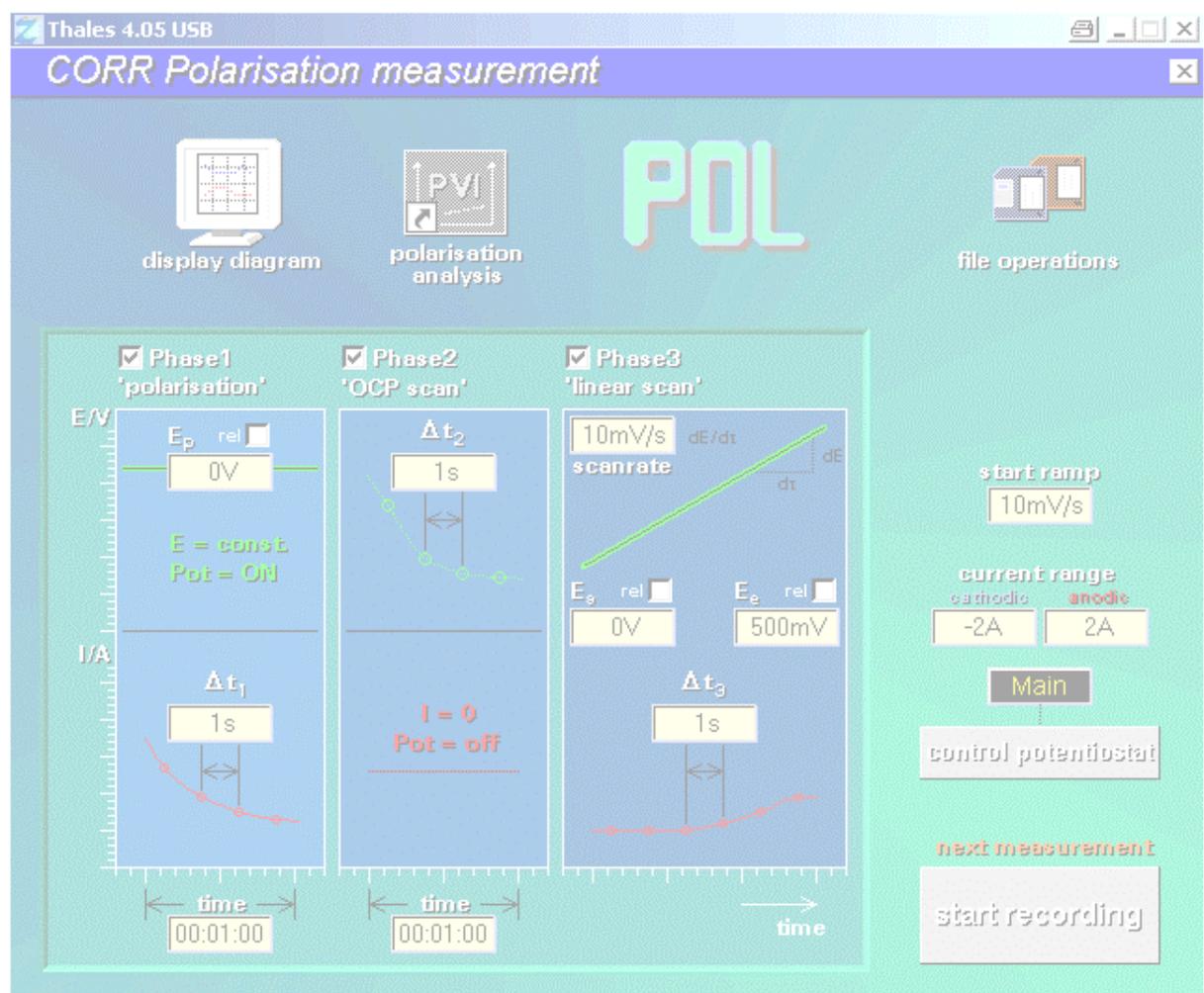


POL

Polarization Measurements



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Introduction

A polarization measurement (PM) consists of three major phases:

