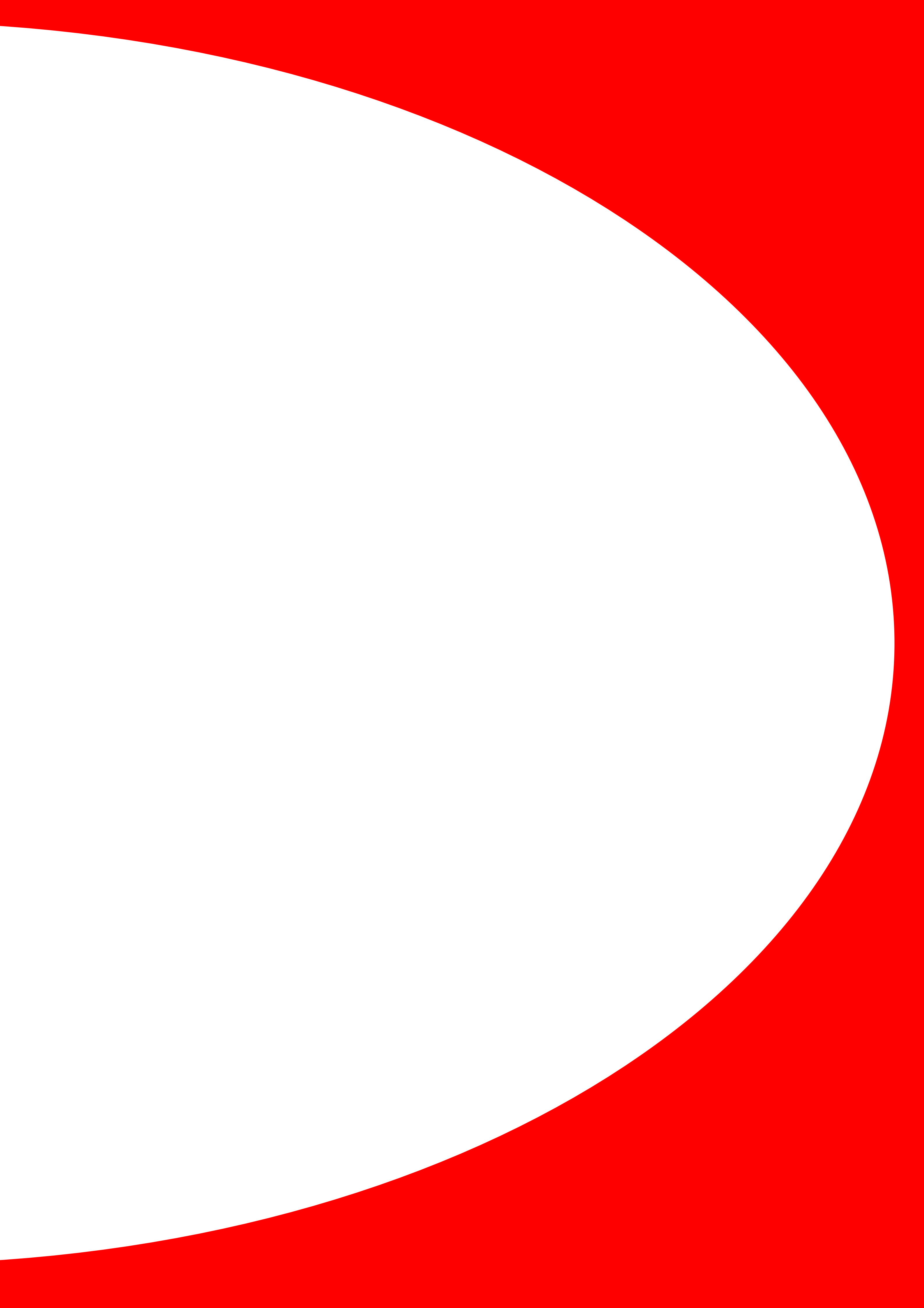


# Installation

## Instrument

If possible, flat pack and store the container the instrument arrived in, this will facilitate the return of instrument should it be necessary. After the instrument has been unpacked check the mains voltage selector switch, it should be set to the correct voltage setting for the mains voltage in your country.

A serial cable and 25 to 9 way adapter † is supplied with the instrument, connect this cable to any free speed serial port, using the adapter if required.

† 25 to 9 way not applicable on Field Machine

Although the instrument is heavily shielded it is advisable to keep the instrument, cables and test cell clear of any strong electromagnetic sources. Mains noise can affect test results especially when using high impedance cells. To obtain the best results shield the test cell using a Faraday cage connected to green instrument earth connector on the back of your instrument.

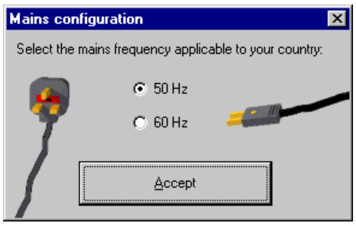
## Software

If a computer was supplied with the instrument, the software will already be installed so this section can be skipped.

Before installing the software, quit any programs that are running. Insert the installation CD-ROM into the CD drive, after a few seconds the install program should self activate if not run setup.exe found in the root of the CD drive. Follow the on screen instructions to install the software.

## Configuration

When starting the Sequencer for the very first time, the *Mains Configuration* program will appear:



Mains configuration

Select the mains frequency applicable to your country:

● 50 Hz

○ 60 Hz

Select the mains frequency that matches your country, the mains frequency is required by the analogue to digital converters (ADC) in the instrument, so they are able to digitally reject mains noise.

## Software

## Overview

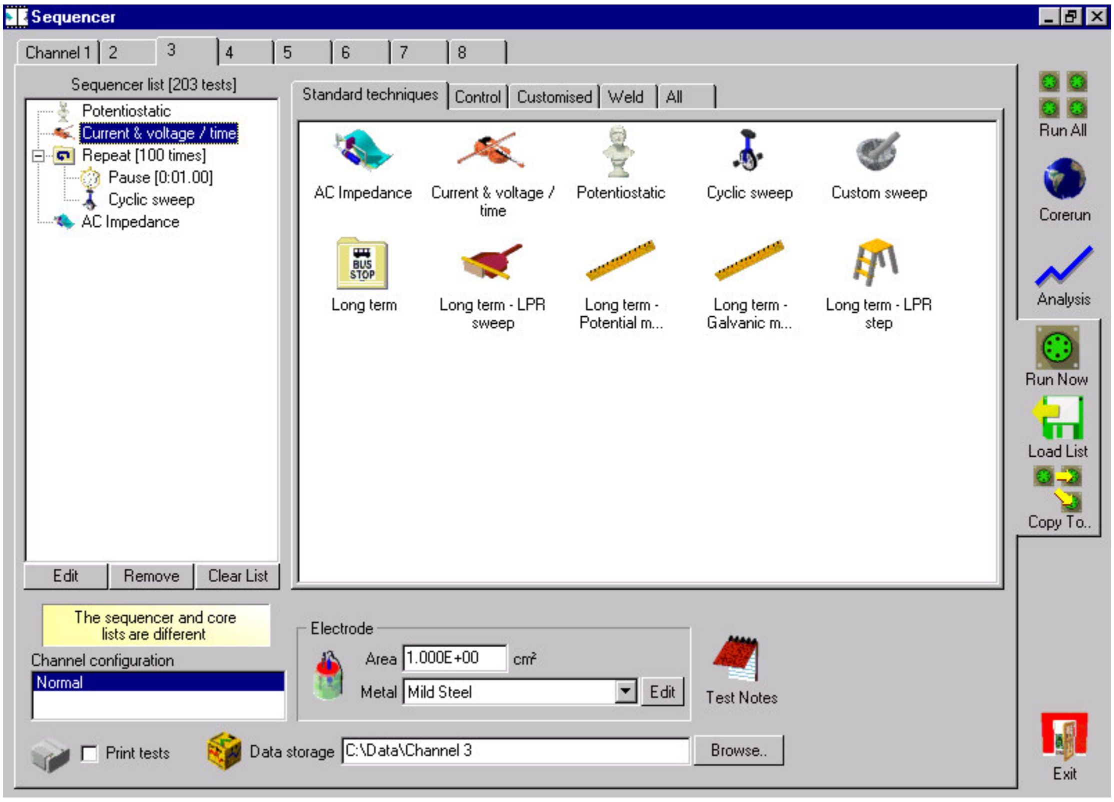
It is worth mentioning at this early stage that any potential shown in any of the programs is always shown or entered with respect to the reference electrode.

