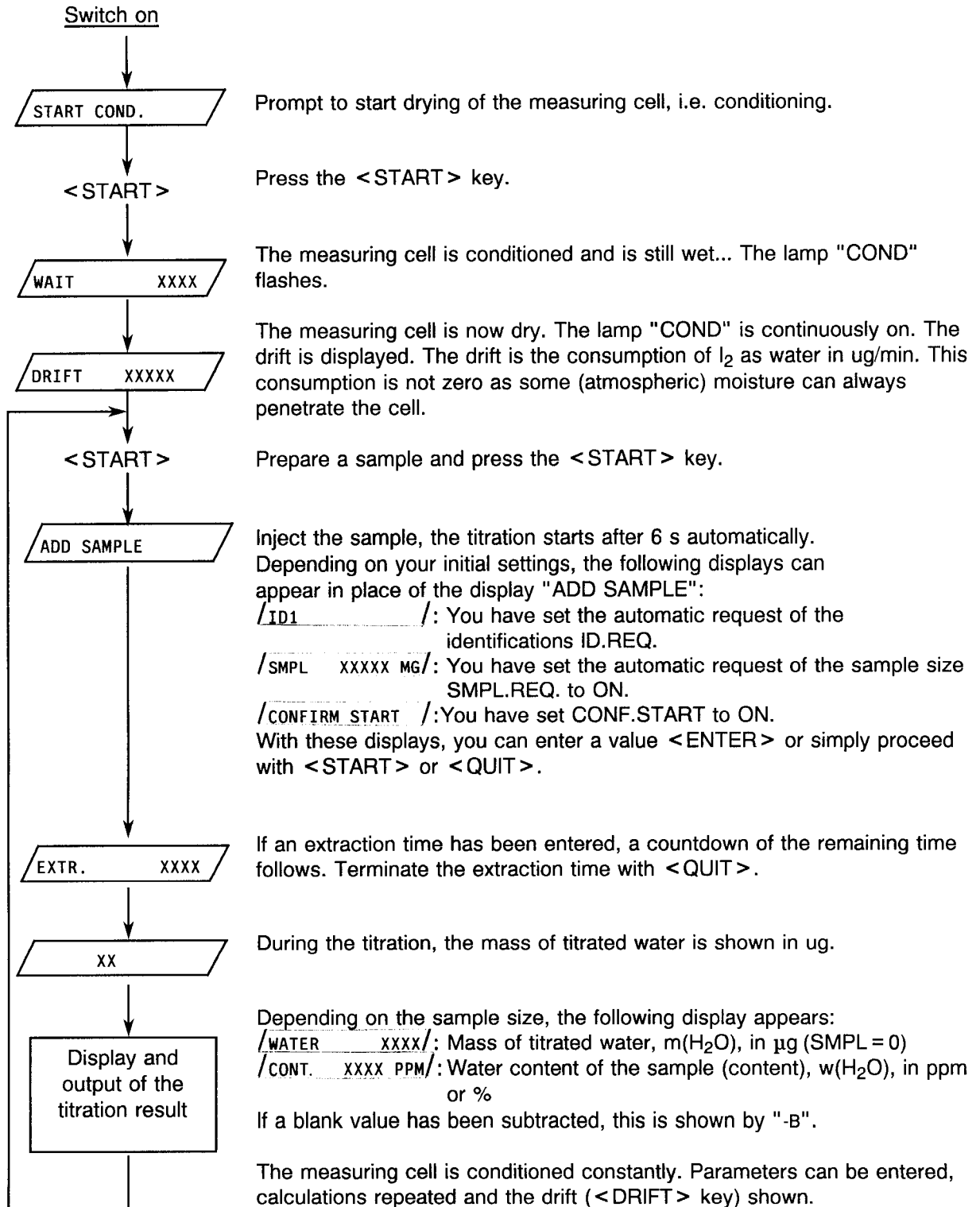
Indicator electrode

A new indicator electrode may need a certain conditioning time for formation of the surface. This can lead to unusually long titration times and to high measurement results. However, these phenomena disappear after a short period of use. To promote the conditioning of a new indicator electrode, leave the electrode immersed in the electrolyte solution with the instrument switched off, e.g. overnight. In obstinate cases, the indicator electrode can be carefully cleaned with a scouring agent and then rinsed with ethanol.

The two Pt wires of the indicator electrode must be as parallel as possible. Check when installing.

3. Operation

3.1 Titration sequence



The <STOP> key aborts the titration and conditioning and leads to the inactive standby mode.

3.2 Calculations, sample data

The titration results are calculated by the following formula:

$$\text{CONT} - \text{B} = \frac{m(\text{H}_2\text{O}) - \text{blank}}{|\text{sample}|} \cdot f$$

- m(H₂O) Mass of the water found in µg, corrected by the drift
 - blank Mass of the water in the blank value in µg
 - sample Sample size in mg. The absolute value is used for the calculation.
 - f Factor
 - f = 10³ if the result is outputted in ppm
 - f = 10⁻¹ if the result is outputted in %
- Switching between ppm and % is automatic.

3.2.1 <CALC DATA> key

The <CALC DATA> key contains data for the result calculation. Enter values and switch to next inquiry with <ENTER>. ▽ following the dialogue text means that the values are selected with <SELECT>. <QUIT> ends the inquiry sequence. The displays show the default values.

| | |
|--|--|
| <div style="border: 1px solid black; padding: 5px; display: inline-block;">BLANK 0 ug</div> | <p><i>Blank value (0...63 999 ug, 5 significant digits, up to 1 decimal place)</i></p> <p>If a blank value is set ≠ 0, it will be used for the result calculation. If a blank value has been subtracted, this is noted by "-B".</p> |
| <div style="border: 1px solid black; padding: 5px; display: inline-block;">DRIFT CORR ▽ AUTO</div> | <p><i>Type of drift value transfer for the drift correction (AUTO, MAN)</i></p> <p>The mass of the water m(H₂O) is continuously corrected by the drift during the titration.</p> <p>AUTO: The drift value is stored on start of the titration.</p> <p>MAN: The drift value is entered manually.</p> <p>With "MAN", the following inquiry appears:</p> |
| <div style="border: 1px solid black; padding: 5px; display: inline-block;">DRIFT 0 UG/MIN</div> | <p><i>Drift value for manual drift correction (0...98 ug/min)</i></p> <p>0 means no drift correction. Entry in steps of 2 ug/min.</p> |

3.2.2 <SMPL DATA> key

The <SMPL DATA> key contains sample data. Enter values and switch to next inquiry with <ENTER>. ▽ following the dialogue text means that the values are selected with <SELECT>. <QUIT> ends the inquiry sequence. The displays show the default values.

| | |
|--|--|
| <div style="border: 1px solid black; padding: 5px; display: inline-block;">SMPL 0 MG</div> <p style="text-align: center;">*five</p> | <p><i>Sample size (0... ± 63 999 mg, 5 significant digits, up to 1 decimal place)</i></p> <p>If the sample size is zero, the content (CONT.) is not calculated and the result is outputted in ug water.</p> <p>The absolute value of the sample size is used for the calculation. A negative sample size shows, e.g. a backweighing.</p> |
| <div style="border: 1px solid black; padding: 5px; display: inline-block;">ID1</div> <p style="text-align: center;">*five</p> | <p><i>Sample identification (up to 8 characters)</i></p> <p>Up to 3 sample identifications Id1...Id3 can be entered.</p> |

3.3 Parameters

3.3.1 <PARAM> key

The <PARAM> key contains the titration parameters.

Enter values and switch to next inquiry with <ENTER>. ▽ following the dialogue text means that the values are selected with <SELECT>. <QUIT> ends the inquiry sequence. The displays show the default values.

| | |
|----------------------------------|--|
| <p>ID.REQ▽ OFF</p> | <p><i>Request of sample identifications after the titration start (ID1, ID1 2, ALL, OFF)</i> After the start, the corresponding identifications are requested.</p> |
| <p>SMPL.REQ▽ OFF</p> | <p><i>Request of the sample size after the titration start (ON, OFF)</i> After the start, the sample size is requested.</p> |
| <p>CONF.START▽ OFF</p> | <p><i>Additional start confirmation (ON, OFF)</i> Additional hold in the titration sequence after entry of the sample size. Can be used, e.g. after a transfer from the balance or to allow an individual sample addition time.</p> |
| <p>D.START 98 UG/MIN</p> | <p><i>Drift threshold for the start of the titration (0...98 ug/min)</i> A titration start is not possible until the drift value is less than the set value. Entry in steps of 2 ug water/min.</p> |
| <p>EXTR. 0 S *five</p> | <p><i>Extraction time (0... ± 63 999 s)</i> Negative extraction time: Wait time during which no I₂ is generated. Positive extraction time: I₂ is generated, but the titration will not be terminated during elapse of the extraction time. Used, e.g. in work with the KF oven.</p> |
| <p>STOP DRIFT▽ AUTO</p> | <p><i>Type of drift value transfer for the stop drift (AUTO, MAN)</i> The stop drift is a necessary criterion for the automatic stop of the titration: The EP voltage and the stop drift must be reached. AUTO: The drift value is stored at the start of the titration. MAN: The drift value is entered manually.</p> |
| <p>DRIFT 20 UG/MIN *five</p> | <p><i>Drift value for the manual stop drift (0...98 ug/min)</i> Entry in steps of 2 ug water/min.</p> |
| <p>DELAY TIME 3 S *five</p> | <p><i>Switch-off delay time (0...99 s)</i> During the switch-off delay time, the indication signal must be in the EP voltage range and the value less than the stop drift for the titration to be ended.</p> |
| <p>REPORT▽ OFF</p> | <p><i>Selection of the automatic result output at the end of the titration (FULL, SHORT, OFF)</i></p> |

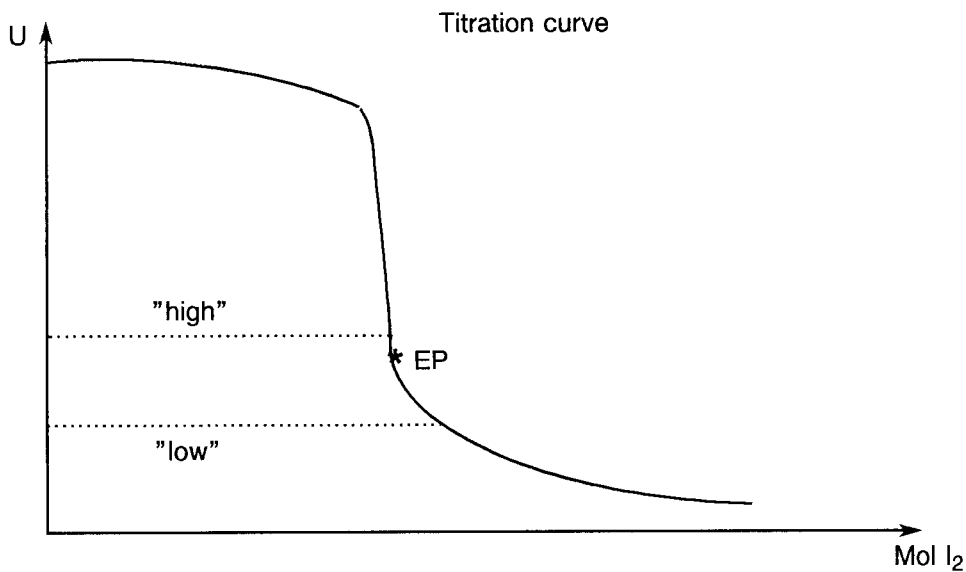
3.3.2 End point voltage

The preset end point voltage can be varied, e.g. with a screwdriver at the potentiometer at the rear of the KF Coulometer.



EP Voltage (high ... low)

Variable adjustment of the end point voltage between the extreme values "high" (high end point voltage, in other words EP lies in the steep region of the titration curve) and "low" (low electrode voltage, in other words EP lies in the flatter region) is possible.



The indicator electrode is activated after a certain period of use in the same reagent, i.e. the titration curve becomes steeper. If the titration curve is too steep, the titration controller can oscillate leading to slowly fluctuating drift values during the conditioning. Corrective action: Set EP voltage lower. End point voltages set too low can, however, prolong the titration and hence have an unfavourable influence on the measurement error.