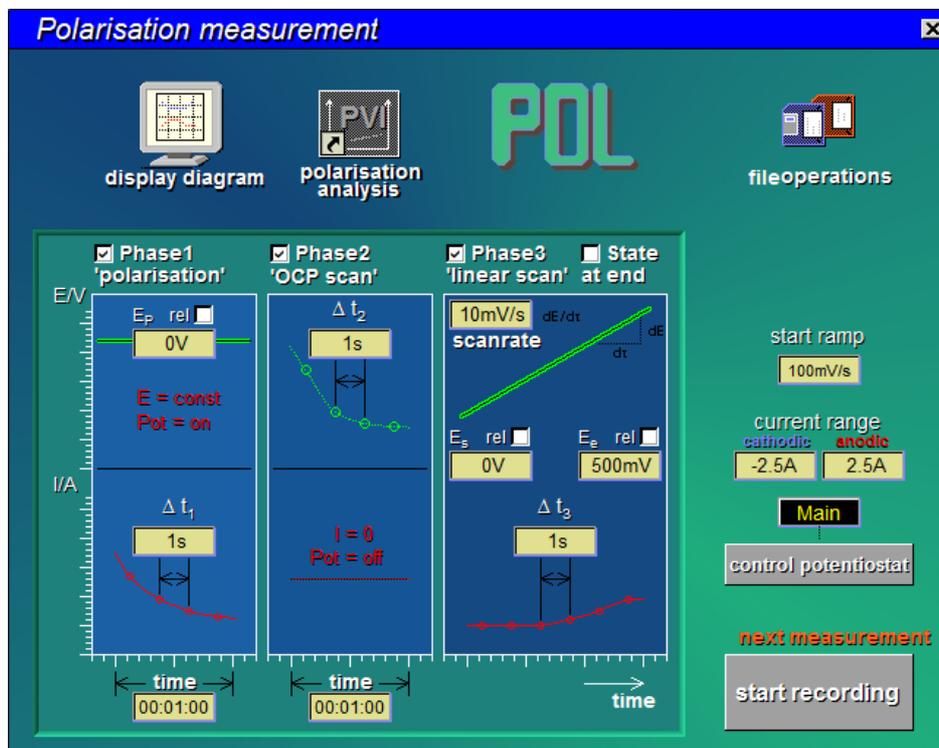
1. Polarization phase (set potential and measure current vs. time) -> Stress object
2. Rest phase (measure open circuit voltage vs. time) -> Relax object
3. current-to-voltage curve (set linear voltage ramp and measure current) -> I/E scan

Each phase can be selected separately and makes POL a versatile measurement tool. With POL the following standard methods can be done easily:

- Polarization Measurement
- Chronoamperometry
- Potentiometry
- Chronopotentiometry
- Cyclic Voltammetry (I/E) with Autoscaling Current

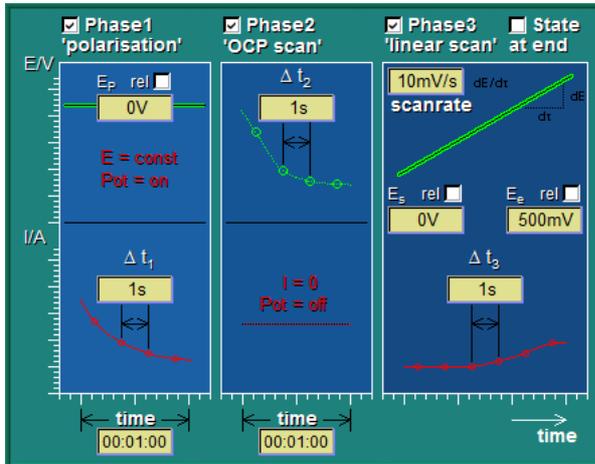
1. Recording Main Menu

POL starts with a graphical menu including a set of buttons and parameter input fields that can be activated by the user.



The functions can easily be recognized because of their titles and symbols which are plotted within the button. The main menu manages a group of sub-functions, which will be described briefly.

1.1 Edit Parameters



setting of measure phases

- polarization
- OCP scan
- linear scan

and their parameters

1.2 Control Potentiostat

control potentiostat

calls up the 'test sampling' panel, allowing the manual setting of the actual state of the ECW. For POL, the potentiostat has to be in the potentiostatic mode. An automatic starting ramp is encountered prior to the POL, if the actual set-potential of the potentiostat differs from the POL starting potential, or if the potentiostat is in the off-state. The ramp will then begin at the actual set-potential or at the actual OCP respectively.

1.3 Start Recording

next measurement

start recording

starts the POL measurement under the condition displayed on the POL control panel.

1.4 Display Diagram



display diagram

displays last POL measurement (no display, if no measurement was already performed or opened as data file from the storage).

1.5 File Operations



data file operations of POL data (save actual and load previous measurement, select i/o device and data path). Loading of a previous measurement will configure the ECW automatically according to the settings of this measurement.