Software is split into two groups, logging and analysis. These programs can be activated from the *Programs* menu item in the *Start* menu. The analysis programs will be found under a menu name of *ACM Instruments*, whilst logging programs will be found under a name matching the serial number of your instrument, for example *Serial number 383*.

## Logging

Two programs make up the logging software, *Sequencer* and *Core running*. The *Sequencer* does the work of setting up a list of tests (a sequence) to be passed to *the Core running* for collection.

# Sequencer



Sequencer list [203 tests]:

- Potentiostatic
- Current & voltage / time
- Repeat [100 times]
  - Pause [0:01.00]
  - Cyclic sweep
- AC Impedance

Channel configuration: Normal

Area: 1.000E+00 cm²

Metal: Mild Steel

☐ Print tests

Data storage: C:\Data\Channel 3

Shown above is the sequencer, in the above example there are eight channels as designated by the *Channel* tabs at the top of the page and channel 3 is selected. To switch between channels, click on the channel number required. Each channel is treated independently, although a sequence of tests can be created on one channel to be copied to other channels.
Directly under the *Channel* tabs on the left hand side of the page is the sequence list. The order in which the *Core running* works through the list is from the top downwards.

To the right of the sequence list are the technique selection tabs, within these tabs are techniques. To add a technique to the sequence list, click once on a technique and a settings page for the added technique will appear. For a description of each technique page, refer to a later section in this manual.

The Sequencer follows these rules when adding a technique to the sequence list:

1) If the last technique of a sequence list is selected, any new technique added will be placed at the end of the list:

when Potentiostatic is clicked

2) If the selected technique is not the last technique, adding a new one to the list will insert it before the selected technique:

when Cyclic Sweep is clicked

If techniques in the sequence list are not in the desired order, drag a technique from one part of the list to another and drop it on top of another technique in the list. When dropped a menu will appear, with options to place the technique before, after or into (see folder section next) the technique it was dropped on.

## Folders

Folders are used by the sequence list when tests need to be grouped together for a special function, for example when a group of techniques are to be repeated a number of times.

## Repeat Folder

The *Repeat Folder* can be found in the *Control* techniques tab, after clicking on it once to add it to the sequence list, a settings form will appear. The number of time to repeat can be set, for this example the repeat folder will be set to 15. Afterwards the sequence list will resemble:


Repeat [15 times]

Now a technique to be repeated can be added into the repeat, for this example *AC Impedance* was clicked:


Repeat [15 times]
AC Impedance

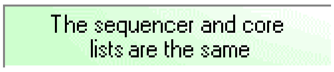
Additional techniques could be added into the repeat by just adding more tests to the sequence list, they will be automatically placed within the repeat folder. To add tests after a repeat and not into the repeat, click on the - symbol next to the repeat folder to close it, any new tests will be added after the repeat.

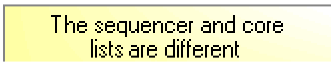
## Long Term

The Long Term folder acts in a similar manor to the repeat folder, except that only certain types of techniques can be placed into it. Only techniques prefixed with the name *Long Term* - such as *Long Term - LPR Sweep* can be placed in a long term folder. The long term folder sets up how many long term readings should be taken over scheduled intervals. For example *Long Term - LPR Sweep* could be placed in the sequence list as a normal test, but only one LPR sweep would be taken, creating only one LPR result. If it was placed within a Long term folder set to record 24 readings, one every hour, then 24 LPR results would be created linked together under one long term graph.

## Updating Sequence List

The core running and sequencer each have their own separate sequence lists, a list can be sent from the sequencer to the core running. On the main page is a section showing if the sequencer and core running lists differ:


The sequencer and core lists are the same


The sequencer and core lists are different

If changes have been made to the sequencer list they can be sent to the core running ready to be collected, in one of three ways:

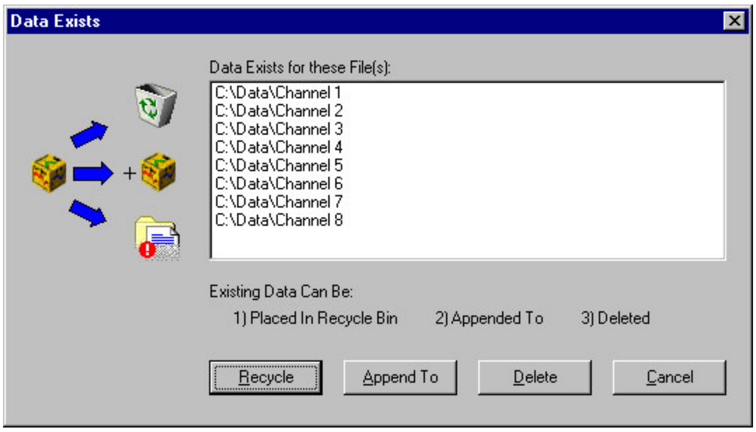
The sequence list for the selected channel only will be sent to the core running, which will be instructed to start logging on that channel straight away.

Every channel will be updated to the core running, logging will then commence on all channels.

The option to update selected channels will be given with this button, note however after the core running has been updated, no channels will automatically start logging.

## Overwriting Existing Data

When existing data is about to be overwritten by a new sequence  (such as selecting *Run Now*) the below page will be displayed:



Data Exists

Data Exists for these File(s):

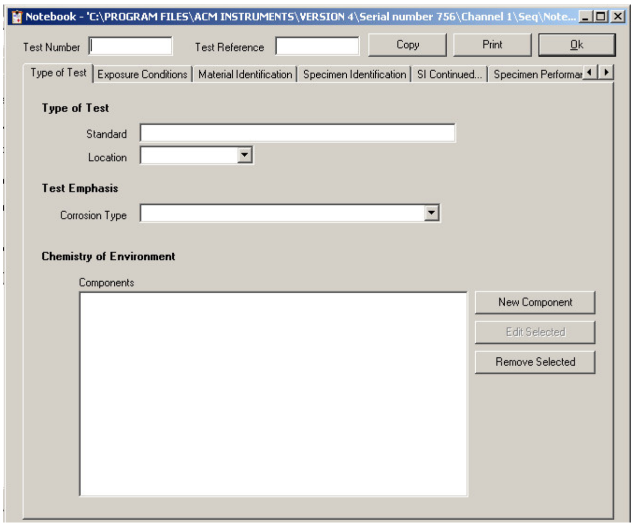
C:\Data\Channel 1
C:\Data\Channel 2
C:\Data\Channel 3
C:\Data\Channel 4
C:\Data\Channel 5
C:\Data\Channel 6
C:\Data\Channel 7
C:\Data\Channel 8

Existing Data Can Be:
1) Placed In Recycle Bin   2) Appended To   3) Deleted

The existing data can be sent to the recycle bin, appended to or deleted permenatly.

## Note book

The note book complies with the ASTM G107 standard, and provides a way of storing detailed information with test data.



Notebook - 'C:\PROGRAM FILES\ACM INSTRUMENTS\VERSION 4\Serial number 756\Channel 1\Seq\Note...

Test Number
Test Reference

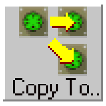
**Type of Test**
Standard
Location

**Test Emphasis**
Corrosion Type

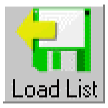
**Chemistry of Environment**
Components

Information can be retrieved from the note book at a later date using the analysis programs, or database searches (on fields / dates) can be used to find data, see later on in this manual for details on how to search.