3.4 Configuration

The <CONFIG> key contains the instrument configuration values. The inquiries are divided into two groups: —AUXILIARIES— and —RS SETTINGS—. The individual inquiries of the groups are reached with <ENTER>. Enter values and switch to next inquiry with <ENTER>. ▽ following the dialogue text means that the values are selected with <SELECT>. <QUIT> ends the inquiry sequence and leads to the title of the group. Pressing <QUIT> again ends the inquiries of the <CONFIG> key. The displays show the default values.

| AUXILIARIES | Inquires of the group AUXILIARIES |
|---------------------|---|
| MP.LIST ▽ OFF | <i>Printout of the list of measured points (ON, OFF)</i> List of measured points water/ug vs time/s during the titration |
| TIME INTER. 1 S | If set to "ON", inquiry: <i>Time interval for the output of the measured points (1...9999 s)</i> |
| BALANCE ▽ SARTORI | <i>Selection of the balance type (SARTORI, METTLER, AT-METT, AND, PRECISA)</i> Sartorius: Types MP8, MC1 Mettler: Types AM, PM and balances with interfaces 011, 012 and 016 Mettler AT: AT model AND: Types ER-60, 120, 180, 182, FR-200, 300 and FX-200, 300, 320 Precisa: Types with RS232C interface |
| PERIPH. ▽ OVEN | <i>Selection of the peripheral device at the "Remote" socket (OVEN, SAMPLER)</i> |
| BEEP ▽ ON | <i>Beep (ON, OFF)</i> Audio signal after the titration and when the KF Coulometer is ready for start after preconditioning. |
| LABEL | <i>Instrument designation for individual identification in the instrument network (up to 8 ASCII characters) for remote control via RS232.</i> |
| PROGRAM 737.0010 | <i>Display of the program version</i> |

| RS SETTINGS | Inquires of the group RS SETTINGS |
|--------------------|--|
| BAUD RATE ▾ 9600 | Baud rate (300, 600, 1200, 2400, 4800, 9600) |
| DATA BIT ▾ 8 | Data bits (7, 8) |
| STOP BIT ▾ 1 | Stop bits (1, 2) |
| PARITY ▾ NONE | Parity (EVEN, ODD, NONE) |
| HANDSHAKE ▾ HWS | Handshake (HWS, HWF, SWCHAR, SWLINE, NONE) see page 28 |
| RS CONTROL ▾ ON | Control via RS232 interface (ON, OFF) OFF means that the receipt of commands via the RS interface is blocked. Data <u>output</u> is always possible. Must be set to ON for data transfer from a balance. |

3.5 Display of the drift

The drift is displayed in ug water/min. It is a measure of the blank consumption of iodine, e.g. by ingress of atmospheric moisture or slow, side-reactions in the KF reagent itself or in the sample matrix which consume iodine.

The drift can be displayed during the conditioning:
Press the <DRIFT> key.

If you wish to show the titration result again, press <DRIFT> once more.

3.6 Data output

3.6.1 <PRINT> key

In addition to the reports printed out at the end of the titration, various other reports can be outputted. There are 2 possibilities to select the reports:

- 1) <PRINT> <SELECT> <ENTER> <SELECT> is pressed repeatedly until the desired report appears in the display.
- 2) <PRINT> <KeyX> <ENTER> Key X is the key under which the appropriate data are entered.

The following list provides an overview of the report possibilities:

| Report | Display with <PRINT> <SELECT> | <Key X> |
|----------------------|----------------------------------|-----------|
| Full result report | FULL | - |
| Short result report | SHORT | - |
| Configuration report | CONFIG | CONFIG |
| Parameter report | PARAM | PARAM |
| Calculation data | CALC DATA | CALC DATA |
| Sample data | SMPL DATA | SMPL DATA |
| All reports | ALL | - |

Example of a full result report:

```
'fr
737 KF Coulometer    0P1/108    737.0010
                    23

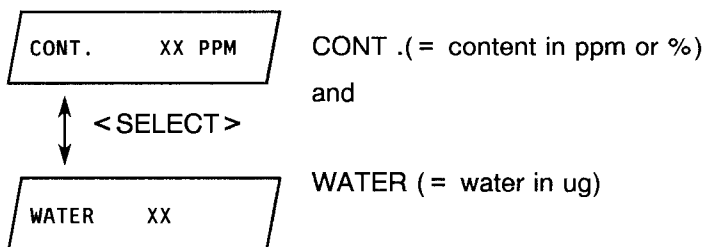
  smp1                32 mg
  id1                 94-05-23
  drift               8 ug/min
  titr.time           42 s
  water               237 ug
  content             0.7406 %
  =====
```

- identification of report type (fr = full report)
- instrument, serial number, program version
- current value of sample counter
- sample size
- sample identification
- drift value
- titr. time, i.e. time during which I₂ was generated
- titrated water
- calculated content
- double dashed line = original report.

Original reports printed out directly following the end of the titration have a double dashed line (=====), whereas recalculated reports are ended with a single dashed line (-----).

3.6.2 Switching the result display

The <SELECT> key can be used to switch the display between



4. Operation via RS232 interface

4.1 General rules

The Coulometer has an extensive remote control facility that allows full control of the titrator via the RS232 interface, i.e. the titrator can receive data from an external controller or it can send data to an external controller. The Coulometer sends $2 \times C_R$ and L_F as termination of a data block, to differentiate between a data line which has C_R and L_F as terminator. The controller terminates its commands with C_R and L_F . If the controller sends more than one command per line, the character ';' is used as separator between the commands.

The commands are grouped logically and are simple to understand. Thus, e.g. for the selection of the report the command

&Mode.Parameter.Report "FULL"

must be sent, but only the boldface characters need be inputted, thus

&M.P.R"FULL" .

All quantities of the Coulometer are collected in groups. For example, the entries for the configuration are in the group

&Config

The "Config" group contains subgroups, e.g. for the setting of the RS interface parameters (RS Settings)

&Config.RSSet

This subgroup in turn contains the individual inquiries for the settings, e.g. the inquiry regarding the baud rate

&Config.RSSet.Baud

or regarding the setting of the parity

&Config.RSSet.Parity

The commands have a hierarchial structure (tree structure). The quantities that appear in this tree are called **objects** in what follows. The baud rate is an object that is called up with the command

&Config.RSSet.Baud

If one is at the desired location in the tree, the value of the appropriate object can be queried, e.g. in the case of the inquiry regarding the baud rate:

&Config.RSSet.Baud \$Q Q for Query

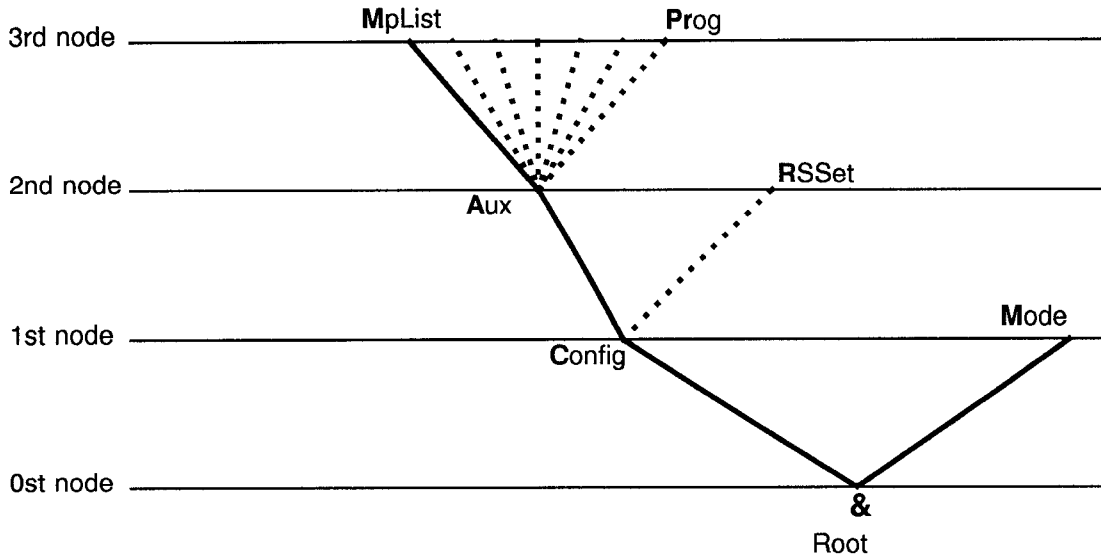
\$Q triggers the output of the value from the Coulometer. Entries that are introduced with the character dollar (\$) trigger something. They are thus called **triggers** in what follows.

Values of objects can not only be requested, however, they can also be modified. Values are always entered in quotation marks ("), e.g.

&Config.RSSet.Baud "9600"

4.1.1 Call up of objects

All objects of the Coulometer are grouped hierarchically. They have a tree structure. A section of this tree is shown below:



Rules

- The root of the tree is designated with &.
- For the call up of an object the nodes (levels) of the tree are marked with a point (.).
- The call up of the objects requires as many letters as necessary to ensure unequivocal assignment of the object. If the call is not unequivocal, the first object in the series is recognised.
- Upper- or lowercase letters can be used
- An object can be assigned a value. Each value is marked at the beginning and end with quotation marks ("). A value can contain up to 8 ASCII characters. Numeric values can include up to 5 digits, a negative sign and a decimal point. Numbers with more than 5 digits will not be accepted. With numbers < 1, it is necessary to enter leading zeros.
- If a new object is not called up, the old object remains current.

Examples:

Call up of the Beep:
&Config.Aux.Beep
or **&C.A.Be**

&C.A.BE or **&c.a.be**

Entry of "OFF" for the beep
&C.A.BE "OFF"

Correct entries of numbers:
"-31227"

"0.1"

incorrect entries:

"1,5" or **" +3"** or **".1"**

- New objects can also be addressed relative to old objects:
A preceding point moves one node **forwards** in the tree.
More than one preceding point moves one node **backwards** in the tree. n nodes backwards require n + 1 preceding points.
- If a jump is to be made back to the root, a preceding & is entered.

From the root to the node 'Aux':
&C.A
 Forwards from the node 'Aux' to 'MpList': **.M**
 Jump from 'MpList' onto the node 'Aux' and selection of a new object at this node 'Prog': **..Pr**
 Jump from the object 'Prog' over the node 'Aux' to the node 'Config' and to the new node 'RSSet': **...R**
 Change from the node 'RSSet' via the root into the node 'Mode': **&M**

4.1.2 Triggers

Triggers initiate an action at the Coulometer, e.g. starting of a mode or sending of data. Triggers are marked with the introducer: **\$**

The following triggers are possible:

| | | |
|----------|-------------------|--|
| \$G | Go: | Starts operations, e.g. start of the titration or setting of the RS232 interface parameters |
| \$S | Stop: | Stops operations, e.g. titration |
| \$Q | Query: | Used for inquiry of all information from the current node in the tree upwards up to and including the values |
| \$Q.P | Path: | Used for inquiry of the path from the root of the tree up to the current node |
| \$Q.H | Highest Index: | Used for inquiry of the number of son nodes of the current node |
| \$Q.N"i" | Name: | Used for inquiry of the name of the son node with index i, i = 1...n |
| \$D | Detailierte Info: | Used for inquiry of the detailed status |
| \$U | qUit: | Used to abort the data flow of the Coulometer, e.g. after \$Q |

The triggers \$G and \$S are linked to objects, see overview table, pages 22ff.

The other triggers, however, can be used at any time and at all locations on the object tree.

Examples:

- Inquiry of the value of the baud rate: **&Config.RSSet.Baud \$Q**
- Inquiry of all values of the node 'RSSet': **&Config.RSSet \$Q**
- Inquiry of the path of the node 'RSSet': **&Config.RSSet \$Q.P**
- Start of the current mode: **&Mode \$G**
- Inquiry of the detailed status: **\$D**

4.1.3 Status and error messages

In order to have an efficient control by an external control device, it must also be possible to query status conditions; they provide information on the status of the Coulometer. The trigger \$D initiates output of the status. Status messages consist of the global status, the detailed status and eventual error messages, e.g. \$\$S.Mode.Titr.ExtrTime;E20. The global status informs on the activity of the process, while the detailed status conditions show the exact activity within the process.

The following **global status conditions** are possible:

| | | |
|-------|--------|---|
| \$G | Go: | The Coulometer is executing the last command. |
| \$R | Ready: | The Coulometer has executed the last command and is ready |
| \$\$S | Stop: | A process has been stopped in an "unnatural manner". |

Detailed status conditions

Status conditions of the global \$G:

| | | |
|----------------|-----------|--|
| \$G .Mode.Cond | .Prog | Instrument is conditioning, wet or with negative drift. |
| | .Ok | Instrument is conditioning, dry. |
| \$G .Mode.Titr | .AddSmpl | Instrument is titrating, display "ADD SAMPLE". |
| | .Req.Id1 | Instrument is titrating, requesting Id1. |
| | .Req.Id2 | Instrument is titrating, requesting Id2. |
| | .Req.Id3 | Instrument is titrating, requesting Id3. |
| | .Req.Smpl | Instrument is titrating, requesting sample size. |
| | .Req.Conf | Instrument is titrating, waiting for start confirmation. |
| | .ExtrTime | Instrument is titrating, extraction time. |
| \$G .Mode.Titr | | Instrument is titrating. |

Status conditions of the global \$R:

| | | |
|----------------|-------|-------------------------------------|
| \$R .Mode.Inac | | Instrument in inactive state. |
| \$R .Mode.Cond | .Ok | Instrument is conditioning, dry. |
| \$R .Mode.Cond | .Prog | Instrument is conditioning, got wet |

Status conditions of the global \$\$S:

The instrument gives the status from which it has been stopped. The detailed status information is therefore identical as for the global status \$G or \$R.

Error messages, errors:

Error messages are appended to the status messages and separated from these by a ";".

- E26 Manual stop.
Exit: The error message disappears on next start.
- E190 Add Water. The EP has been exceeded.
Exit: Add water or &m \$S.
- E191 Blank Error. The blank value is larger than the titrated water mass.
Exit: Enter new blank or &m \$G.
- E192 Generator Electrode.
Exit: Rectify fault or &m \$S.
- E193 Indicator electrode.
Exit: Rectify fault or &m \$S.
- E194 Water R. Mass of titrated water larger than 65 535 ug.
Exit: &m \$G.
- E195 Sample Unfit: Overshooting of the EP during the titration.
Exit: Disappears when OK again.