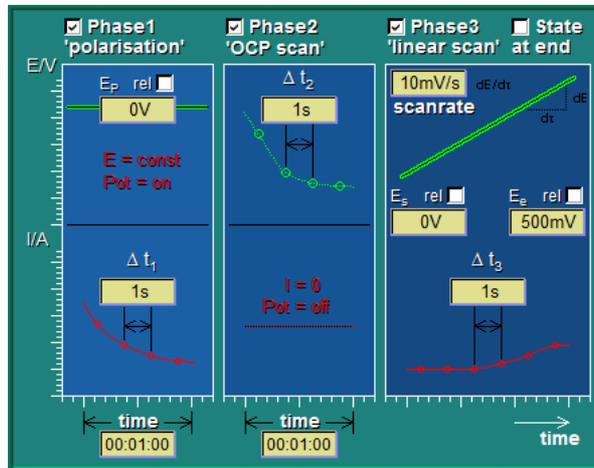
1.6 Dynamic CV - Analysis



calls PVI evaluation program for POL data.

2. Edit Parameters

The actual control parameters in the POL control panel are indicated in the main box providing all three different measurement phases. See below.



Overview of necessary control information:

Parameter	Name	Unit	Range
P_{Pol}	Polarization	V	+ -4 V (abs/rel)
T_{Pol}	Polarization time	s	0 or 10 – 36000 s
T_{SPo}	Polarization sampling time	s	0.5 – 60 s
T_{Re}	Time at the rest potential	s	0 or 10 – 36000 s
T_{SRe}	Rest sampling time	s	0.5 – 60 s
P_{IEs}	Starting potential	V	+ -4 V (abs/rel)
P_{IEe}	End potential	V	+ -4 V (abs/rel)
V_{IE}	Slew rate	mV/s	0.1 – 5 mV/s
T_{sIE}	Scan sampling time	s	0.5 – 10 s
I_{max}	Maximum current	A	1 μ A – 3 A (abs)

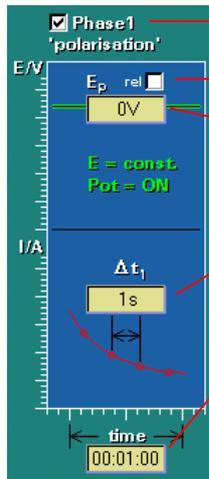
These parameters can directly be modified.



Additional notes:

Potential settings may be absolute, when there is no specification or the abbreviation "abs" following the numeric value. But they can also refer to the actual rest potential, when the specification "rel" is following the numeric value. The rest potential is being updated during all phases, with the potentiostat switched off. After the measurement has been finished the starting conditions of the potentiostat are restored. The potentiostat will remain switched off if there is an interruption. The reference potential in the menu "potential" should have been preset to P_{pol} already and the starting potential P_{IEs} should be zero relative to the rest potential in order to avoid delay times during the starting phases.

2.1 Polarization Phase P_o (Phase 1)



- ▶ Polarization phase is enable when the box 'Phase 1' is checked.
- ▶ Polarization potential is relative to OCP when box 'rel' is checked.
- ▶ Enter here the relative/absolute polarization potential.
- ▶ Time resolution of polarization measurement.
- ▶ Duration of polarization phase entered in [hh:mm:ss].

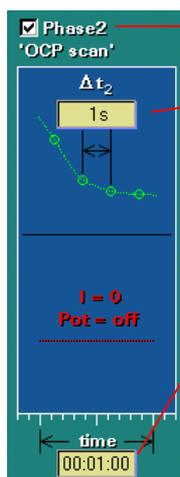
Starting phase P_{os}

The starting phase will be used to avoid high transient currents if the potential dominating in the beginning (P_s) is unequal to the polarization potential (P_{pol}). P_s will be the dominating rest potential if the potentiostat is switched off in the beginning; otherwise P_s will be the reference/ideal potential. The starting phase P_{os} is shown by a message window and describes the transition from P_s to P_{pol} with a constant, but not exactly specified voltage slew rate (about 250 mV/s). The current during this procedure is checked, tolerating twice the value of the maximum current I_{max} . There is no registration during this phase. If P_{os} is switched off as a result of current overrun or after pressing the <POS1> key, no data will be saved.

Main phase P_o

During the main phase the potentiostat is on, the potential P_o is steady and the current I_{po} through the cell is controlled and registered optionally. Here, the maximum current I_{max} is tolerated. Data will be lost if P_{os} is cut off as a result of current overrun or if the <POS1> key is pressed.