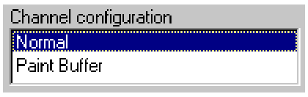
## Advanced Features of the Sequencer



The *Copy To..* button is used to copy all settings from one channel to another, be careful when using this function that no two channels have the same file name when updating to the core running.



The settings (sequence list, and note book settings) from a previously gathered sequence can be loaded into the selected channel.

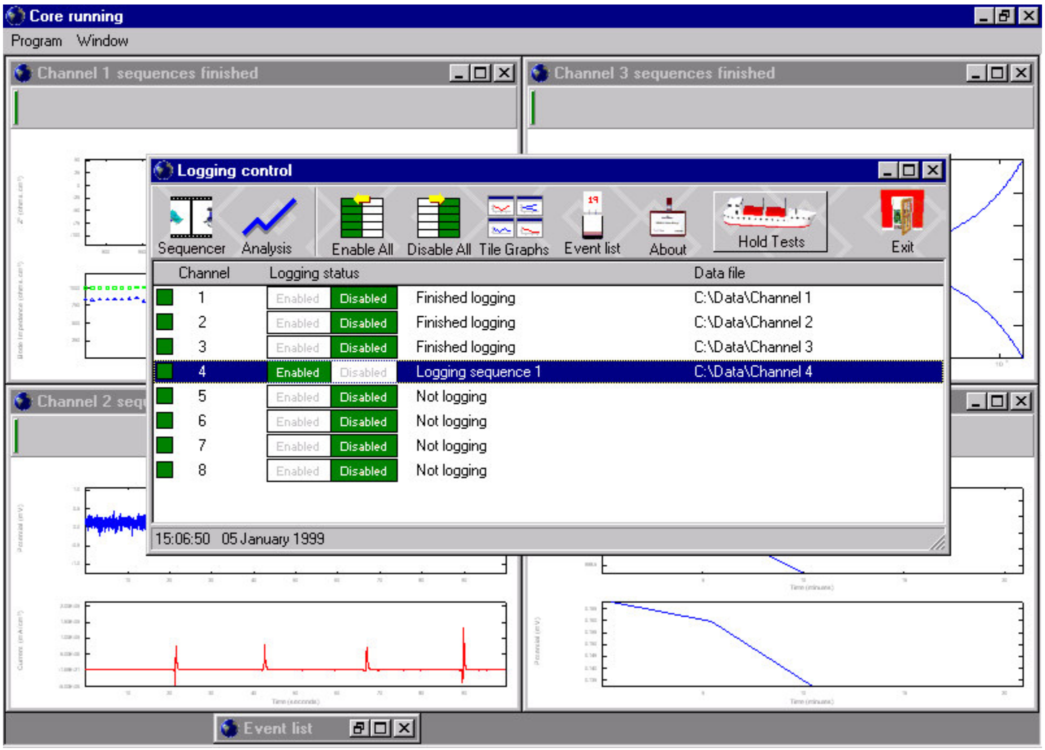

Channel configuration

Normal

Paint Buffer

If a paint buffer option is available, it will be listed within the channel configuration section. Select *Paint Buffer* if the paint buffer is being used, otherwise *Normal* should be selected.

## Core running



| | Channel | Logging status | | | Data file |
|---|---|---|---|---|---|
| 🟩 | 1 | Enabled | Disabled | Finished logging | C:\Data\Channel 1 |
| 🟩 | 2 | Enabled | Disabled | Finished logging | C:\Data\Channel 2 |
| 🟩 | 3 | Enabled | Disabled | Finished logging | C:\Data\Channel 3 |
| 🟩 | 4 | Enabled | Disabled | Logging sequence 1 | C:\Data\Channel 4 |
| 🟩 | 5 | Enabled | Disabled | Not logging | |
| 🟩 | 6 | Enabled | Disabled | Not logging | |
| 🟩 | 7 | Enabled | Disabled | Not logging | |
| 🟩 | 8 | Enabled | Disabled | Not logging | |

15:06:50 05 January 1999

Each channel is controlled from *Logging Control* within the Core running program, to start data collection click the Enabled box for the channel in question, the green marker will move over to Enabled showing logging is taking place for that channel.

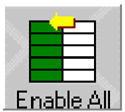
To the left of each channel number in *Logging Control* is a 🟩 box, these boxes indicate if a particular channel is actively running a technique at that precise moment, light green indicates logging is taking place. On a multiplexed instrument such as a Gill 8, only one channel will have the green light, but on a parallel system all channels could be lighted at the same time. The green box is also mirrored on a channels logging graphical page ▌. Clicking once on this area displays a list of tests the core is working through, the active test highlighted in the list, clicking again on the green square will hide the list.

To facilitate navigating around the core running a number of key presses and mouse clicks can switch between different windows they are:

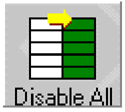
> To switch to the *Logging Control* window - press the *insert* key, pressing *insert* again when *Logging Control* is active will hide it. Another way of switching to *logging control* uses the mouse, right click the mouse on the grey tool bar on any of the data graphs.
>
> To switch to a channel's graphical page press *Shift + F1-8 (1 for channel 1).* Alternatively right click the mouse anywhere on a channel line:
>
> 🟩 2 Enabled Disabled Finished logging
>
> in *Logging Control* to switch to a channel's graphical page.

Taking Control

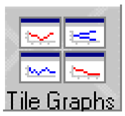
*Logging Control* buttons:



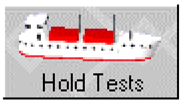
Starts logging on any channel not logging (same as *Run All* on the sequencer*)*.



Disables all channels. The disable is instantaneous and does not wait for any tests to finish.



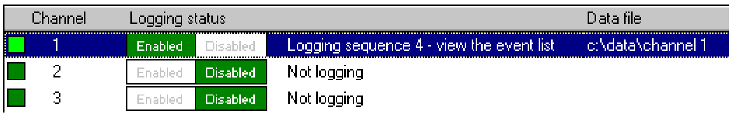
Pressing this button minimises *Logging Control* and arranges each channels graphic page so they are all visible.



*Hold Tests* acts as a toggle button, whilst this button is pressed down no techniques will be started, until the button is released.

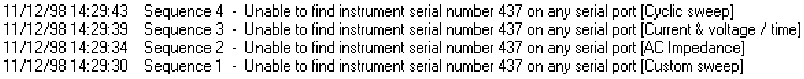
Event List

The *Event List* only comes into play when there is a problem on one of the channels, such as an instrument communication problem. An example will be created - a sequence of tests was started on channel 1 with the instrument turned off, the *Logging Control* window will display:



| | Channel | Logging status | | | Data file |
|---|---|---|---|---|---|
| 🟩 | 1 | Enabled | Disabled | Logging sequence 4 - view the event list | c:\data\channel 1 |
| 🟩 | 2 | Enabled | Disabled | Not logging | |
| 🟩 | 3 | Enabled | Disabled | Not logging | |

Notice the line of text - *view event list*, this signifies the event list contains details that should be looked into. The event list window can be selected by clicking *Event List* from the *Window* menu, for our example the event list contains:



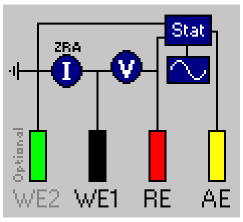
```
11/12/98 14:29:43   Sequence 4  -  Unable to find instrument serial number 437 on any serial port [Cyclic sweep]
11/12/98 14:29:39   Sequence 3  -  Unable to find instrument serial number 437 on any serial port [Current & voltage / time]
11/12/98 14:29:34   Sequence 2  -  Unable to find instrument serial number 437 on any serial port [AC Impedance]
11/12/98 14:29:30   Sequence 1  -  Unable to find instrument serial number 437 on any serial port [Custom sweep]
```

Locking your Computer

To stop unauthorised access to the core running during testing, Windows NT can be locked. To lock Windows NT, press CTRL + ALT + DELETE together and select *Lock Workstation*, the core running will carry on logging unaffected.