RS receive errors:

- E36 Parity } Exit: <QUIT> and set appropriate parameter on the two devices to the
- E37 Stop Bit } same value.
- E38 Overrun error. At least 1 character could not be read.
Exit: <QUIT>
- E39 Overflow of the internal receive buffer of the Coulometer (> 82 characters).
Exit: <QUIT>

RS send errors

- E40 DSR = OFF } Handshake was not satisfactory for longer than 1 s.
- E41 DCD = ON } Exit: <QUIT>
- E42 CTS = OFF } Is the receiver switched on and ready to receive?
- E43 Transmission by the Coulometer was interrupted with XOFF for at least 3 s.
Exit: Send XON or <QUIT>.
- E44 The RS interface parameters are no longer the same for both devices. Reset.
- E45 The receive buffer of the Coulometer contains an incomplete character string (L_F missing).
Transmission of the Coulometer is therefore blocked.
Exit: Send L_F or <QUIT>.

4.2 Remote control commands

4.2.1 Overview

| Object | Function | Input range | see... |
|--------------------|---|--|----------|
| & | Root | | |
| Mode | Mode | \$G, \$S | 4.2.2.1 |
| .DriftDisp | Drift display | \$G, \$S | 4.2.2.2 |
| .Parameter | Parameters | | |
| .IReq | Request of Ids after the start | id1, id1&2, all, OFF | 4.2.2.3 |
| .SReq | Request of sample size after the start | ON, OFF | 4.2.2.3 |
| .ConfStart | Start confirmation | ON, OFF | 4.2.2.3 |
| .DriftStart | Drift threshold for start | 0...98 | 4.2.2.4 |
| .ExtrT | Extraction time | 0... ± 63999 | 4.2.2.5 |
| .StopDrift | Stop drift | | |
| .Select | Type of drift value transfer | auto, man | 4.2.2.6 |
| .Drift | Drift value on manual transfer | 0...98 | 4.2.2.6 |
| .TimeDelay | Switch-off delay time | 0...99 | 4.2.2.7 |
| .Report | Report output at end of titration | full, short, OFF | 4.2.2.8 |
| .CalcData | Data for result calculation | | |
| .Blank | Blank value | 0...63999 | 4.2.2.9 |
| .DCor | Drift correction | | |
| .Select | Type of drift value transfer | auto, man | 4.2.2.10 |
| .Drift | Drift value on manual transfer | 0...98 | 4.2.2.10 |
| Config | Configuration | | |
| .Aux | Setting of various auxiliary functions | | |
| .MpList | Mp list during the titration | | |
| .Select | Selection | ON, OFF | 4.2.2.11 |
| .Interval | Time interval for the output | 1...9999 | 4.2.2.11 |
| .Balance | Selection of the balance | Sartorius, Mettler, Mettler AT, AND, Precisa | 4.2.2.12 |
| .Periph | Peripheral at the "Remote" socket | Oven, Sampler | 4.2.2.13 |
| .Beep | Audio signal | ON, OFF | 4.2.2.14 |
| .DevName | Device name | 8 ASCII Char | 4.2.2.15 |
| .Prog | Program version | read only | 4.2.2.16 |
| .RSSet | Settings for RS232 | \$G | 4.2.2.17 |
| .Baud | Baud rate | special (300...9600) | 4.2.2.18 |
| .DataBit | Data bits | 7,8 | 4.2.2.18 |
| .StopBit | Stop bits | 1,2 | 4.2.2.18 |
| .Parity | Parity | even, odd, none | 4.2.2.18 |
| .Handsh | Handshake | HWs, HWf, SWchar, SWline, none | 4.2.2.18 |
| SmplData | Sample data | | |
| .SmplSize | Sample size | 0... ± 63999 | 4.2.2.19 |
| .Id1 | Sample identification 1 | up to 8 ASCII characters | 4.2.2.19 |
| .Id2 | Sample identification 2 | up to 8 ASCII characters | 4.2.2.19 |
| .Id3 | Sample identification 3 | up to 8 ASCII characters | 4.2.2.19 |

| Object | Function | Input range | see... |
|---------------------|---|--|----------|
| & | Root | | |
| Info | Information | | |
| .Report | Send formatted reports | \$G | 4.2.2.20 |
| .Select | Selection of the reports | configuration, parameters, smpl data, calc, full, short, all | 4.2.2.20 |
| .TitrResults | Results of the determination | | |
| .RunNo | Sample counter | read only | 4.2.2.21 |
| .Content | Calculated water content | read only | 4.2.2.21 |
| .UnitContent | Unit of water content | read only | 4.2.2.21 |
| .Water | Mass of titrated water | read only | 4.2.2.21 |
| .TitrTime | Titration time | read only | 4.2.2.21 |
| .StartDrift | Drift value | read only | 4.2.2.21 |
| Setup | Setting the operating mode | | |
| .IdReport | Output of report identification | ON, OFF | 4.2.2.22 |
| .Mode | Settings for the sequence | | |
| .StartWait | Hold directly after start | ON, OFF | 4.2.2.23 |
| .AutolInfo | Automatic information on changes | | |
| .R | If instrument in "Ready" condition | ON,OFF | 4.2.2.24 |
| .G | If instrument has been started | ON,OFF | 4.2.2.24 |
| .S | If instrument in "Stop" condition | ON,OFF | 4.2.2.24 |
| .B | Start of the actual titration | ON,OFF | 4.2.2.24 |
| .F | End of the determination | ON,OFF | 4.2.2.24 |
| .E | On error message | ON,OFF | 4.2.2.24 |
| .O | Conditioning OK | ON,OFF | 4.2.2.24 |
| .N | Conditioning Not OK | ON,OFF | 4.2.2.24 |
| .Re | If in request after titration start | ON,OFF | 4.2.2.24 |
| .RC | Recalculation of results | ON,OFF | 4.2.2.24 |
| .GC | Go Command | ON, OFF | 4.2.2.24 |
| .PowerOn | Simulation "Power on" | \$G | 4.2.2.25 |
| .Initialise | Set values to default | \$G | 4.2.2.26 |
| .Select | Selection | Mode, Config, Setup SmplData | 4.2.2.26 |
| .InstrNo | Instrument identification | \$G | 4.2.2.27 |
| .Value | Entry | up to 8 ASCII characters | 4.2.2.27 |
| .Ramlnit | Initialisation, see pages 43, 44 | \$G | 4.2.2.28 |

4.2.2 Description of the remote control commands

4.2.2.1 **Mode** \$G, \$S

Start or stop (\$G, \$S). \$G starts the preconditioning or the titration. \$S always leads to the inactive state. In requests for identification and the sample mass after the titration start, \$G is also used to continue to next step (see 4.2.2.3).

4.2.2.2 **Mode.DriftDisp** \$G, \$S

Switch display of the drift during the conditioning on/off. Corresponds to the <DRIFT> key.

4.2.2.3 **Mode.Parameter.IReq** id1, id1&id2, all, **OFF**
Mode.Parameter.SReq **ON, OFF**
Mode.Parameter.ConfStart **ON, OFF**

Automatic requests after the start of the titration. Advance of the titration by entry of the requested values or with &M \$G, see 4.2.2.1.

4.2.2.4 **Mode.Parameter.DriftStart** 0...98

Drift threshold for the start of the titration in ug/min. Entry in steps of 2 ug/min.

If the drift is above the set value, the titration can not be started and start commands are ignored (status message \$R.Mode.Cond.Prog).

4.2.2.5 **Mode.Parameter.ExtrT** 0... ± 63 999

Extraction time in s.

A negative extraction time corresponds to a wait time before the titration.

During the positive extraction time, I₂ is generated, but the titration will not be ended during the extraction time.

4.2.2.6 **Mode.Parameter.StopDrift.Select** man, **auto**
Mode.Parameter.StopDrift.Drift 0...20...98

Drift threshold for termination of the titration in ug/min.

.Select: Type of drift value acquisition. With "auto", the drift value at the start of the titration is valid. The drift value can be viewed in node &Info.TitrResults.StartDrift (4.2.2.21).

.Drift: Drift value for "man". Entry in steps of 2 ug/min.

4.2.2.7 **Mode.Parameter.TimeDelay** 0...3...99

Switch-off delay time in s.

4.2.2.8 **Mode.Parameter.Report** full, short, **OFF**

Type of report output at the end of the titration. OFF means no report output.

4.2.2.9 **Mode.CalcData.Blank** 0...63 999, 5 significant digits, up to 1 decimal place
Blank value in ug.

4.2.2.10 **Mode.CalcData.DCor.Select** man, **auto**
Mode.CalcData.DCor.Drift 0...98

Drift correction in ug/min. The drift correction is continuously performed during the titration.

- .Select: Type of drift value acquisition. With "auto", the drift value at the start of the titration is valid. The drift value can be viewed in node &Info.TitrResults.StartDrift (4.2.2.21).
- .Drift: Drift value for "man". Entry in steps of 2 ug/min.

4.2.2.11 **Config.Aux.MpList.Select** ON, **OFF**
Config.Aux.MpList.Interval 1...9999

Output of a list of measured points water/ug versus time/s during the titration.

.Interval: Time interval in s for output of the list of measured points.

4.2.2.12 **Config.Aux.Balance** **Sartorius**, Mettler, Mettler AT, AND, Precisa
Selection of the balance type.

4.2.2.13 **Config.Aux.Periph** **Oven**, Sampler
Selection of the peripheral device at the "Remote" socket.
With "Oven", the "ready (= end)" signal is static and a pulse is sent on stopping the Coulometer (see page 51).

4.2.2.14 **Config.Aux.Beep** ON, OFF
Switch audio signal on/off.

4.2.2.15 **Config.Aux.DevName** up to 8 ASCII characters
Name of the device for interconnections with several units. It is advisable to use only the characters A...Z (ASCII No. 65...90), a...z (ASCII No. 97...122) and 0...9 (ASCII No. 48...57) when the Setup.AutoInfo function (4.2.2.24) is used at the same time.

4.2.2.16 **Config.Aux.Prog** read only
Output of the program version.
On receipt of \$Q, the Coulometer sends: "737.0010".

4.2.2.17 **Config.RSSet** \$G
\$G sets all RS settings. After the setting of the interface parameters, wait at least 2 s for the components to stabilise.

| | | |
|----------|-----------------------------|---|
| 4.2.2.18 | Config.RSSet.Baud | 300, 600, 1200, 2400, 4800, 9600 |
| | Config.RSSet.DataBit | 7, 8 |
| | Config.RSSet.StopBit | 1, 2 |
| | Config.RSSet.Parity | even, odd, none |
| | Config.RSSet.Handsh | HWs , HWf, SWchar, SWline, none |

Setting of the values for data transfer via RS interface.: Baud rate, data bits, stop bits, parity and type of handshake, see also page 28 ff.

Setting of the values must be initiated with \$G immediately after entry of the values, see 4.2.2.17.

| | | |
|----------|--------------------------|---|
| 4.2.2.19 | SmplData.SmplSize | -63999... 0 ...63 999, 5 significant digits, up to 1 decimal place |
| | SmplData.Id1 | up to 8 ASCII characters |
| | SmplData.Id2 | up to 8 ASCII characters |
| | SmplData.Id3 | up to 8 ASCII characters |

Sample data.

.SmplSize: Sample size in mg.

| | | |
|----------|---------------------------|--|
| 4.2.2.20 | Info.Report | \$G |
| | Info.Report.Select | configuration, parameters, smpl data, calc, full, short, all |

\$G sends the selected report.

configuration: Configurations report. Not accessible during a titration.

parameters: Parameter report of the current method. Only "live" parameters during a titration.

smpl data: Sample data.

calc: Calculation data.

full: Full result report of the last titration.

short: Short result report of the last titration.

all: All reports.

Reports sent automatically by the Coulometer start with a space (ASCII 32) and '. This is followed by an individual identification for each report. Reports requested via RS232 (\$G) have the same individual identification of the report blocks, but are started with ' only (no preceding space).

| | | |
|----------|------------------------------------|-----------|
| 4.2.2.21 | Info.TitrResults.RunNo | read only |
| | Info.TitrResults.Content | read only |
| | Info.TitrResults.UnitCont | read only |
| | Info.TitrResults.Water | read only |
| | Info.TitrResults.TitrTime | read only |
| | Info.TitrResults.StartDrift | read only |

Request for the current titration results:

.RunNo: Current status of the sample counter.

.Content: Calculated water content.

.UnitCont: Unit of the calculated water content (% or ppm)

.Water: Titrated water mass in ug.

.TitrTime: Titration time in s.

.StartDrift: Drift value at start of titration in ug/min.

| | | |
|----------|-----------------------|-----------------|
| 4.2.2.22 | Setup.IdReport | ON , OFF |
|----------|-----------------------|-----------------|

Switch output of the report identification of the formatted reports on/off.

| | | |
|----------|-----------------------------|----------------|
| 4.2.2.23 | Setup.Mode.StartWait | ON, OFF |
|----------|-----------------------------|----------------|

Wait time after the start. The data of the last titration are retained during this time.