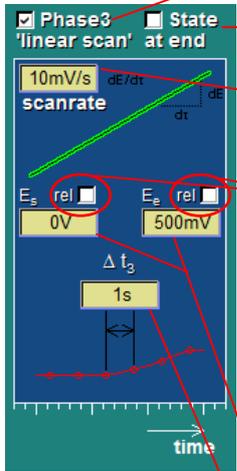
2.2 Rest Phase (Phase 2)



- ▶ Rest phase is enable when the box 'Phase 2' is checked.
- ▶ Time resolution of rest potential measurement.
- ▶ Duration of rest phase entered in [hh:mm:ss].

During the rest phase the potentiostat is turned off and the rest potential P_{re} is controlled and registered optionally.

2.3 Current-to-Voltage Curve IE (Phase 3)



Linear IE scan is enable when the box 'Phase 3' is checked.

Hold the end potential when the box 'State at end' is checked. If unchecked the potentiostat is switched off.

The ramps are driven by the defined slew rate and the data is registered in steps of the actual resolution of the potential.

The start and end potential can be defined in two ways:
 a) absolute potentials reference $E_o=0V$
 b) relative potentials reference $E_o=OC$ (open circuit potential)

The selected mode is indicated by a blue tick within the checkbox. It can also be indicated during input by means of appending the corresponding abbreviation (rel) or (abs) to the actual value.

The relative mode function offers the possibility to investigate different probes in a defined potential range relative to the open circuit potential (ocp). The ocp is set to the actual value through a change to the submenu 'test sampling' of the EIS impedance measurement program. In addition, the potentiostat has to be switched off. There is no ocp control during an I/E recording.

Start potential E_s and end potential E_e of the linear IE scan.

Time resolution defines the step width of the measured I/E data.

With this part of the measurement, the standard current-to-voltage curve is used as a subroutine. That is why the settings in the I/E menus (e.g., changes in the potential) affect PM measurements and vice versa.

Starting phase IE_s

The starting phase will help to avoid high transient currents if the potential P_b dominating in the beginning is unequal to P_{IEs} . P_b will be the rest potential if the potentiostat is switched off at that time (with the P_o phase lacking) or if the standby phase R_e has been run through. If not, P_s will be the reference potential (with the P_o phase lacking) or the polarization potential P_{pol} . The starting phase IE_s is indicated by a running numerical display of the measurement data, with the current-to-voltage graphic being inactive. The UI_s phase consists of a transition from P_b to P_{IEs} with a constant, but not exactly specified voltage slew rate (about 250 mV/s). The current during this procedure is checked, tolerating twice the value of the maximum current I_{max} . There is no registration during this phase. If P_{os} is switched off as a result of current overrun or after pressing the <POS1> key, no data will be saved.

Main phase IE

The potentiostat has been activated and the potential P_{IE} is changed from the starting potential P_{IEs} to the end potential P_{IEe} with a constant and defined voltage slew rate V_{IE} . The current through the cell is both controlled and registered with auto ranging active. Potential as well as current are integrated between the samples, thus automatically defining a Nyquist filter by defining the scan rate ($1/T_{SIE}$). The current is checked and the maximum current I_{max} is tolerated. If IE is switched off as a result of current overrun or after pressing the <POS1> key, no data will be saved.

2.4 Current Range



The anodic as well as the cathodic current range can be selected independently. The exceeding of one of the limits would cause the actual ramp to be interrupted and the corresponding end potential to be corrected. Finally, the next ramp will be started with respect to the corrected edge potentials.

2.5 Start Ramp



Slew rate of start phase. Start ramp from actual potential to next set potential.