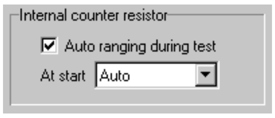
# Common Elements

Various areas on set-up pages are common to most techniques, these are:

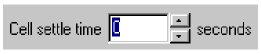


Each set-up page has a diagram of any cell connections required for the test to function correctly, optional connections are shown in light grey.

**All potentials measured or inputted are displayed with respect to the reference electrode.**


Internal counter resistor
☑ Auto ranging during test
At start Auto

This section allows the precise control of internal count resistors at the beginning and during a test. *Auto ranging during test* if checked, allows the instrument to change to the best-suited count resistor whilst the test is running. At the start of the test the count resistor can be fixed on a certain resistor, or be automatically found before a test is started. These two options can be combined in any order, for example it is possible to automatically find the best count resistor at the start of the test, and then to fix on this resistor during the test (*Auto ranging during test* unchecked, and *At Start* to *Auto*).


Cell settle time 0 seconds

*Cell Settle Time* allows the cell to settle before a test begins, during a settle delay the instrument is coupled to the cell, at the test start potential.
This option should not be confused with a *Pause technique* (*Pause* technique - waits a pre-set time between tests, the cell is isolated).

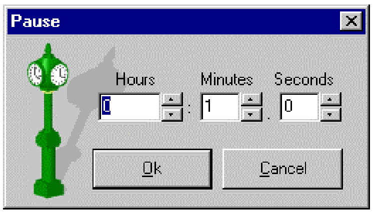

☑ Offset test to the rest potential

The option for offsetting to the rest potential is available when a technique applies a potential. For normal use this option should be on (checked), even if testing dummy resistor test cells. An example of *Offset test to rest potential* and a Cyclic test –

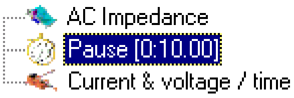
> Assuming a Cyclic technique was set to sweep from -100mV to +100mV and there was a rest potential of -53mV (**All potentials are with respect to reference electrode)** the actual potential of the sweep would be -153mv to +47mV.

## Pause

The pause technique can used to place a delay between two techniques, whilst a pause is taking place the channel is isolated from the cell.


Pause

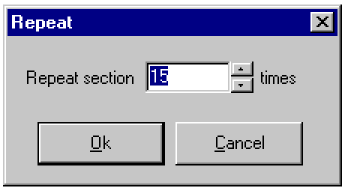
Hours 0 : Minutes 1 . Seconds 0


AC Impedance
Pause [0:10.00]
Current & voltage / time

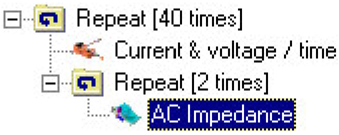
## Repeat

Techniques to be repeated are placed within a repeat folder, *folders* are covered earlier in this manual (sequencer section).

If a *Repeat* folder is selected when a technique is added to the list, the technique will placed inside the repeat folder. After a *Repeat* folder is populated with the required techniques, click the [-] sign next to the folder to close it, adding a technique to the list  now will place the technique after the *Repeat* and not into it.


Repeat

Repeat section 15 times


Repeat [40 times]
- Current & voltage / time
- Repeat [2 times]
  - AC Impedance

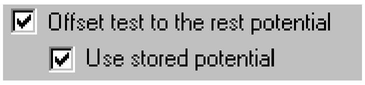
In the above illustration the core running would collect:

*Current & voltage / time*
*AC Impedance*
*AC Impedance*

All repeated 40 times.

## Measure and Store Rest Potential

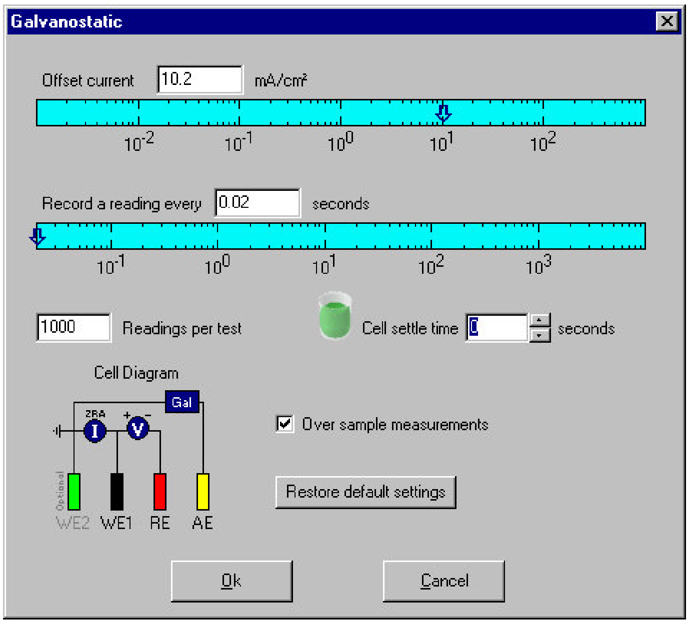
With this technique the Rest potential can be measured and stored (normally at the beginning of a sequence of tests), to be used at a later time. Each subsequent logging technique can use this stored rest potential, instead of measuring a new rest potential at the start if it's test:


☑ Offset test to the rest potential
  ☑ Use stored potential

Typically this technique can be used remove the effects of Rest potential drift caused by polarising techniques.

Please note if *Use stored potential* is used in any techniques, a *Measure and Store Rest Potential* technique MUST be placed earlier in the sequence list for the test to run correctly.

# Galvanostatic


Galvanostatic

Offset current 10.2 mA/cm²

Record a reading every 0.02 seconds

1000 Readings per test

Cell settle time 0 seconds

☑ Over sample measurements

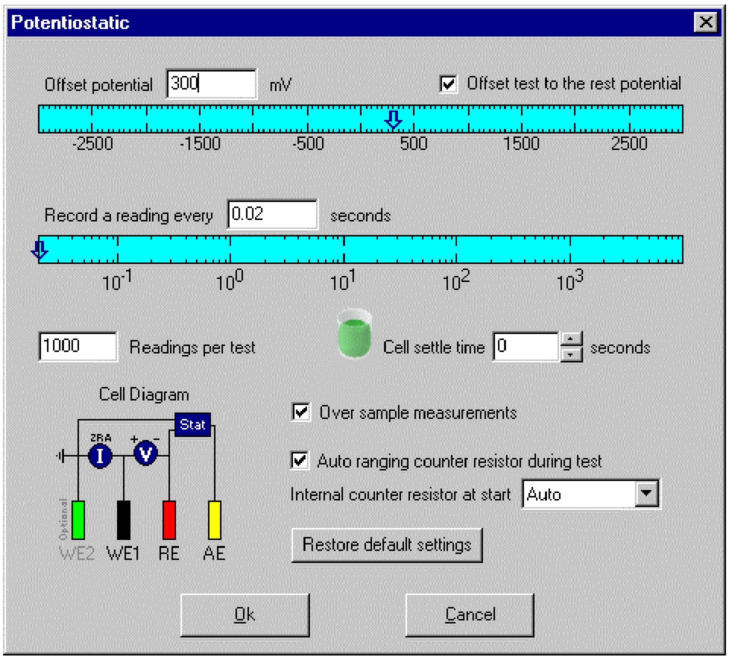
The Galvanostatic technique sets a specified static offset current (area corrected) whilst monitoring the potential response. Care must be taken when choosing the offset current, a current should be picked the instrument is able to supply, for example:

> Assuming the cell impedance is 1000 ohms the maximum offset current is 15mA, this is calculated from
>
> $$\frac{\text{maximum compliance potential}}{\text{cell impedance}} \qquad \frac{15000\text{ mV}}{1000\text{ ohms}}$$

Using *Over sample measurements* greatly improves measurement accuracy, many readings are averaged for each point.