Is set to OFF when switching on the Coulometer.

| | | |
|----------|-------------------------|---------|
| 4.2.2.24 | Setup.AutInfo.R | ON, OFF |
| | Setup.AutInfo.G | ON, OFF |
| | Setup.AutInfo.S | ON, OFF |
| | Setup.AutInfo.B | ON, OFF |
| | Setup.AutInfo.F | ON, OFF |
| | Setup.AutInfo.E | ON, OFF |
| | Setup.AutInfo.O | ON, OFF |
| | Setup.AutInfo.N | ON, OFF |
| | Setup.AutInfo.Re | ON, OFF |
| | Setup.AutInfo.RC | ON, OFF |
| | Setup.AutInfo.GC | ON, OFF |

ON means the Coulometer reports automatically when the corresponding change starts.

| | |
|-----|--|
| .R | Ready: 'Ready' state has been reached. |
| .G | Go: Instrument has been started. |
| .S | Stop: 'Stop' state has been reached. |
| .B | Begin: Start of the titration (after requests). |
| .F | Final: End of the determination, the closing steps are being performed. |
| .E | Error. Message together with error number, see page 21. |
| .O | Conditioning OK, dry. |
| .N | Conditioning Not OK, wet |
| .Re | Request: Request of the sample identification, the sample size after titration start or "Confirm Start". |
| .RC | Recalculation: Recalculation |
| .GC | Go Command: The Coulometer has received a start command. |

If a change occurs which requires a message, the Coulometer sends space (ASCII 32) and ! as introducers. The name of the device is then sent (see 4.2.2.15). Special ASCII characters in the device name are ignored. If no device name has been entered, only ! is sent. Finally, the 737 sends information regarding the node which initiated the message.

Example: !Otto".R": With device "Otto", node .R initiated the message.

4.2.2.25 **Setup.PowerOn** \$G

Simulation of 'Power on'. The device is in the same condition as after power on: Error messages are cleared, StartWait (see 4.2.2.23) is set to OFF and the current sample number to 0.

4.2.2.26 **Setup.Initialise** \$G **Setup.Initialise.Select** Mode, Config, Setup, SmpIData

Setting of default values for the following groups:

| | |
|-----------|--|
| Mode: | All values under tree section &Mode. |
| Config: | All values under tree section &Config. |
| Setup: | All values under tree section &SetUp. |
| SmpIData: | All values under tree section &SmpIData. |

The action must be initiated with &SetUp.Initialise \$G.

4.2.2.27 **Setup.InstrNo** \$G **Setup.InstrNo.Value** up to 8 ASCII characters

Device identification for output in the reports.

The value must be written in with &SetUp.InstrNo \$G.

4.2.2.28 **Setup.RamInit** \$G

Initialised instrument as in the diagnostic test, see pages 43 and 44: All parameters are set to their default value and error messages are cleared.

4.3. Characteristics of the RS232 interface

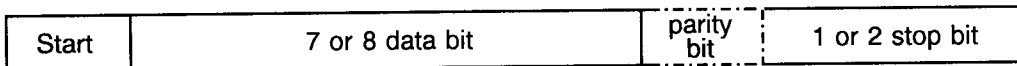
4.3.1 Data transfer protocol

The Coulometer is configured as DTE (Data Terminal Equipment).
The RS232 interface has the following technical specifications:

Data interface in accordance with the RS 232C standard. Data transfer parameters can be selected.

Max. line length: 80 characters + C_R L_F
 Control characters: C_R (ASCII DEC 13)
 L_F (ASCII DEC 10)
 XON (ASCII DEC 17)
 XOFF (ASCII DEC 19)

Cable length: max. ca. 15 m



For interconnections of the Coulometer with non-Metrohm units, only a shielded data cable (e.g. METROHM D.104.0201) may be used. The cable shielding must be faultlessly earthed at both units (pay attention to current loops; always use star-head earthing). Only connectors with adequate shielding may be used (e.g. METROHM K.210.0001 with K.210.9004).

4.3.2 Handshake

4.3.2.1 Software-Handshake , SWCHAR

Handshake inputs at the Coulometer (CTS, DSR, DCD) are not checked.

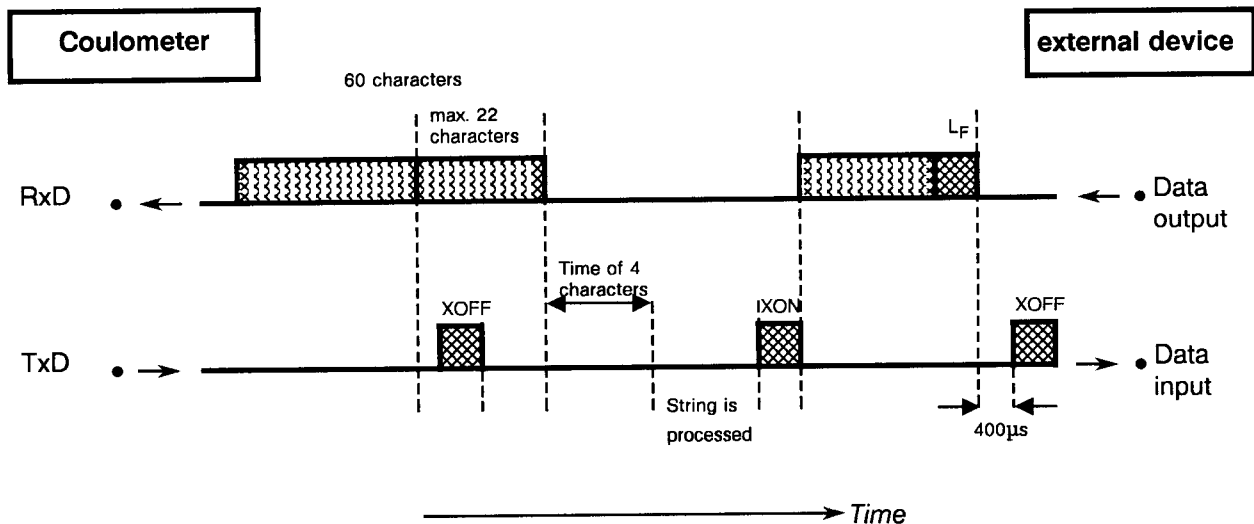
Handshake outputs (DTR, RTS) are set by the Coulometer.

As soon as a L_F is recognised, the Coulometer sends XOFF. It can then receive 6 extra characters and store them.

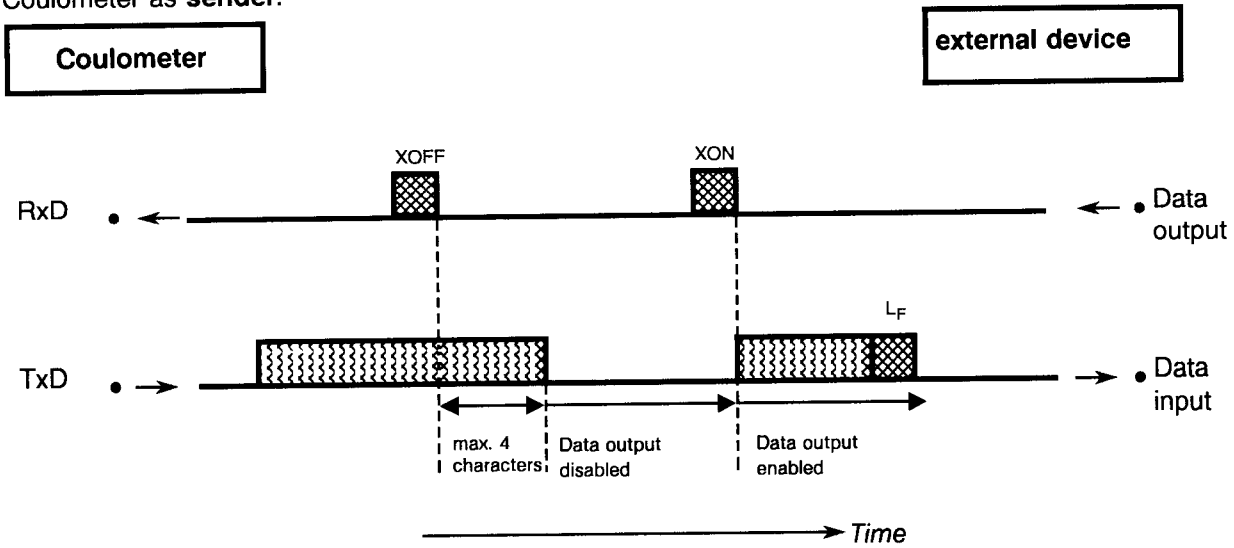
However, the Coulometer also sends XOFF if its input buffer contains 60 characters. After this, it can receive maximum 22 extra characters (incl. L_F).

If the transmission is interrupted for the time of 4 characters after the Coulometer has sent XOFF, the string received earlier is processed even if no L_F has been sent.

Coulometer as receiver:



Coulometer as sender:



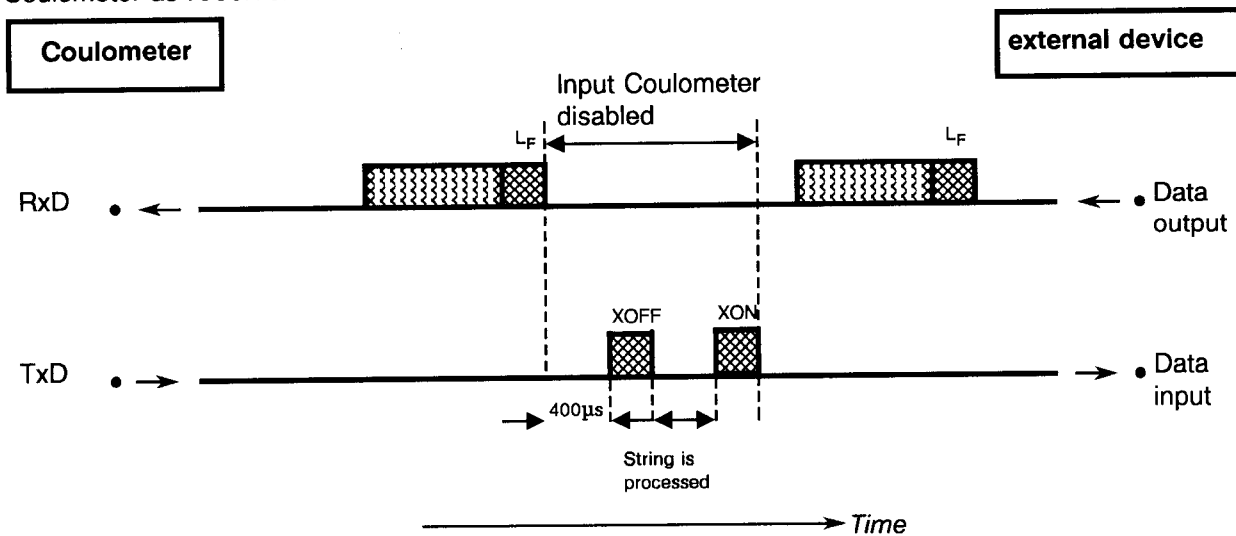
4.3.2.2 Software-Handshake , SWLINE

Handshake inputs at the Coulometer (CTS, DSR, DCD) are not checked.

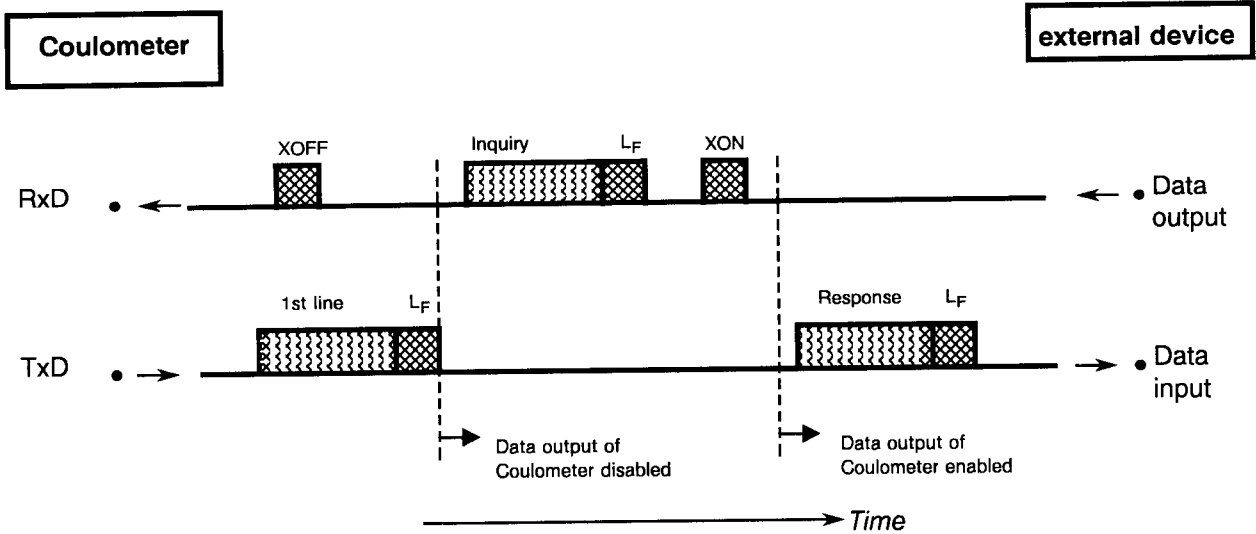
Handshake outputs (DTR, RTS) are set by the Coulometer.

The Coulometer is equipped with an input buffer that can accommodate a string of up to 80 characters + C_{RLF} . As soon as an L_F is recognised, the Coulometer sends XOFF. After this, it can receive maximum 6 extra characters and store them. The string sent previously is now processed by the Coulometer. Afterwards, the Coulometer sends XON and is again ready to receive.