3. Control Potentiostat

control potentiostat

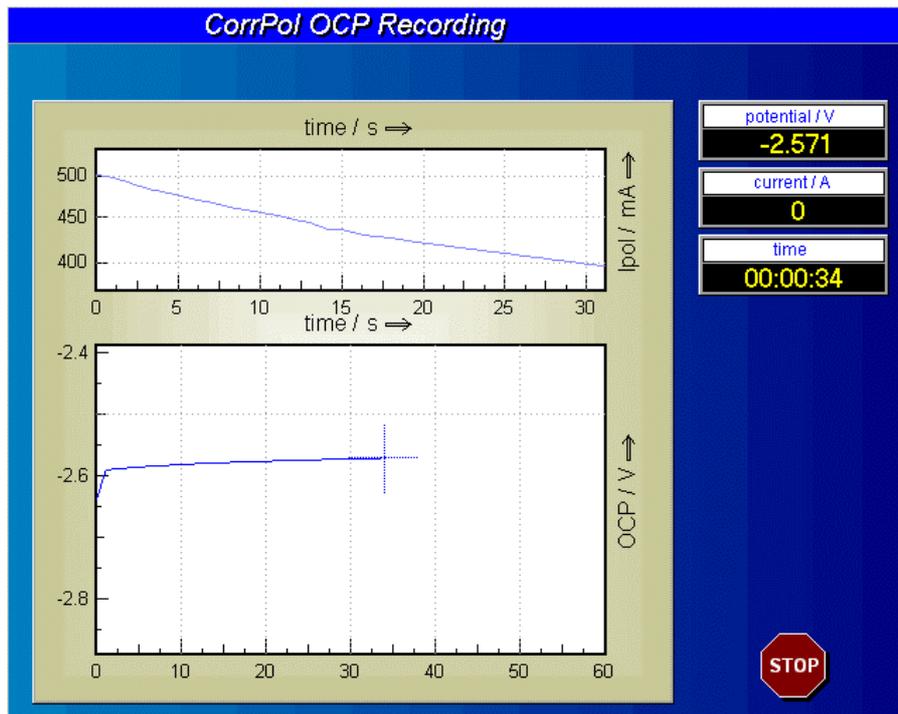
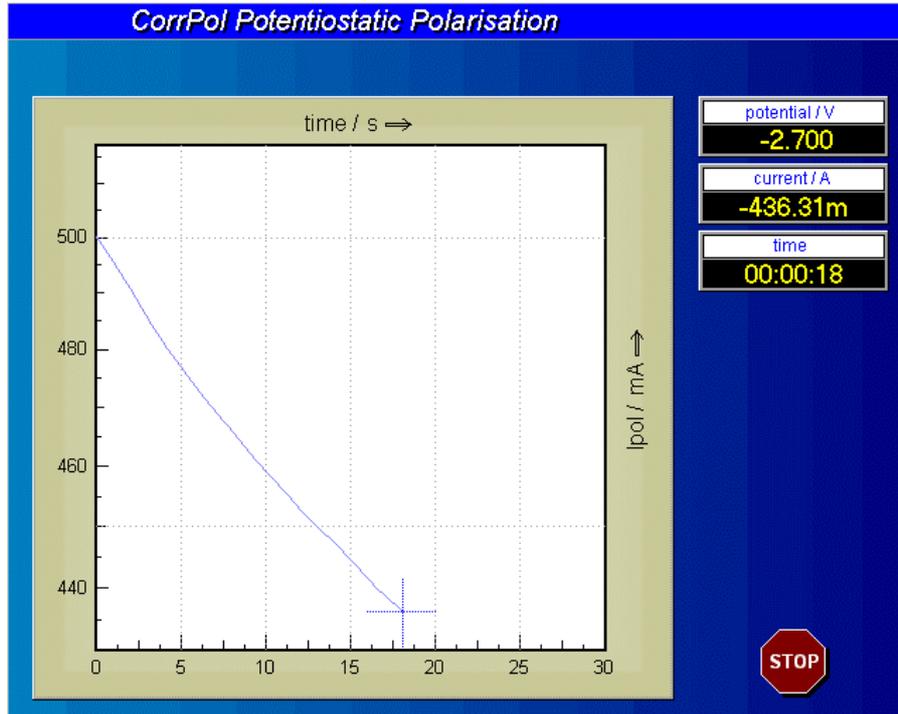
This option enters the potentiostat control panel of the ECW. The state of the potentiostat is set there. Here the potentiostat can be set to the CV starting potential and switched on manually, what will avoid the automatic insertion of a start ramp between the actual potential and the CV starting potential. The ramp speed may be altered in the general measurement set-up under the point "DC-more".