A *Cell settle time* delays at the test offset current, it can be used to give the cell a chance to settle down after the offset current has been applied.

# Potentiostatic



Potentiostatic

Offset potential 300 mV

☑ Offset test to the rest potential

Record a reading every 0.02 seconds

1000 Readings per test

Cell settle time 0 seconds

☑ Over sample measurements

☑ Auto ranging counter resistor during test

Internal counter resistor at start Auto

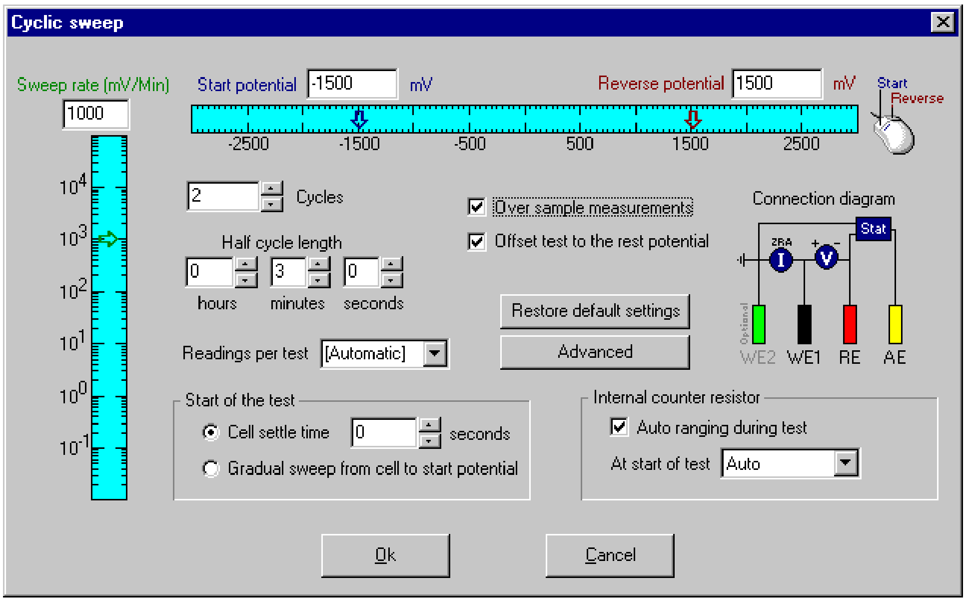
Potentiostatic applies a constant DC offset potential to the cell whilst measuring galvanic readings at up to 50/60 measurements per second. The maximum read rate is governed by the mains frequency, 0.02 for 50Hz and .0166667 for 60Hz. In addition to a DC offset potential, the test can be offset to the cell rest potential.

*Over Sample Measurements* can reduce aliasing errors, for example, if a reading was to be recorded every second, with over sample switched on, the instrument will record at roughly 30 measurements per second. An averaged result would be obtained from these 30 measurements, thus reducing aliasing errors. It should be noted over sampling will only work when the read rate less than the mains frequency.

2

As with *Current & Voltage / time*, if count resisters are permitted to auto range during the test and a count resistor change happens, a delay will occur from when the new count resistor was set. The delay allows the cell to settle a set length of time, dependant upon the resistor set. Whilst a delay is taking place the test continues, but no data is stored during the delay, later in the analysis a straight line will be present from the change point to the point after the delay has finished.

# Cyclic Sweep



Cyclic sweep

Sweep rate (mV/Min): 1000

Start potential: -1500 mV

Reverse potential: 1500 mV

Cycles: 2

Half cycle length: 0 hours, 3 minutes, 0 seconds

Readings per test: [Automatic]

☑ Over sample measurements

☑ Offset test to the rest potential

Start of the test

● Cell settle time: 0 seconds

○ Gradual sweep from cell to start potential

Internal counter resistor

☑ Auto ranging during test

At start of test: Auto

A cyclic sweep technique performs a sweep from the start potential to the reverse potential over a set number of cycles. One complete cycle goes from the start to reverse back to the start potential, half cycles can be specified to finish on a reverse potential.

The rate at which the sweep progresses is controlled by either the *Sweep rate* or the *Half cycle length* sections, these two sections are linked together, changing one affects the other ones values.

Using *Over sample measurements* greatly improves the smoothness of recorded data, many readings are averaged for each point. The exact number of readings averaged per point depends upon the sweep rate and *Readings per test*.

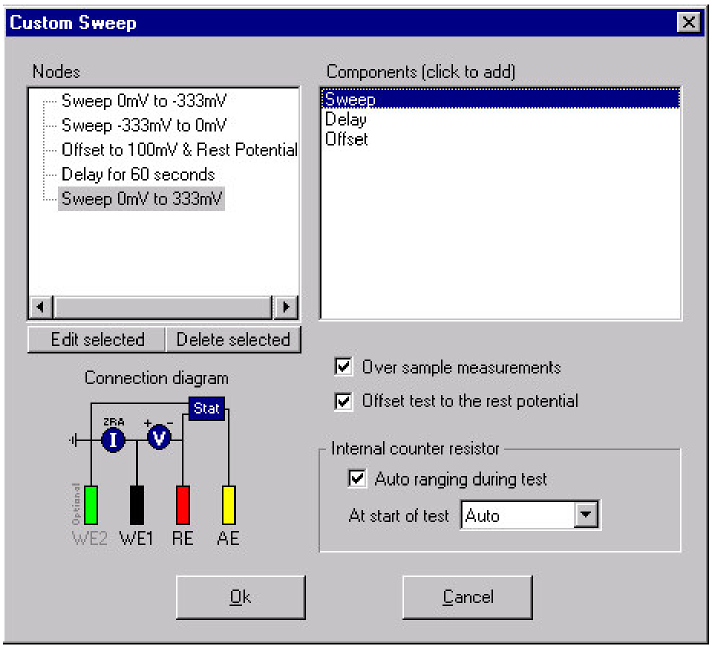
*Cell settle time* delays at the start potential, which includes the rest potential if *Include rest potential* is checked, if the internal counter resistor option *At Start of test* is set to *Auto* the best internal count resistor during the delay will be found.

*Gradual sweep from cell to start potential* stops the cell from being jolted when the start potential is applied. Instead of setting the start potential, a gradual sweep from the cell rest potential to the start potential at a rate determined by *Sweep rate* is performed.

## Advanced Settings

A positive current limit can be set on the advanced page, once the limit has been reached there are options to either end the test, or to reverse the sweep.

# Custom Sweep



Custom Sweep

Nodes

- Sweep 0mV to -333mV
- Sweep -333mV to 0mV
- Offset to 100mV & Rest Potential
- Delay for 60 seconds
- Sweep 0mV to 333mV

Components (click to add)

- Sweep
- Delay
- Offset

☑ Over sample measurements

☑ Offset test to the rest potential

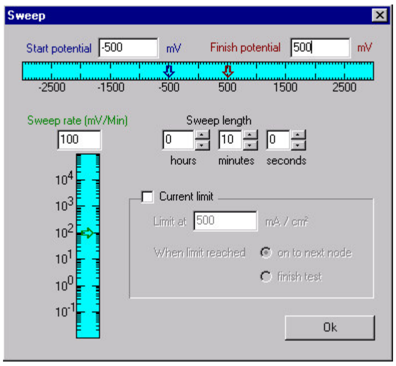
Internal counter resistor

☑ Auto ranging during test

At start of test: Auto

A custom sweep is quite similar to the cyclic sweep technique, except that user defined sweeps can be constructed with delays and re-offsets. A custom sweep is constructed from *Nodes*, to add a *Node* click one of the available *Components*:

## Sweep



Sweep

Start potential: -500 mV

Finish potential: 500 mV

Sweep rate (mV/Min): 100

Sweep length: 0 hours, 10 minutes, 0 seconds

☐ Current limit

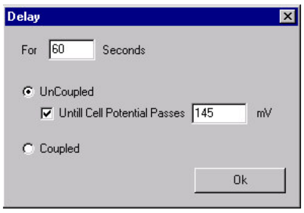
Limit at: 500 mA / cm²

When limit reached: ● on to next node ○ finish test

A sweep from a *start* to a *finish* potential at a defined *sweep rate* or *sweep length*.