Coulometer as receiver:



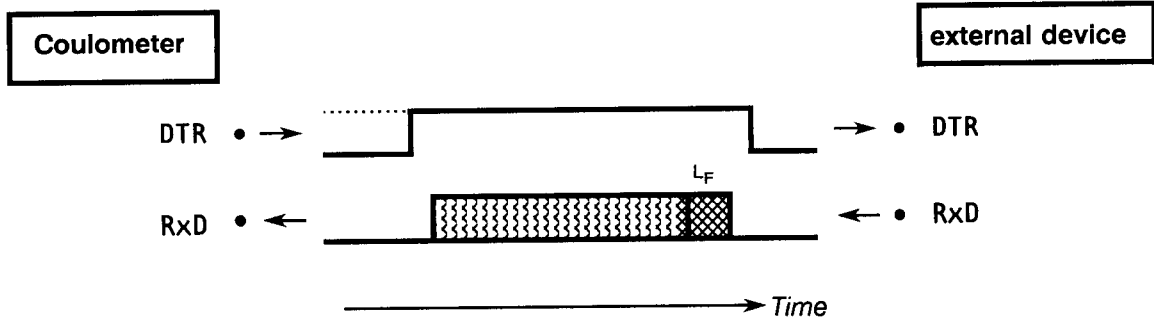
Coulometer as **sender**:



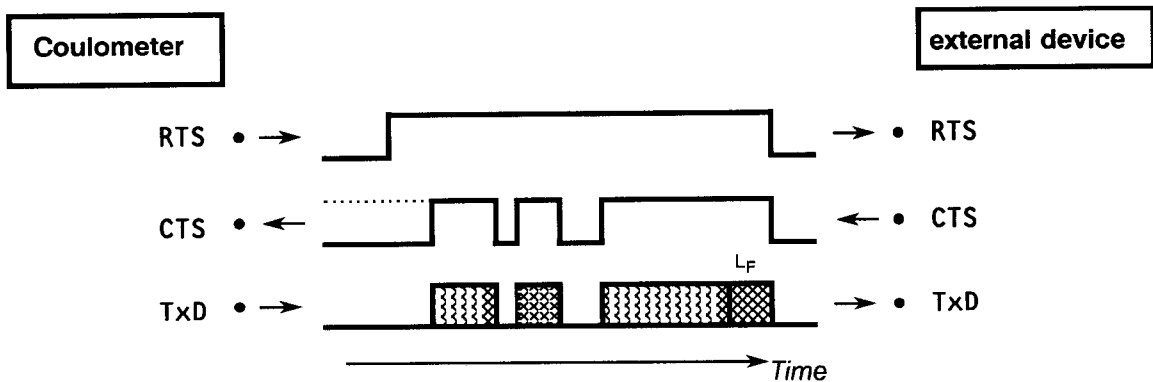
The transmission of the Coulometer can be stopped by the external device with XOFF. After receipt of XOFF, the Coulometer completes transmission of the line already started. If the data output is disabled for more than 3 s by XOFF, E43 appears in the display.

4.3.2.3 Hardware-Handshake, HWS

Coulometer as **receiver**:



Coulometer as **sender**:

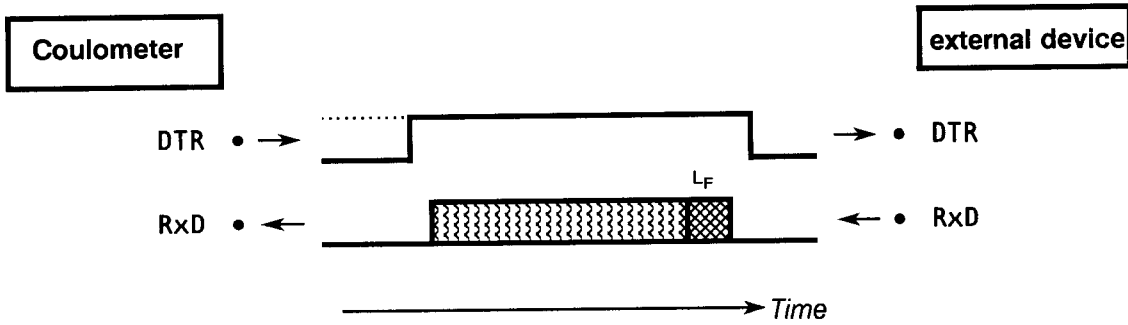


The data flow can be interrupted by deactivation of the CTS line.

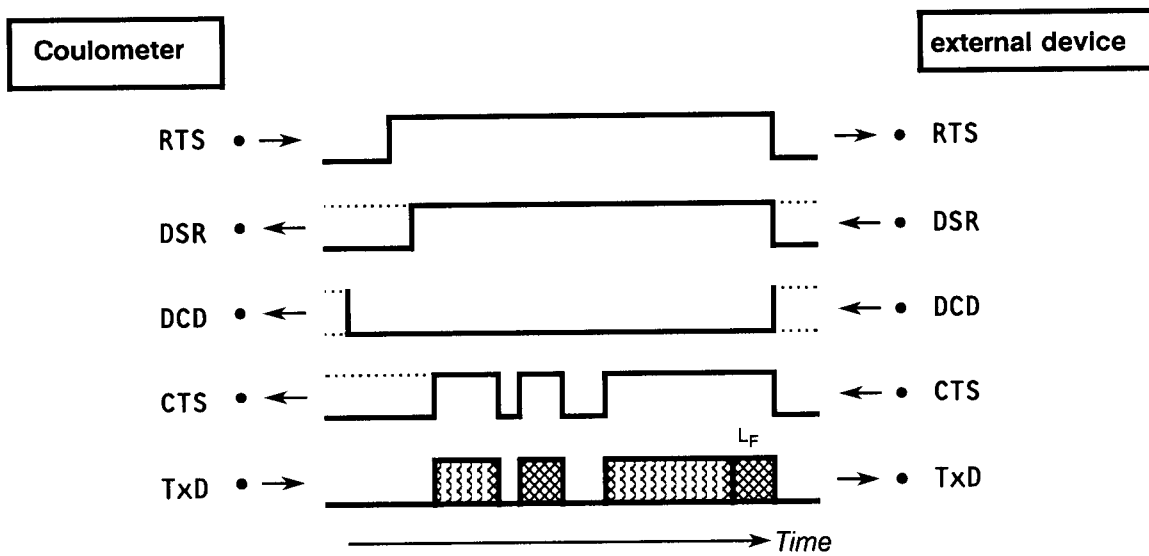
4.3.2.4 Hardware-Handshake, HWF

All handshake inputs are checked at the Coulometer, handshake outputs are set.

Coulometer as receiver:

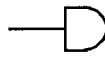
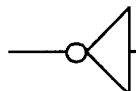
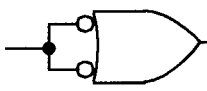
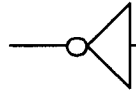
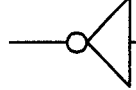
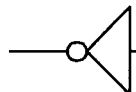
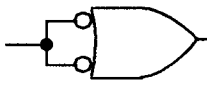


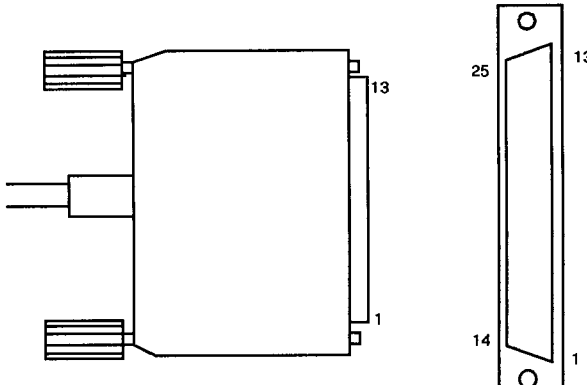
Coulometer as sender:



The data flow can be interrupted by deactivation of the CTS line.

4.3.3 Pin assignment

| | | external |
|---|---|-------------------------------------|
| <h2>RS 232C Interface</h2> | | |
| <p>Transmitted data (TxD). If no data are transmitted, the line is held in the "ON" condition. Data are transmitted only when CTS and DSR are in the "ON" condition and DCD is in the "OFF" condition.</p> |  | <p>E 2 Transmitted Data</p> |
| <p>Received data (RxD) Data are received only when DCD is "ON".</p> |  | <p>E 3 Received Data</p> |
| <p>Request to Send (RTS) ON condition: Coulometer is ready to send data.</p> |  | <p>E 4 Request to Send</p> |
| <p>Clear to Send (CTS) ON condition: Remote station is ready to receive data.</p> |  | <p>E 5 Clear to Send</p> |
| <p>Data Set Ready (DSR) ON condition: The transmission line is connected.</p> |  | <p>E 6 Data Set Ready</p> |
| <p>Signal Ground (GND)</p> | | <p>E 7 Signal Ground</p> |
| <p>Data Carrier Detect (DCD) ON condition: The level of the received signal is within the tolerance range (remote station is ready to send data).</p> |  | <p>E 8 Data Carrier Detect</p> |
| <p>Data Terminal Ready (DTR) ON condition: Coulometer is ready to receive data.</p> |  | <p>E 20 Data Terminal Ready</p> |

| | | |
|--|--|-----------------|
| <p>RS 232C (continued)</p> <p>Protective earth Direct connection from cable connector to protective earth of instrument.</p> <p>Polarity allocation of the signals</p> <ul style="list-style-type: none">- Data lines (TxD, RxD)<ul style="list-style-type: none">Voltage negative (< -3 V): signal status "ONE"Voltage positive (> +3 V): signal status "ZERO"- Control or message lines (CTS, DSR, DCD, RTS, DTR)<ul style="list-style-type: none">Voltage negative (< -3 V): OFF statusVoltage positive (> +3 V): ON status <p>In the transition region from +3 V to -3 V the signal status is undefined.</p> <p>Driver 14C88</p> <p>Receiver 14C89</p> <p> } in compliance with EIA RS 232C specification</p> <p>Contact arrangement at connector (female) for RS 232 C socket (male)</p>  <p>Ordering numbers: K.210.9004 und K.210.0001</p> <p>View from solder side of connector</p> | | <p>external</p> |
| <p>No liability whatsoever will be accepted for damage or injury caused by improper interconnection of instruments.</p> | | |

4.4 What can you do if the data transfer does not work?

| Problem | Questions for remedial action |
|--|---|
| No characters can be received on a connected printer | <ul style="list-style-type: none">- Are the instruments and the connection cables plugged in properly?- Is the printer set to "on-line" and receipt with serial interface?- Are baud rate, data bit and parity settings the same for both devices?- Is the handshake set properly? <p>If everything appears to be in order, try to print out a report with the key sequence <PRINT> <SMPL DATA> <ENTER>. If this report is printed out correctly, check whether under the key <PARAM>, >report a report is preselected.</p> |
| No data transmission occurs and the display of the Coulometer shows an error message | <ul style="list-style-type: none">- E40-42: Transmission error. Is the cable properly wired and connected? Is the printer switched on and set to "on-line"?- E43: Data output of the Coulometer disabled for longer than 3 s by XOFF.- E36-39: Receive error. Are the settings of the RS232 data transmission parameters the same for both devices? |
| The received characters are garbled | <ul style="list-style-type: none">- Are the data bit and the parity settings the same for both devices?- Is the baud rate setting the same for both devices? |

5. Error messages, rectification of malfunctions

5.1 Titration problems, troubleshooting

| Problem | Corrective action |
|-----------------------------|---|
| Printing not possible | See measures, page 34. |
| Drift too high | <ul style="list-style-type: none"> - Water-containing deposits in the measuring cell: shake measuring cell. - Reagent exhausted: Change reagent. - Moisture ingress into measuring cell: <ul style="list-style-type: none"> . Molecular sieves exhausted? . Septum still OK? . Seals still OK (measuring cell with diaphragm)? - Diaphragm contaminated or not dry (measuring cell with diaphragm), see page 4. - Sample matrix consumes iodine. Change reagent more frequently or extract water of sample so that sample need not be added to the measuring cell. - In work with KF oven: <ul style="list-style-type: none"> . Gas flow too high? . Screw fittings tight? |
| Drift frequently negative | Add water and check whether the drift becomes negative again. <ul style="list-style-type: none"> - Stir somewhat faster (iodine clouds). - Activate indicator electrode, see page 8. - Set end point voltage lower, see page 12. |
| Fluctuating drift | <ul style="list-style-type: none"> - Moisture penetrates measuring cell: <ul style="list-style-type: none"> . Molecular sieves exhausted? . Septum still OK? . Seals still OK (measuring cell with diaphragm)? - Set end point voltage lower, see page 12. |
| Measurement result too high | <ul style="list-style-type: none"> - Measuring cell not yet conditioned well: Shake measuring cell and wait until drift is stable. - The indicator electrode is passivated. Clean electrode, see page 8. - Set stop drift higher, see page 11. - Set switch-off delay time lower, see page 11. |
| Titration times too long | <ul style="list-style-type: none"> - Check whether drift is stable during conditioning; if not, take same measures as for drift problem. - Water quantity too high. Guide values for the sample size, see page 6. - Set stop drift higher, see page 11. - Set switch-off delay time lower, see page 11. - Set end point voltage higher, see page 12. |

5.2 Error and special messages

| | |
|----------------------|---|
| ADD WATER | The electrolyte solution is in the excess iodine range. Exit: Add water. |
| BATTERY | The battery is low and must be replaced. Call Metrohm Service. In the mean time you may continue as follows: <ul style="list-style-type: none">- Initialize RAM (press key <9> right after switching on the Coulometer, then press <ENTER>, then <QUIT>).- Enter your configuration data, your parameters and sample data. The error message will reappear if you switch the Coulometer off and on again. |
| BLANK ERROR | The blank value is larger than the mass of the titrated water. Exit: Enter new BLANK. |
| GEN ELECTRODE | The voltage drop across the generator system is too high. Possible causes: <ul style="list-style-type: none">- Wrong or exhausted electrolyte- With measuring cells with diaphragm: Clogged diaphragm or bubbles between platinum gauze and diaphragm- Stirring too fast, bubbles remain in the generator electrode- With diaphragm-free measuring cells: Too much solvent was added to the electrolyte resulting in a conductivity that is too low. → Change solution.- Generator electrode not connected, faulty or break in the connecting cable. Exit: Rectify fault |
| IND ELECTRODE | The voltage at the indicator electrode is too high. Possible causes: <ul style="list-style-type: none">- Wrong electrolyte- Indicator electrode not connected, faulty or break in the connecting cable Exit: Rectify fault |
| MANUAL STOP | A titration or the conditioning was terminated manually. Exit: <START > |
| PROM ERROR | Checksum error. Corrective action: Call Metrohm service. |
| SAMPLE UNFIT | Possible causes and corrective action: <ul style="list-style-type: none">- Stirring too slow: stir faster- End point voltage too high for the fast reagent used: Set end point voltage lower using the potentiometer at the rear of the instrument.- Sample releases iodine: Change solution; the sample is not suitable for the KF titration. |
| WATER R | Mass of titrated water exceeds 65 535 µg. (The mass of water effectively present is thus the sum of the displayed value and 65 535 µg.) |

Error messages in connection with the data transfer

If neither a computer nor a printer is attached, the report output at the end of the titration must be switched off.