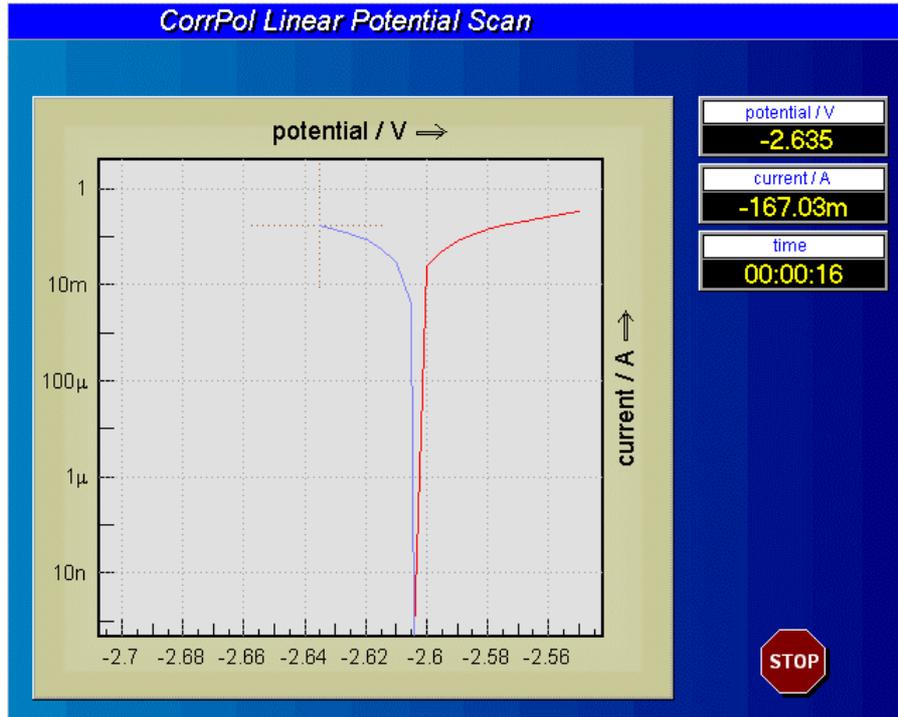
Also the final condition of the potentiostat can be selected here. If a CV measurement is started with the potentiostat switched on, the potentiostat will remain operational after the CV measurement has been completed. If the potentiostat has been switched off before starting a CV measurement it will be switched off at the end of the measurement.

4. Next Measurement



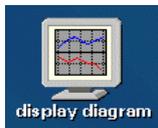
After all parameters have been set properly, a new measurement can be started by activating the button <start recording>. If a new measurement is started, the program will prompt the recording windows (phase 1...3) shown below.



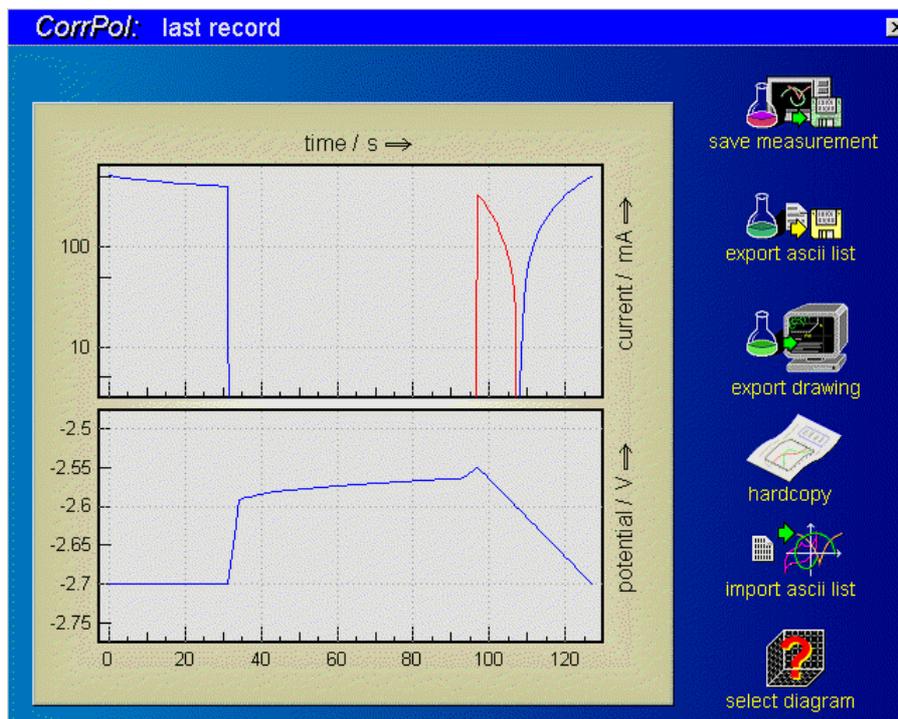


The measured U-I data is plotted online in the data window. At the upper right of the recording window three 'digital' instruments indicate the numeric values of the actually measured data triplett <U,I,t>. A running measurement can be terminated by using the <ESCAPE> function or by pressing the stop-button.

5. Display Diagram



The 'display diagram' function will be entered automatically after completion of a POL, and it may be recalled from the POL control parameter as well as by means of the central pull-down menu, if a the measurement is present. That spectrum will be displayed on the screen. Data can be exported to other programs by the standard i/o functions.