A positive current limit can be imposed, when reached the test can either move onto the next node or the test can be finished.

## Delay



Delay

For 60 Seconds

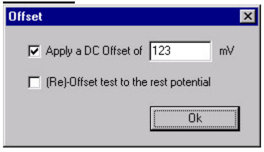
● UnCoupled

☑ Untill Cell Potential Passes 145 mV

○ Coupled

A delay for a predefined length of time, either coupled to or isolated from the cell. If the delay is coupled the cell potential is held at the last set potential - normally the finish potential of the previous sweep node, or an offset potential (see below).
If the cell is isolated, there is an option to end a delay if the cell potential passes a set potential.

## Offset



Offset

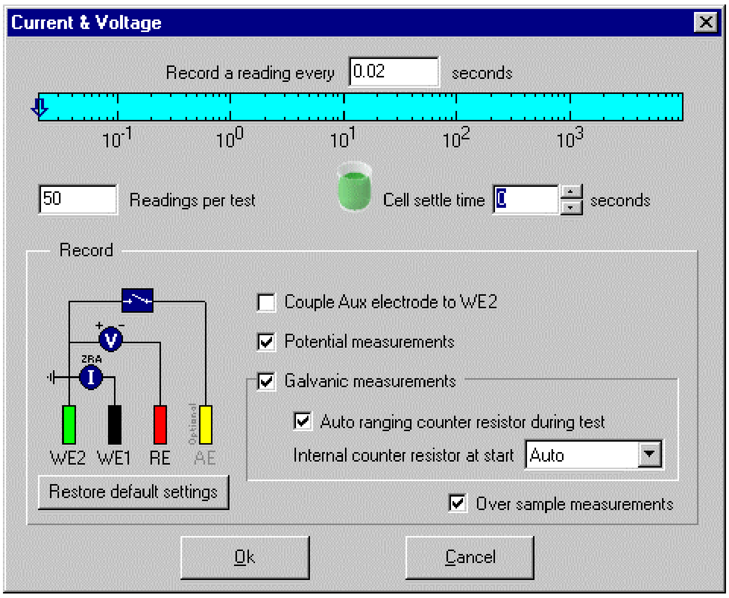
☑ Apply a DC Offset of 123 mV

☐ (Re)-Offset test to the rest potential

The offset can be used to set a potential before a delay, or to re-measure and re-offset to the rest potential before a sweep node.

Using *Over sample measurements* greatly improves the smoothness of recorded data,  many readings are averaged for each point.

# Current & Voltage / time



Current & Voltage

Record a reading every 0.02 seconds

50 Readings per test

Cell settle time 0 seconds

Record

☐ Couple Aux electrode to WE2

☑ Potential measurements

☑ Galvanic measurements

☑ Auto ranging counter resistor during test

Internal counter resistor at start Auto

☑ Over sample measurements

Current & Voltage permits the continuous measurement of potential and galvanic readings. Should the read rate be infrequent (one point every 15 minutes) consider using a *Long Term* test with *Potential Measurement* and *Galvanic Measurement* tests. The *Long Term* has the advantage of freeing up the instrument during measurements, where as *Current & Voltage* holds on the instrument during measurements.

The maximum read rate for *Current & Voltage* is limited by the mains frequency, 0.02 for 50Hz and .0166667 for 60Hz. With *Over Sample Measurements* aliasing errors can be reduced, for example, if a reading was to be recorded once a second, with over sample switched on, the instrument will record at roughly 30 measurements per second, an averaged result would be obtained from these 30 measurements, thus reducing aliasing errors. It should be noted over sampling will only work when the read rate less than the mains frequency.

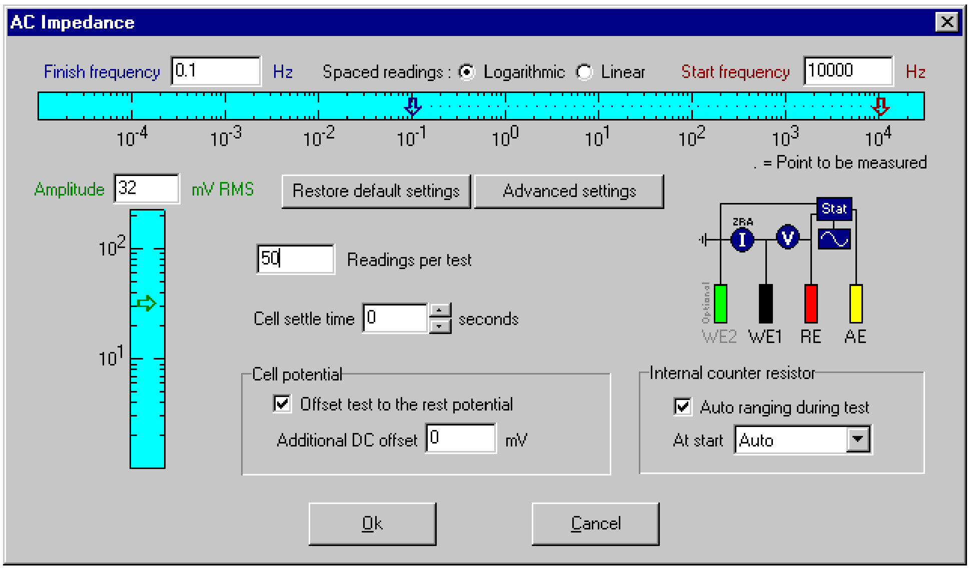
2

If count resisters are permitted to auto range during the test and a count resistor change happens, a delay will occur from when the new count resistor was set. The delay allows the cell to settle a set length of time, dependant upon the resistor set. Whilst a delay is taking place the test continues, but no data is stored during the delay, later in the analysis a straight line will be present from the change point to the point after the delay has finished.

*Couple Aux Electrode to WE2* allows 3 electrode cells to record galvanic measurements, the AE connection normally used to polarise the test can be switched to act as WE2, so WE2 does not have to be connected.

Potential Gain – an internal gain can be applied to the potential measurement to increase resolution, maximum recordable potentials for each gain are: 10x ±750mV
100x ±75mV 1000x ±7.5mV

# AC Impedance



AC Impedance

Finish frequency 0.1 Hz

Spaced readings : ● Logarithmic ○ Linear

Start frequency 10000 Hz

Amplitude 32 mV RMS

50 Readings per test

Cell settle time 0 seconds

Cell potential

☑ Offset test to the rest potential

Additional DC offset 0 mV

Internal counter resistor

☑ Auto ranging during test

At start Auto

Whilst an AC Impedance test is running the time constant of the instrument is altered by switching the potentiostat into a faster mode, more suited to an AC Impedance test.

For normal testing *Spaced readings* should be set on *Logarithmic* (where the same number of points are collected across each decade), also the *Start Frequency* should be higher than the *Finish Frequency*, this enables the initial count resistor to be found more quickly.

The *Amplitude* used for the AC signal plays an important role in obtaining the best possible results, always use the highest amplitude possible, a test at 30mV AC will yield better results than a test at 4mV AC.

The cell potential applied to the cell is affected by two options, firstly is the option *Offset test to the rest potential* that biases the test around the rest potential if checked, the second option allows a DC Offset to be applied (with respect to RE). An example of both options, a cell has a rest potential of 133mV, if *Offset test to the rest potential* is checked and there is a DC Offset of 200mV to be applied then the test would be done at 333mV.