Receive error:

E36 Parity
E37 Stop bit } Exit: <QUIT> and set corresponding
parameter on both devices to the same
value.

E38 Overrun error. At least 1 character could not be read.

Exit: <QUIT>

E39 Overflow of the receive buffer of the Coulometer (> 82 characters).

Exit: <QUIT>

Send errors:

E40 DSR = OFF
E41 DCD = ON
E42 CTS = OFF } Handshake was not satisfied for longer than 1 s.
Exit: <QUIT>
Is the receiver switched on and ready to
receive?

E43 Transmission by the Coulometer was interrupted with XOFF for at least 3 s.

Exit: <QUIT>

E44 The RS interface parameters are no longer the same for both devices.
Set them equally.

E45 The receive buffer of the Coulometer contains an incomplete character
string (L_F missing). Transmission by the Coulometer is thus blocked.

Exit: Send L_F or <QUIT>.

5.3 Diagnostic test

Virtually all faults on the 737 Coulometer arise through faulty operation and handling of the instrument, the electrodes, the solutions, etc. Many of these faults are shown in the display of the KF Coulometer or can be recognised in the result.

If there are valid reasons for suspecting a malfunction of the 737 Coulometer, the diagnostic test is simple to perform and will help isolate the cause.

Required aids

- Ready to use generator electrode (or short circuit "Gen.El" input with Service Cable 3.496.5080 1)
- Cable with suitable connector for "Ind.El." input² (e.g. 3.496.5070 Service Cable)¹
- Resistance decade for values 1 200 Ω , 1 850 Ω , 1/4 W. If no decade is available, individual resistors can be used. See page 39.
- perhaps test plug 3.496.8480 (see point 8)

Procedure

- Perform the diagnostic steps in the order given and compare with the reactions of the Coulometer (indented). On agreement, proceed to the next instruction.
- If the instrument does not show the expected reaction, it is first advisable to repeat the diagnostic test as an operating error could have occurred. However, several wrong reactions indicate a malfunction.
- If repetitions are necessary for more accurate observations, a return to the next re-entry point marked by \gg must be effected. Possibly restart the diagnosis from the beginning, see point 3.
- If any of the points 9 - 12 need be repeated, repeat the whole block 9 - 12.
- On inquiries, always specify the serial number of the instrument and if applicable the No. of the faulty diagnostic step.
- Corrective actions in the opened instrument may be undertaken only by METROHM service!

Preparations

The generator electrode remains attached and immersed in the anolyte solution or short-circuit Gen. El input with service cable 3.496.5080.

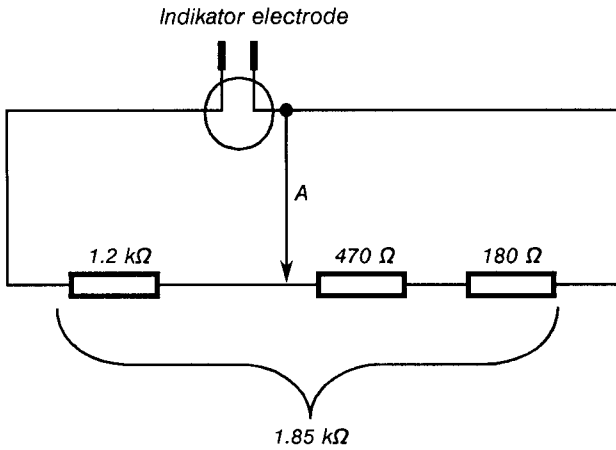
The indicator electrode is withdrawn and a resistance decade attached in its place using a cable with a suitable connector² (e.g. 3.496.5070¹). If no decade is available, see arrangement overlief.

¹ Not part of the standard equipment of the 737.
Can possibly be prepared from a faulty electrode.

² If no suitable cable is available:
Take indicator electrode out of cell. Carefully attach decade or resistor combination with laboratory cable and test clips to the platinum wires of the indicator electrode.
(Warning: Do not bend platinum wires!)

Note only if no resistance decade is available:

All required resistance values can be produced with 3 individual resistors of 1 200 Ω, 470 Ω and 180 Ω (accuracy class 1%, ± W) and appropriate connecting cables (laboratory cable and connector clips). However, when switching to other values at the electrode input, **no interruption** ($R \neq \Omega$) must occur. This is possible by proceeding according to the following arrangement.



- Arrangement for point 9 Bridge A closed
- Arrangement for point 10 Bridge A open
- Arrangement for point 11 Bridge A closed
- Arrangement for point 12 Bridge A open

Fig. 1 Replacement circuit for resistor switch-box

1. Check switch-on routine

Switch on instrument and observe switch-on routine.

8888888888888888

XXXXXXXXXXXXXXXXXX

|. |. |. |. |. |. |. |. |. |. |. |. |. |. |.

START COND.

2. Test NOVRAM

Press <PARAM> possibly several times until display shows

DELAY TIME XX X

Enter the delay "10 s" with the numeric keys and press <ENTER>.

REPORT ▽ XXXX

X = selected report

Switch off instrument.

After 5 s switch on instrument and wait for startup routine to end.

Press <PARAM> possibly several times until display shows

DELAY TIME 10 S

The display of the delay must be 10 s.

➤ 3. **Select diagnosis**

Switch off instrument then after about 5 s switch on again and immediately press the <9> key (press briefly only after switching on).

DIAGNOSIS▽ INIT

➤ 4. **RAM testen**

Press <SELECT> possibly several times until display shows

DIAGNOSIS▽ RAM

<ENTER>

RAM TEST OK

<QUIT>

DIAGNOSIS▽ RAM

➤ 5. **Timer test**

Press <SELECT> possibly several times until display shows

DIAGNOSIS▽ TIMER

<ENTER>

TIMER TEST OK

<QUIT>.

DIAGNOSIS▽ TIMER

➤ 6. **Display test**

Press <SELECT> possibly several times until display shows

DIAGNOSIS▽ DISPL

<ENTER>

The display test starts and the display is filled in the following sequence:

8888888888888888

XXXXXXXXXXXXXXXXXX

I . I . I . I . I . I . I . I . I . I . I . I . I . I . I . I .

0123456789ABCDEF

GHIJKLMNOPQRSTUVWXYZ

WXYZ[\]*+ - / < = > % ▽

At the end of the test, the following appears:

DIAGNOSIS ▽ DISPL

➤ 7. Keypad + LED test

Press <SELECT> possibly several times until display shows

DIAGNOSIS ▽ KEYB

<ENTER>

The LED "COND" lights up.

LED COND ON

<ENTER>

The LED "COND" goes out.

LED COND OFF

<ENTER>

CONTINUE TEST

Finally, check all keys except QUIT.

The pressed key is shown in the display,

<QUIT>

DIAGNOSIS ▽ KEYB

| No | Key |
|----|-----------|
| 1 | 7 |
| 2 | 4 |
| 3 | 1 |
| 4 | 0 |
| 5 | 8 |
| 6 | 5 |
| 7 | 2 |
| 8 | . |
| 9 | 9 |
| 10 | 6 |
| 11 | 3 |
| 12 | - |
| 13 | ENTER |
| 14 | CLEAR |
| 15 | SELECT |
| 17 | PARAM |
| 18 | CONFIG |
| 19 | CALC/DATA |
| 20 | SMPL/DATA |
| 21 | DRIFT |
| 22 | PRINT |
| 23 | STOP |
| 24 | START |

➤ 8. RS 232 test

This test is meaningful only if the 737 KF Coulometer is used interconnected with other instruments via the 'RS 232' connection. In addition, a 3.496.8480 Test Plug normally used in the repair service is required for this test. However, this plug can also be purchased by customers under the above number.

For the sake of completeness, the procedure is described here.

(If a diagnostic test of the RS232 interface is not required, continue with point 9.)

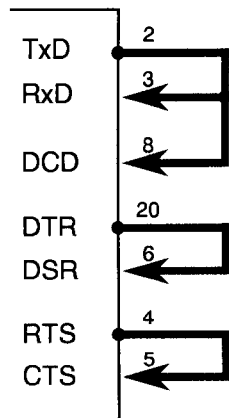


Fig. 2 Connections in the 3.496.8480 Plug

Press <SELECT> possibly several times until display shows

DIAGNOSIS▽ RS232

<ENTER>

RS CONNECTOR

Insert 3.496.8480 Plug in 'RS 232' port (do not switch off instrument, pay attention to alignment of the plug!).

<ENTER>

The test runs automatically. If no error is found, 'RS TESTED' appears after ca. 3 s. Otherwise, an error message is shown. If no test plug is connected, 'ERROR RS' appears.

Remove test plug.

<QUIT>

DIAGNOSIS▽ RS232

➤ 9. Check function "start readiness"

Switch test decade to 1.2 k. If no decade available, see replacement circuit, page 39: bridge A closed.

Mark current position of potentiometer 'EP Voltage' (rear of instrument) and set it to the left stop (to "low").

<START>

ADD WATER

10. Function check 'value less than end point before start'

Switch test decade to 1.85 k (or replacement circuit: A open).

counts forward
WAIT 00XXX

positive value

The LED "COND" flashes.

If display shows 'GEN ELEKTRODE' or 'IND ELEKTRODE', please check the corresponding connections.

6.2 Connection of a printer


Any printer with an RS232 interface can be connected to the RS232 interface of the Coulometer.

If a **balance** has to be attached at the same time as a printer, the 6.2125.030 Socket-Outlet Adapter must be used. The printer must be plugged into the "data out" receptacle of the socket-outlet adapter. It can be operated only with the simple hardware handshake (HWs) or without a handshake.

The following Table services to facilitate the connection of several printers:

| Printer | Cable | Settings on Coulometer | Settings on printer |
|--------------------------|------------|---|---|
| Citizen iDP560 RS | 6.2125.050 | Baud rate: 9600 Data bits: 7 Stop bits: 1 Parity: even Handshake: HWs | Dip switches 1 on 2 off } 3 off } 9600 baud 4 off } 5 on 7 bits 6 - 7 off } even 8 on } parity Set printer on-line with <sel> |
| Seiko DPU-411 | 6.2125.020 | Baud rate: 9600 Data bits: 7 Stop bits: 1 Parity: even Handshake: HWs | Dip switches: DIP01 1 off serial 2 off no auto LF 3 on 40 characters 4 on character type 5 off zero represent. 6 off } 7 on } USA 8 on } -character set Set printer on-lone |

Other printers can be attached:

| Printer | Cable | Settings on 737 | Settings on printer |
|--|------------|---|--|
| Epson printer with 6-pin round connector ¹⁾ | 6.2125.040 | Data bits: 8 Parity: none Handshake: HWs | 8 bits no parity |
| Epson printer with #8148 interface | 6.2125.050 | Data bits: 7 Parity: even Handshake: HWs | 7 bits even parity |
| HP: Think Jet | 6.2125.050 | Baud rate: 9600 Data bits: 7 Parity: even Handshake: HWs | Switches:  |

6. Interconnections with other devices

Ensure that the set operating voltage matches the mains voltage before you switch on the instrument. The mains cables supplied with the instrument are three core and fitted with a plug with an earthing pin. If another plug has to be fitted, the yellow/green conductor must be connected to the protective earth. If no socket with earthing is available, the instrument must be connected to a perfect earth conductor via the earthing socket. Each break in the earthing inside or outside the instrument can make this a hazard. If the instrument is opened or if parts are removed from it, certain components can be live if the instrument is connected to the mains.

The mains cable must therefore always be disconnected when certain settings are made or parts replaced.

6.1 Coulometer with work stand

The base of the stand can be mounted on the left or right.

For work with the 707 KF Oven, the work stand must be on the left of the Coulometer. It is advisable to fix the stand in the rear position.

The instruments are setup and connected as shown in Fig. 6-1.