A graphic displays the polarization phase online at the upper part of the window. The measured current is shown on the logarithmic Y-axis; the dedicated time is displayed linearly on the X-axis. Anodic power is marked red, cathodic blue. The refresh rate is 1/s, independent of the measuring rate. If the polarization time is set to zero, no graphic will be generated.

The rest phase is displayed online as a graphic at the lower part of the window. The measured potential and the dedicated time is shown on the Y-axis and linearly on the X-axis respectively. The refresh rate is 1/s, independent of the measuring rate. No graphic will be generated if the rest time is set to zero.

The current-to-voltage curve is displayed as a usual XY-plot. This graphic still remains visible after the measurement has been finished. Then, the functions of the current-to-voltage-end-of-measurement-menus are available.

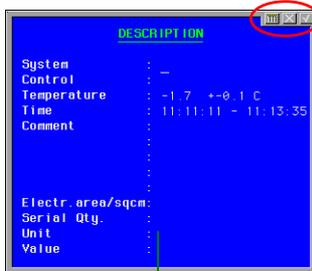
5.1 Save Measurement

The complete data record can not be handled as a standard current-to-voltage curve, because the polarization measurement consists of complete triples of potential, current and time (analogue to I/E measurements of the IE phase) as well as incomplete triples (zero current of the rest phase). This does not apply for the IE phase, where data can be stored and reported as usual. The functions of the IE menu can be applied to the current-to-voltage curve.

The complete record of all three measuring phases can be saved with the "save" function of the PM file menu as a PVI measuring curve. For this set of data, all functions of the PVI program are available, for example, the generation of a data list of the complete measurement record. In the IE menu, however, only the IE phase is accessible.

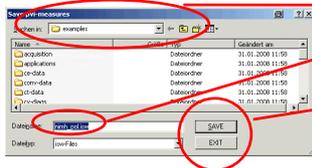


Save the recorded data + the measurement parameters + the comments to the hard disk and return to the *POL* page.



When clicking on either of the buttons a **description** box opens where you may input your measurement parameters and comments. Some lines will be filled automatically by the software (e.g. potential, current and measuring time) others are free for the user to fill. User parameters are not used by the software for any calculation. They are for the user's information only.

Accept the inputs by clicking on the button in the upper right corner or reject the inputs by clicking on the button. Click on the button to call the calculator.



In the following browser navigate to the desired path, input a file name and click on the button to save the data or click on the button to cancel the saving.

Note: It is recommended to use one of the save-functions in order to store (and recall later) the ECW state corresponding with that measurement.

5.2 Create Data List



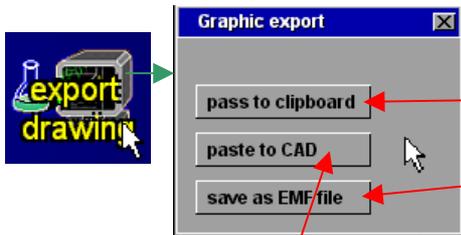
Creates an ASCII list of the data:

and copies it to the Windows® clipboard

or saves it as plain text-file

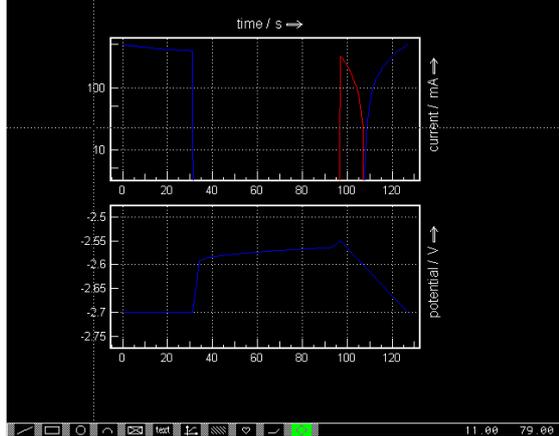
or copies it to **ZEdit**

5.3 Export Drawing



Passes the actual graph as high quality vector graphic to the Windows clipboard

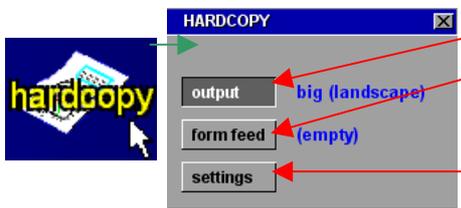
saves the graph as an EMF graphic file

Transfers the graph to the CAD graphic editor section of Thales.

Edit the graph manually in the CAD section of Thales for later clipboard or EMF export.