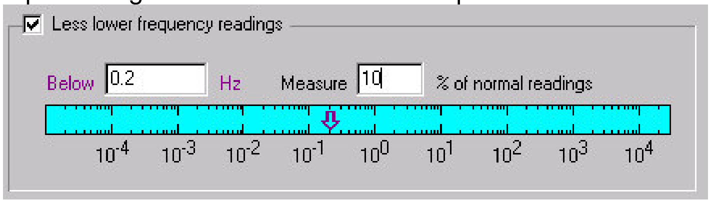
## Advanced Settings

Each instrument undergoes a very extensive calibration procedure over a series of 8 decades of cell load, 10 amplitudes and the full range of frequencies to produce a calibration matrix we call the P.A.I.R. table (Phase and Impedance Reduction). This is then applied to the raw data to reduce cable and instrument induced errors. The PAIR option is on by default, it can be accessed on the Advanced options page.

Averaging over a number of points can improve AC Impedance results, the averaging options are advanced in that a time penalty of unnecessary averaging is avoided. There are two averaging options which complement each other - *Integration factor* and *Maximum Integration Cycles*. Starting with *Maximum Integration Cycles* this controls the maximum number of averages allowed. The other option *Integration factor* specifies the percentage error until the result can be passed. For example with a 5% error setting the averaging will continue until the averaged result drift from averaging drift is less than 5%. *Low, Medium and High* translate into 8%, 0.75% and 0.25% respectively.

The lower the frequency the longer each point will take to collect, at high frequencies many averages can be done in the blink of an eye, but at very slow frequencies data collection will slow down dramatically. To speed up a test at these lower frequencies *Single Integration Factor below 1 Hz* can be checked, it effectively turns off averaging below 1 Hz.

*Less lower frequency readings* can also speed up a test, by collecting less points below a certain frequency, a percentage of the normal number of points can be set.

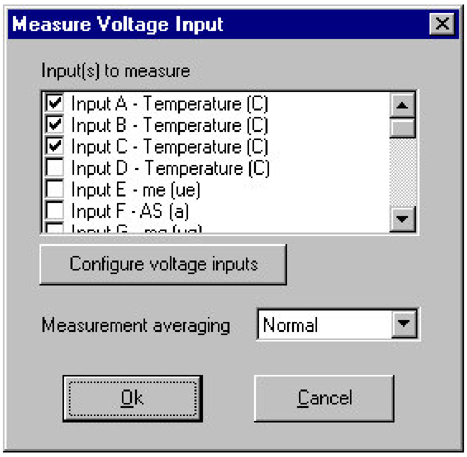


☑ Less lower frequency readings

Below 0.2 Hz

Measure 10 % of normal readings

# Voltage Inputs

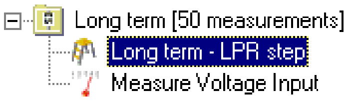

**Measure Voltage Input**

Input(s) to measure

☑ Input A - Temperature (C)
☑ Input B - Temperature (C)
☑ Input C - Temperature (C)
☐ Input D - Temperature (C)
☐ Input E - me (ue)
☐ Input F - AS (a)

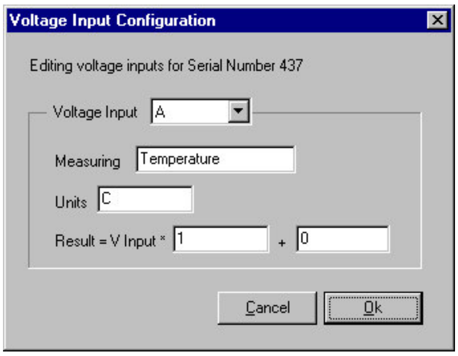
Measurement averaging: Normal

A voltage input mesurement technique can be placed anywhere in a sequence list, for example:



The voltage input can be placed within a long term folder, so that measurements can be taken along with other long term measurements. When setting up a voltage input measurement for the first time the voltage inputs for that instrument have to be configured to mirror what is being mesured, this is done by clicking the ‘Configure voltage inputs’button on the *Measure voltage input* selection page.

### Configuring Voltage Inputs


**Voltage Input Configuration**

Editing voltage inputs for Serial Number 437

Voltage Input: A

Measuring: Temperature

Units: C

Result = V Input * 1 + 0

The measurement range of a voltage inputs is –9000mV to +9000mV, to adapt the recorded potential to different units (ie Temperature) a result = mx+c formula is provided, for example:

Result = V Input * 0.5 + -200

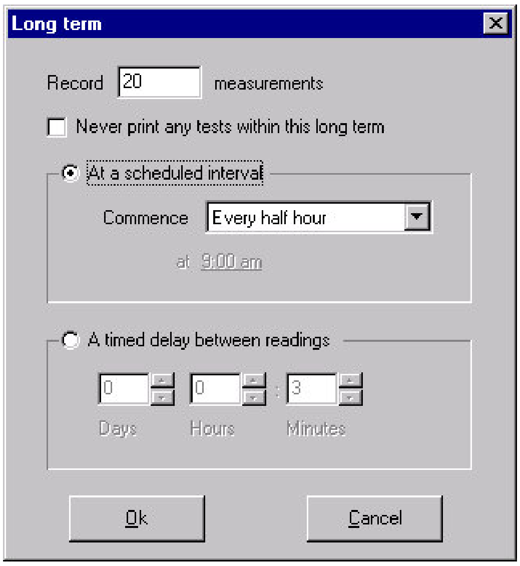
Would take the measured potential, half it and subtract 200 (mV) from it. When voltage inputs are configured, changing the configuration will affect all voltage inputs for a particular instrument.

### Core running and Voltage inputs

After a voltage input has been measured the core running creates a option button in the lower right hand corner of a channels data graph:

Clicking on the V Input item switches the graph from displaying normal techniques to a voltage input graph:
If two or more voltage inputs have the same *measuring* title and *units*, a combined graph showing these inputs can be selected in the *V Input* selection box. The key box shows which line corresponds to which input

# Long Term



Record 20 measurements

☐ Never print any tests within this long term

● At a scheduled interval

Commence Every half hour

at 9:00 am

○ A timed delay between readings

0 Days 0 Hours : 3 Minutes

The long term technique is a folder, into which certain tests can be inserted (tests with names prefixed by [*Long Term* -]), these tests include:

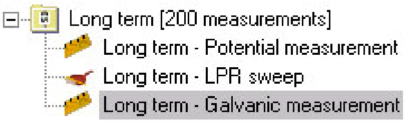
Long Term - Potential Measurement
Long Term - Galvanic Measurement
Long Term - LPR Sweep
Long Term - LPR Step

The long term technique it's self contains a number of options - *Read* x *Measurements* set how many times the tests within the long term folder should be repeated. The interval between each long term measurement is set by either *At a scheduled interval,* or *A timed delay between readings*. To ensure long term readings are evenly spaced, subsequent long term reads are scheduled from when a long term reading begins, not from when the tests within the long term finish. For example if a long term test is scheduled to measure every 5 minutes and contained within the long term test is an LPR sweep that will last approximately 2 minutes, then an LPR sweep will be recorded every 5 minutes regardless of the time taken for the LPR sweep.

Why use a Long term test?

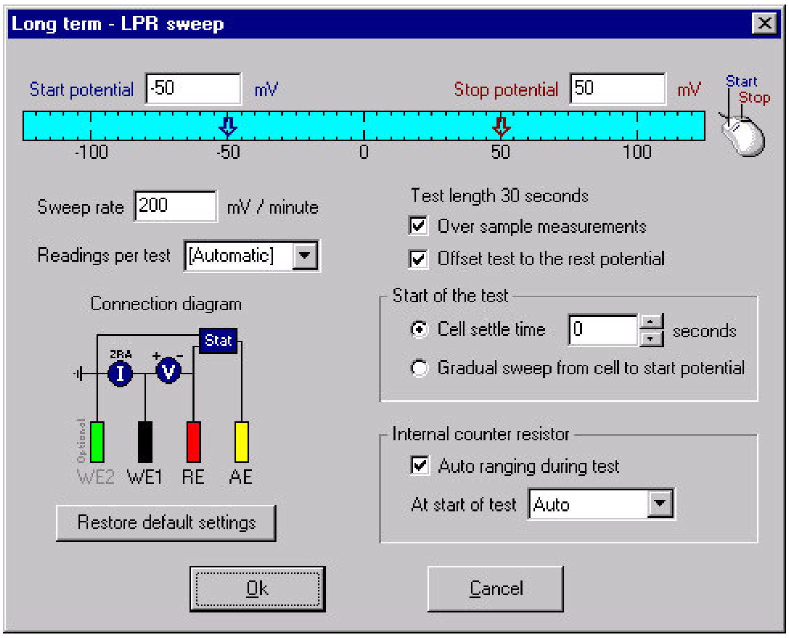
Apart from providing scheduled times, the main benefit from using a long term folder is that all results generated by techniques contained within the folder are linked together, so that they can be presented on a result vs time graph in the analysis program.