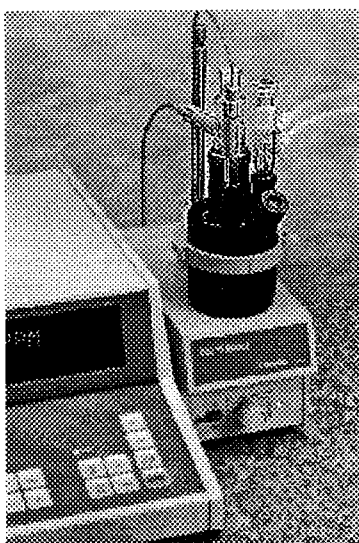


Fig. 6-1 a: With 703 Ti Stand



Fig. 6-1 b: With 728 Stirrer

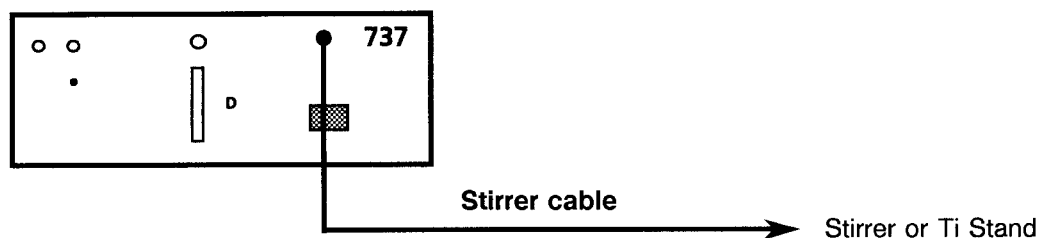


Fig. 6-1: Setting up the Coulometer and attaching the work stand

Procedure

Switch on instrument and then immediately press the <9> key briefly.

DIAGNOSIS ∇ INIT

<ENTER>

DIAGNOSIS ∇ INIT

Switch off instrument.

Note for error case

If, despite a positive diagnostic test, proper functioning of the instrument should be in doubt, there are 2 more error possibilities namely

- a) faulty generator electrode or
- b) faulty generator electrode current, i.e. fault in instrument.

Information on this is provided by repeating your determination with a different, good electrode.

If no second electrode is available, an electrical specialist (electronic engineer or service electrician) can check the electrode current, as follows.

Aids required (to check the electrode current):

- 3.496.5080 Service Cable or 2 normal laboratory cables with clips for attachment to "Gen.El" socket in the interior of the instrument after removal of the cover plate (2 M4 screws).
- Universal unit or ammeter for measured value 357 mA dc

Procedure

- Attach measuring instrument using cable (see above) to "Gen.El", select dc current range.
- Power on and select diagnosis by pressing key <9>.
- Press <SELECT> possibly several times until display shows

DIAGNOSIS ∇ IO

- Press <ENTER> possibly several times until display shows

IG ON

The ammeter records a constant current of 357 mA (\pm 1 mA; take tolerance of measuring instrument into account)
Correct current flow (on wrong result) points to an error in the generator electrode.

11. Check switch-off point

With existing settings, switch test decade to 1.2 k (or replacement circuit: A closed).

The beep sounds briefly after ca. 5 s.

The LED "COND" is continuously on.

DRIFT 0000X

positive vlaue

12. Check on the calibration

With existing settings, switch test decade to 1.85 k (or replacement circuit: A closed).

DRIFT 00XXX

The numeric value must be 00324...00338.

13. Check alarm "no indicator electrode"

With existing settings, switch test decade to ∞ or disconnect cable.

IND ELECTRODE

Reset test decade to 1.85 k.

DRIFT 00XXX

*The numeric value must be 00324...00338.
Please consider tolerance of decade.*

14. Check alarm "no generator electrode"

Disconnect "Gen.El".



GEN ELECTRODE

End of test. Switch off instrument.

Reset potentiometer 'EP Voltage' to the original setting and remove marking.

Initialise RAM (carry out only in an emergency!)

On the odd occasion, large interference signals (e.g. power spikes, lightning) may have an adverse effect on the processor functions and hence lead to a system crash. After a system crash, the RAM area must be initialised. Although the basic instrument data are retained, the RAM initialisation should be performed only if necessary as the stored user data (electrode calibration data, selected buffers, configurations, etc.) are cleared.

| Printer | Cable | Settings on 737 | Settings on printer |
|---------------------------|------------|---|--|
| HP: Deskjet ¹⁾ | 6.2125.050 | Baud rate: 9600 Data bits: 8 Parity: none Handshake: HWs | A:  B:  |
| Kodak Diconix 180 si | 6.2125.050 | Baud rate: 9600 Data bits: 7 Parity: even Handshake: HWs | Epson emulation 7 bits even parity |

1): On simultaneous connection of a balance which can operate only with 7 bits, "parity space" should be set on the balance, whereas printer and Coulometer operate with 8 bits, no parity.

6.3 Connection of a balance

The following balances can be connected to the RS232 output of the Coulometer.

| Balance | Cables |
|---------------------------------------|--|
| Sartorius MP8, MC1 | 6.2125.070 |
| Mettler AM, PM | from Mettler: ME 33995: Green wire to pin 2, brown to pin 3, white to pin 7, yellow to pin 20 of the 25-pin connector. |
| Interface 016: | Cable part of standard equipment of interface 016: Red wire to pin 3, white wire to pin 7 of the 25-pin connector |
| Interface 011 or 012 | 6.2125.020 |
| Mettler AT | from Mettler: ME 33995: Green wire to pin 2, brown to pin 3, white to pin 7, yellow to pin 20 of the 25-pin connector. |
| AND Types ER-60, 120, 180, 182 | 6.2125.020 |
| Types FR-200, 300 | 6.2125.020 |
| Types FX-200, 300, 320 | 6.2125.020 |
| with RS232 interface (OP-03) | |
| Precisa Balances with RS232 interface | 6.2125.080 |

The balance type must be preselected on the Coulometer with the <CONFIG> key. Balance and printer can be attached simultaneously using the 6.2125.030 Socket-Outlet Adapter. The balance must then be plugged into the "data in" receptacle of the socket-outlet adapter.

The weight must be sent by the balance in g. The Coulometer automatically converts the value into mg. A special input unit supplied by the balance manufacturer can be used for entry of not only the weight but also the sample identifications by the balance. The addresses of the identification may possibly have to be preselected on the input unit.

| Balance | Id#1 | Id#2 | Id#3 |
|--------------|------------|------------|------------|
| Sartorius | ID.1 or 26 | ID.2 or 24 | C-20 or 23 |
| Mettler (AT) | C (ID#1) | B (ID#2) | A (c20) |

6.4 Connection of the 707 KF Oven

The oven is connected as follows:

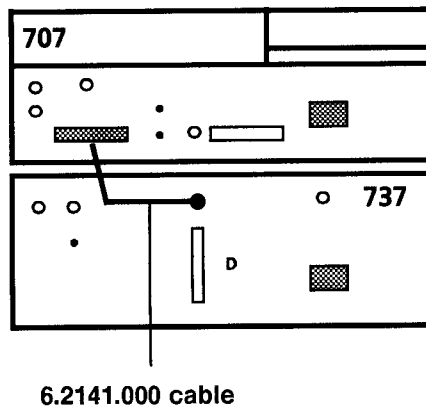


Fig. 6-2: Connection of the 707 KF Oven

For work with the 707 KF Oven, the work stand must be mounted on the left of the instrument.

Setting up the instruments:

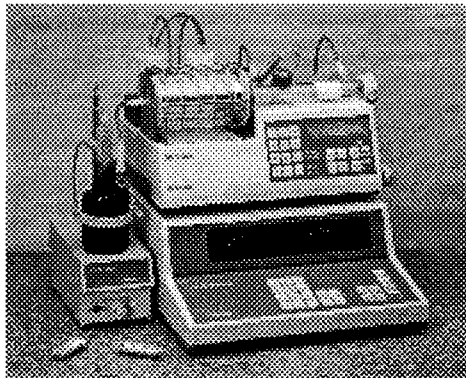


Fig. 6-3 a: Measuring cell without diaphragm

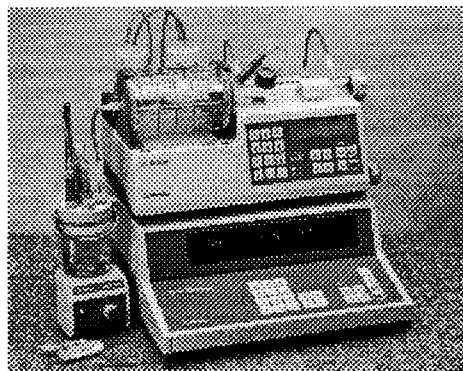


Fig. 6-3 b: Measuring cell with diaphragm

Fig. 6-3: Setting up the Coulometer with 707 KF Oven

6.5 Connection of a computer

The computer is connected as follows:

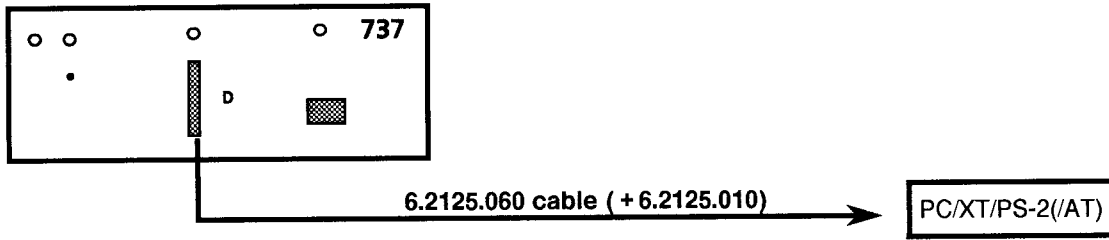


Fig. 6-4: Connection of a computer

For the attachment of IBM® AT computers, the 6.2125.010 Adapter is also needed.

Preselection on Coulometer:

RS232 settings: depending on control program of the computer

Program package for the data transfer Coulometer↔computer,
3½" Diskette

6.6020.000

7. Appendix

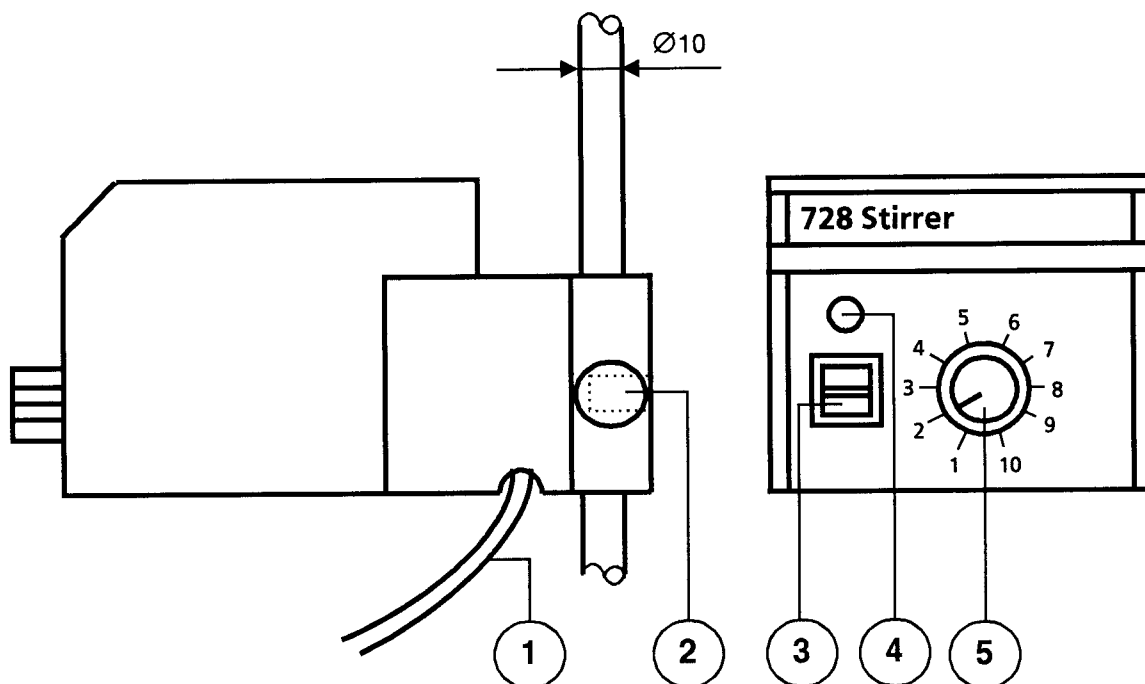
7.1 Technical specifications

| | |
|---|--|
| End point indication | Voltametric |
| Titration speed | Max. 2 mg/min, slower in the vicinity of the end point |
| Determination range | |
| Instrument range | 1 ... 65 535 µg water |
| Usual in practice | 10 ... 10 000 µg water |
| Resolution | |
| $m(\text{H}_2\text{O}) \leq 99,9 \mu\text{g}$ | 0,1 µg |
| $m(\text{H}_2\text{O}) \geq 100 \mu\text{g}$ | 1 µg |
| Measurement error | |
| $m(\text{H}_2\text{O}) \leq 1000 \mu\text{g}$ | ≤ ca. 5 µg |
| $m(\text{H}_2\text{O}) \geq 1000 \mu\text{g}$ | ≤ ca. ± 0,5% |
| Materials | |
| Housing | Light metal injection moulding, multicoat stove enamel |
| Keypad covers | Polycarbonate (PC) |
| Display | Vacuum fluorescent display, 16 characters |
| Character height | 17 mm |
| RS232 interface | For printer and balance connection or computer connection: full remote control by external control device |
| "Remote" socket | For oven or sample changer connection |
| Input signals | Start |
| Output signals | Ready/conditioning OK |
| Ambient temperature | |
| Nominal operational range | 5 ... 40 °C |
| Storage, transport | - 20 ... 60 °C |
| Safety specifications | Construction and testing in accordance with IEC publication 1010, protection class I. These Instructions for Use contain information and warnings which must be heeded and complied with by the user to ensure safe operation of the instrument. |
| Mains connection | |
| Voltage | 100, 117, 220, 240 V ± 10%, switchable; for 230 V set to 240 V |
| Frequency | 50 or 60 Hz |
| Power consumption | ca. 40 VA |
| Fuses | 0,4 A (slow-blow) for 100 and 117 V 0,2 A (slow-blow) for 220 and 240 V |
| Dimensions w × h × d | 570 mm × 290 mm × 520 mm |
| Weight | ca. 8,5 kg |