5.4 Hardcopy



Create a bitmap copy of the screen for export or printing:
Perform the selected output operation

Do a form feed

Select the output properties:
landscape = big format (DIN-A4) to printer
upright = small format (DIN-A5) to printer
to clipboard = sends screen to the Windows clipboard
save as file = saves the screen content as a bitmap file

5.5 Import Data List



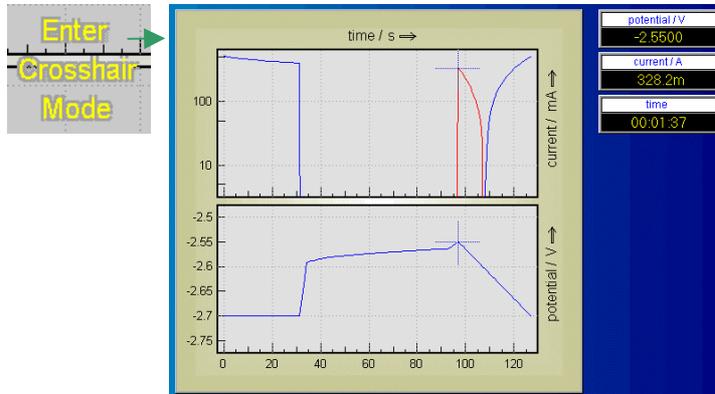
- inactive within POL -

5.6 Select Diagram



- inactive within POL -

5.7 Enter Crosshair Mode



Move cursors through the curves with the mouse. The course leads through the history of the data source: moving from left to right means moving from the first samples recorded to later ones.

Use the cursor-left/right keys for fine steps. You leave the crosshair mode by clicking the middle mouse key or the <ESCAPE> button. If more than one data set is displayed, use the cursor-up/down keys to change from one curve to the next.

6. File Operations



The 'file operation' function will recall the last record or save the actual one. This function works like in all file menus within Thales.

6.1 Open



This function allows you to load/save POL data from/to the storage of your PC. Click on the icon and decide in the submenu, whether you want to open previously stored POL data or save actual POL data.

If there are no data in memory, this sub-menu is skipped.

After your decide to **Open**, a file browser will open displaying only CV data files (file extension **.isw**).



Select a path and a file and click on the **LOAD** button to load the file to the *Thales* software. Click on the **Display Diagram** icon on the POL main page to display the data in a graphical form.



The next box shows the measurement parameters of the loaded data. Click on it if you want to close it.

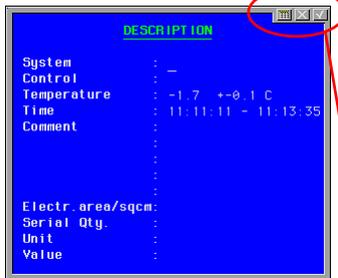
NOTE: Opening a file will switch off the potentiostat and configures the potentiostat according to the loaded measurement-settings automatically. Loading a measurement file is an easy way to perform a new measurement with previously used settings.

6.2 Save

The complete data record can not be handled as a standard current-to-voltage curve, because the polarization measurement consists of complete triples of potential, current and time (analogue to I/E measurements of the IE phase) as well as incomplete triples (zero current of the rest phase). This does not apply for the IE phase, where data can be stored and reported as usual. The functions of the IE menu can be applied to the current-to-voltage curve.

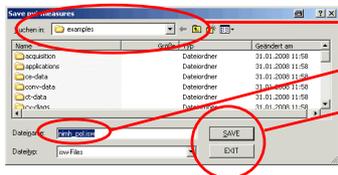
The complete record of all three measuring phases can be saved with the "save" function of the PM file menu as a PVI measuring curve. For this set of data, all functions of the PVI program are available, for example, the generation of a data list of the complete measurement record. In the IE menu, however, only the IE phase is accessible.

If there are data in memory, usually after an POL data measurement, you may want for **Save** them as data file on storage.



A **description** box opens where you may input your measurement parameters and comments. Some lines will be filled automatically by the software (e.g. potential, current and measuring time) others are free for the user to fill and are not used for any calculation. The last line is reserved to put in the area value of the working electrode under test in order to enable area relative calculations.

Accept the inputs by clicking on the button in the upper right corner or reject the inputs by clicking on the button. Click on the button to call the calculator.

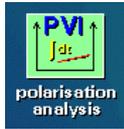


In the following browser navigate to the desired path, Input a file name and

Click on the button to save the data.

Click on the button to cancel the saving.

7. Polarization Analysis



POL data has to be analyzed by PVI-Analysis. For more information see PVI manual.