7.2 Pin assignment of the "Remote" socket

| | | external |
|---|---|--|
| <p>Start</p> <p>Signal must be applied for min. 1.1 s and be reset at the latest 1 s before end of titration.</p> | <p>low level: $U < 0.2 \text{ V}$ $I < 0.6 \text{ mA}$</p> | <p>2 Start</p> |
| <p>Start Ground</p> <p>Ready +, Ready -</p> <p>Active when conditioning OK, no titration started. Not active when conditioning not OK or titration running. With preselection PERIPH."SAMPLER" (<CONFIG> key), the active signal is set inactive once every second.</p> | <p>Active: $I_c = 12 \text{ mA}$ $U_{CE} < 1.5 \text{ V}$</p> <p>Not active: $I_c < 10 \mu\text{A}$ $U_{CE} \text{ max. } 30 \text{ V}$</p> | <p>1 Start Ground</p> <p>4 Ready + 5 Ready -</p> |
| <p>No liability whatsoever will be accepted for damage arising from improper interconnections of devices.</p> | | |

7.3 Instructions for Use for 728 Magnetic Stirrer



- (1) Connecting cable
- (2) Fastening screw
- (3) On/off switch
- (4) Pilot lamp, supply
- (5) Regulation of stirring speed

Fastening The stirrer is mounted on a stand rod $\varnothing = 10$ mm. It is fixed at the appropriate height using screw (2) so that it can be swung out to the left or right from the working position.

Power supply $U_{DC} \approx 8$ V

Stirring speed stabilised, $n \approx 200 \dots 1900$ min⁻¹ (without load)

Stirring bar PTFE coating, magnetic core

| | Length | Dimensions | Shape |
|------------|--------|----------------------|-------|
| 6.1903.000 | 8 mm | $\varnothing = 4$ mm | ○ |
| 6.1903.010 | 12 mm | $\varnothing = 4$ mm | ○ |
| 6.1903.020 | 16 mm | $\varnothing = 4$ mm | ○ |
| 6.1903.030 | 25 mm | $\varnothing = 5$ mm | ○ |
| 6.1906.000 | 42 mm | - | △ |
| 6.1906.010 | 25 mm | - | △ |
| 6.1906.020 | 26 mm | - | oval |

7.4 Warranty

The warranty regarding our products is limited to rectification free of charge in our workshops of defects that can be proved to be due to material, design or manufacturing faults which appear within 12 months from the day of delivery. Transport costs are chargeable to the purchaser.

For day and night operation, the warranty is valid for 6 months.

Glass breakage in the case of electrodes or other glass parts is not covered by the warranty. Checks which are not a result of material or manufacturing faults are also charged during the warranty period. For parts of outside manufacture insofar as these constitute an appreciable part of our instrument, the warranty stipulations of the manufacturer in question apply.

With regard to the guarantee of accuracy, the technical specifications in the Instructions for Use are authoritative.

Concerning defects in material, construction or design as well as the absence of guaranteed features, the purchaser has no rights or claims except those mentioned above.

If damage of the packaging is evident on receipt of a consignment or if the goods show signs of transport damage after unpacking, the carrier must be informed immediately and a written damage report demanded. Lack of an official damage report releases METROHM from any liability to pay compensation.

If any instruments and parts have to be returned, the original packaging should be used if at all possible. This applies above all to instruments, electrodes, burette cylinders and PTFE pistons. Before embedment in wood shavings or similar material, the parts must be packed in a dustproof package (for instruments, use of a plastic bag is imperative). If open assemblies are enclosed in the scope of delivery that are sensitive to electromagnetic voltages (e.g. data interfaces etc.) these must be returned in the associated original protective packaging (e.g. conductive protective bag). (Exception: assemblies with built-in voltage source belong in a non-conductive protective packaging). For damage which arises as a result of non-compliance with these instructions, no warranty responsibility whatsoever will be accepted by METROHM.

7.5 Standard equipment and ordering designations

7.5.1 737 KF Coulometer

KF Coulometer with **measuring cell without diaphragm**, 50 Hz **2.737.0112**
including the following accessories:

| | | |
|---|--|------------|
| 1 | Double Pt electrode with SGJ 14/15 | 6.0341.100 |
| 1 | Generator electrode with SGJ 29/22 | 6.0342.100 |
| 1 | Drying tube with SGJ 19/17 | 6.1403.030 |
| 1 | Glass stopper with SGJ 14/15 | 6.1437.000 |
| 1 | Dispensing/aspirating tube with SGJ 14/15 | 6.1439.010 |
| 1 | Stopper SGJ 14/M10 for work with the KF Oven | 6.1446.060 |
| 2 | Threaded stoppers M8 for work with a pump | 6.1446.080 |
| 2 | Sets of septa (5 per set) | 6.1448.020 |
| 1 | Titration vessel, 200 mL, brown glass with SGJ | 6.1455.313 |
| 1 | Tubing connection M6, 10.5 cm, for work with the KF Oven | 6.1805.070 |
| 1 | Tubing connection M8, 50 cm, for work with a pump | 6.1805.200 |
| 1 | Stirring bar, 25 mm | 6.1903.030 |
| 1 | Titration vessel holder | 6.2047.000 |
| 1 | Electrode cable, connector F, 1 m, for indicator electrode | 6.2104.020 |
| 1 | Electrode cable, connector H, 1 m, for generator electrode | 6.2104.120 |
| 1 | Screw cap | 6.2701.040 |
| 3 | PTFE ground joint sleeves SGJ 14 | 6.2713.000 |
| 1 | PTFE ground joint sleeve SGJ 29 | 6.2713.010 |
| 1 | PTFE ground joint sleeve SGJ 19 | 6.2713.020 |
| 1 | Stopper with nipple M10, nipple for work with the KF Oven | 6.2730.030 |
| 1 | Funnel | 6.2738.000 |
| 1 | Bottle molecular sieves, 250 g | 6.2811.000 |
| 1 | Syringe, 1 mL | 6.2816.000 |
| 1 | Needle for syringe | 6.2816.010 |
| 1 | Mains cable, cable plug to customer's specifications | |
| | Type SEV 12 (Switzerland ...) | 6.2122.010 |
| | Type CEE(7),VII (Germany...) | 6.2122.030 |
| | Type NEMA/ASA (USA...) | 6.2122.060 |
| 1 | Dust cover | 6.2723.250 |
| 1 | Set Instructions for Use for 737 KF Coulometer | 8.737.1003 |

KF Coulometer with **diaphragm-free measuring cell**, 60 Hz **2.737.0113**
Accessories as above.

| | |
|---|-------------------|
| KF Coulometer with measuring cell with diaphragm , 50 Hz including the following accessories: | 2.737.0012 |
| 1 Magnetic stirrer | 1.728.0010 |
| 1 Double Pt electrode | 6.0338.100 |
| 1 Generator electrode | 6.0339.000 |
| 1 Drying tube | 6.1403.030 |
| 1 Titration vessel upper half | 6.1414.040 |
| 1 Titration vessel lower half, 200 mL | 6.1415.310 |
| 1 Threaded stopper for closing the gas inlet tube | 6.1446.040 |
| 2 Sets septa (5 per set) | 6.1448.010 |
| 1 Set seals for titration vessel upper half | 6.1454.000 |
| 1 Gas inlet tube | 6.1617.010 |
| 1 PTFE tubing connection, 10.5 cm | 6.1805.070 |
| 1 Stirring bar, 16 mm | 6.1903.020 |
| 1 Stirring bar, 25 mm | 6.1903.030 |
| 1 Stand support | 6.2001.040 |
| 1 Clamping ring, 10 cm | 6.2013.010 |
| 1 Stand rod, 25 cm | 6.2016.030 |
| 1 Electrode cable, connector F, 1 m, for indicator electrode | 6.2104.020 |
| 1 Extension cable for magnetic stirrer | 6.2108.100 |
| 1 PTFE ground joint sleeve, SGJ 19 | 6.2713.020 |
| 3 Screw nipples | 6.2730.010 |
| 1 Funnel | 6.2738.000 |
| 1 Bottle molecular sieves, 250 g | 6.2811.000 |
| 1 Syringe, 1 m | 6.2816.000 |
| 1 Needle for syringe | 6.2816.010 |
| 1 Mains cable, cable plug to customer's specifications | |
| Type SEV 12 (Switzerland...) | 6.2122.010 |
| Type CEE(7), VII (Germany...) | 6.2122.030 |
| Type NEMA/ASA (USA...) | 6.2122.060 |
| 1 Dust cover | 6.2723.250 |
| 1 Set Instructions for Use | 8.737.1003 |

| | |
|--|-------------------|
| KF Coulometer with measuring cell with diaphragm , 60 Hz Accessories as above. | 2.737.0013 |
|--|-------------------|

7.5.2 Options

| | |
|---|------------|
| Ti Stand ; compact titration stand with built-in stirrer and pump for the addition of fresh solution and for siphoning off waste solution | 2.703.0010 |
|---|------------|

Measuring cell

| | |
|--|------------|
| Measuring cell without diaphragm, complete | 6.5405.000 |
| Sealing ring for measuring cell with diaphragm | 6.1454.030 |
| Magnetic stirring bar, length | |
| 12 mm | 6.1903.010 |
| 16 mm | 6.1903.020 |
| 25 mm | 6.1903.030 |

KF Oven

| | |
|--|------------|
| 707 KF Oven with automatic drive of the weighing boat and built-in air pump | 2.707.0010 |
| Connecting cable | 6.2141.000 |
| 688 KF oven | |
| Mains with 220...240 V | 2.688.0014 |
| Mains with 100...120 V | 2.688.0015 |

Automatic sample metering and addition

| | |
|---|------------|
| 708 Sampling Unit for sample metering | 2.708.0010 |
| 698 Autosampler, sample changer | 2.698.0010 |
| Cable 737 KF Coulometer – 698 Autosampler | 6.2128.050 |
| Cable 708 Sampling Unit – 698 Autosampler | 6.2128.040 |

Printers

| | |
|--|------------|
| Citizen printer iDP560 RS | 2.140.0014 |
| Cable 737 KF Coulometer – Citizen printer iDP560 RS | 6.2125.050 |
| Seiko printer DPU-411, 220 V | 2.141.0014 |
| Seiko printer DPU-411, 120 V | 2.141.0015 |
| Cable 737 KF Coulometer – Seiko printer DPU-411 | 6.2125.020 |
| Cable 737 KF Coulometer – EPSON printer with 6-pin round connector | 6.2125.040 |
| Cable 737 KF Coulometer – EPSON printer with RS interface #8148 | 6.2125.050 |
| Cable 737 KF Coulometer – HP Think Jet, HP Desk Jet | 6.2125.050 |
| Cable 737 KF Coulometer – Kodak Diconix 180 si | 6.2125.050 |
| Socket-outlet adapter for simultaneous attachment of a balance | 6.2125.030 |

Balance connection

| | |
|--|---------------------|
| Sartorius balances MP8, connecting cable | 6.2125.070 |
| Mettler AT, PM balances and balances with interface 016 | Cables from Mettler |
| Mettler balances with interface 011 or 012 | 6.2125.020 |
| AND balances (with RS232 interface OP-3), connecting cable | 6.2125.020 |
| Precisa balances, connecting cable | 6.2125.080 |
| Socket-outlet adapter for simultaneous attachment of a printer | 6.2125.030 |

Computer connection, control via RS232 C interface

| | |
|--|-------------------------|
| Cable 737 KF Coulometer – IBM® PC/XT/PS-2 or compatibles | 6.2125.060 |
| Cable 737 KF Coulometer – IBM® AT | 6.2125.060 + 6.2125.010 |
| RS232 C extension cable | 6.2125.020 |
| Program package for data transfer 737 KF Coulometer – IBM® PC or compatibles, 3½" diskette | 6.6020.000 |

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EC Declaration of Conformity

The METROHM AG company, Herisau, Switzerland hereby certifies, that the instrument:

737 KF Coulometer

meets the requirements of EC Directives 89/336/EWG and 73/23/EWG.

Source of the specifications:

- EN 50081-1 Electromagnetic compatibility, basic specification
Emitted Interference
- EN 50082-1 Electromagnetic compatibility, basic specification
Interference Immunity
- EN 61010 Safety requirements for electrical laboratory measurement
and control equipment

Description of the instrument:

Instrument for coulometric water determinations according to Karl Fischer.

Herisau, December 6, 1995

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