Example use of long term :



Long term [200 measurements]
- Long term - Potential measurement
- Long term - LPR sweep
- Long term - Galvanic measurement

# Long Term - LPR Sweep



Start potential -50 mV

Stop potential 50 mV

Sweep rate 200 mV / minute

Readings per test [Automatic]

Test length 30 seconds

☑ Over sample measurements

☑ Offset test to the rest potential

Start of the test

● Cell settle time 0 seconds

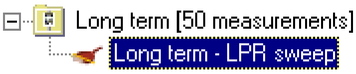
○ Gradual sweep from cell to start potential

Internal counter resistor

☑ Auto ranging during test

At start of test Auto

The LPR sweep technique calculates an LPR result from a polarisation sweep. The LPR Sweep technique can be placed inside a Long Term folder (to be repeated at scheduled intervals, all results will be shown using a result vs time graph), or can be placed on its own anywhere in a sequence list.



within a Long term folder

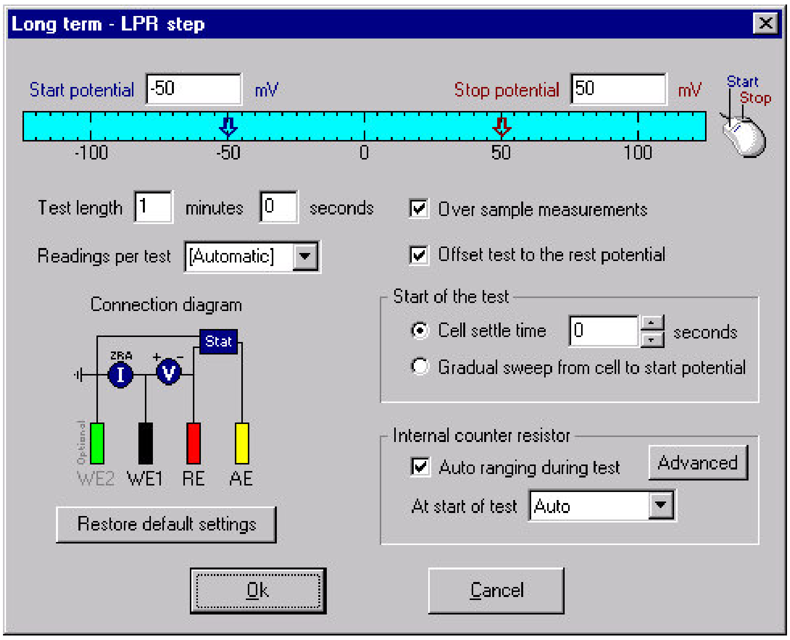
single LPR test

Using *Over sample measurements* greatly improves the smoothness of recorded data,  many readings are averaged for each point. The exact number of readings averaged per point depends upon the sweep rate and *Readings per test*. Readings per test should be set to *[Automatic]* to gain the best sample / average rate.

*Cell settle time* delays at the start potential, which includes the rest potential if *Include rest potential* is checked, if the internal counter resistor option *At Start of test* is set to *Auto* the best internal count resistor during the delay will be found.

*Gradual sweep from cell to start potential* stops the cell from being jolted when the start potential is applied. Instead of setting the start potential, a gradual sweep from the cell rest potential to the start potential at a rate determined by *Sweep rate* is performed.

# Long Term - LPR Step


Long term - LPR step

Start potential -50 mV

Stop potential 50 mV

Test length 1 minutes 0 seconds

Readings per test [Automatic]

☑ Over sample measurements

☑ Offset test to the rest potential

Start of the test

● Cell settle time 0 seconds

○ Gradual sweep from cell to start potential

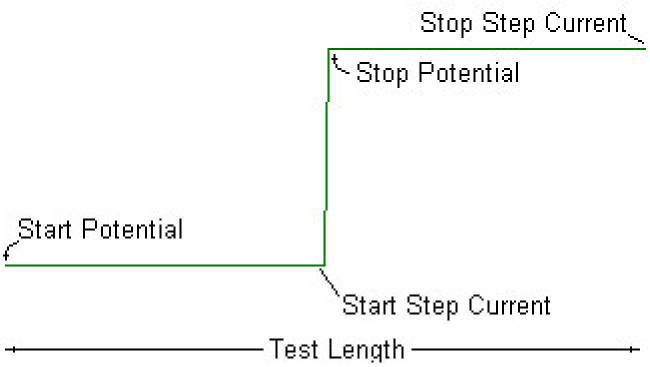
Internal counter resistor

☑ Auto ranging during test

At start of test Auto

The LPR Step begins the first half of the test at the start potential, at exactly half way through the test a ‘start step current’ is stored, the step is applied to the stop potential. Just before the end of the test a ‘stop step current’ is stored, from these two stored currents a LPR result is calculated.

Step Diagram



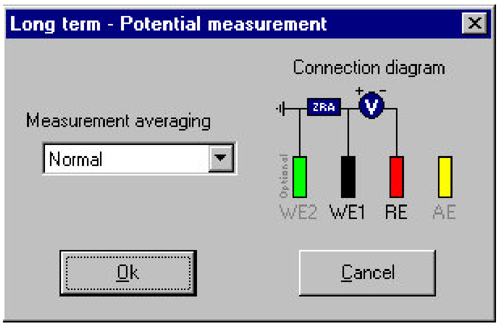
## Advanced Settings


Advanced count resistor setting

Count resistor at step Auto

Half way through the test various count resistor options can be applied when the stop potential is set. The *Auto* option reduces the internal count resistor to 10 ohms, after which it will auto range. If *Leave on current* is chosen, the internal CR will not change unless it needs to (assuming *Auto ranging during test* is selected).

# Long Term - Potential Measurement

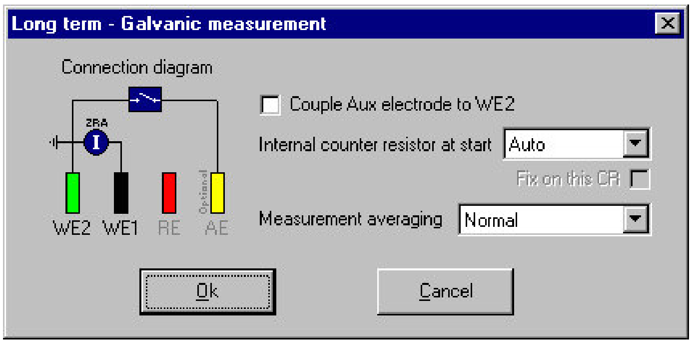


Measurement averaging: Normal

Potential Measurement only serves a purpose if it is placed within a long term folder, this is because the technique only gives a potential result and does not shown any graphical data it’s self. There is only one setting for potential measurement - *Measurement averaging*, the actual averages taken for each setting are as follows:

| Measurement Averaging | Number of Averages |
|---|---|
| Normal | 20 |
| High | 40 |
| Very High | 60 |

# Long Term - Galvanic Measurement



☐ Couple Aux electrode to WE2

Internal counter resistor at start: Auto

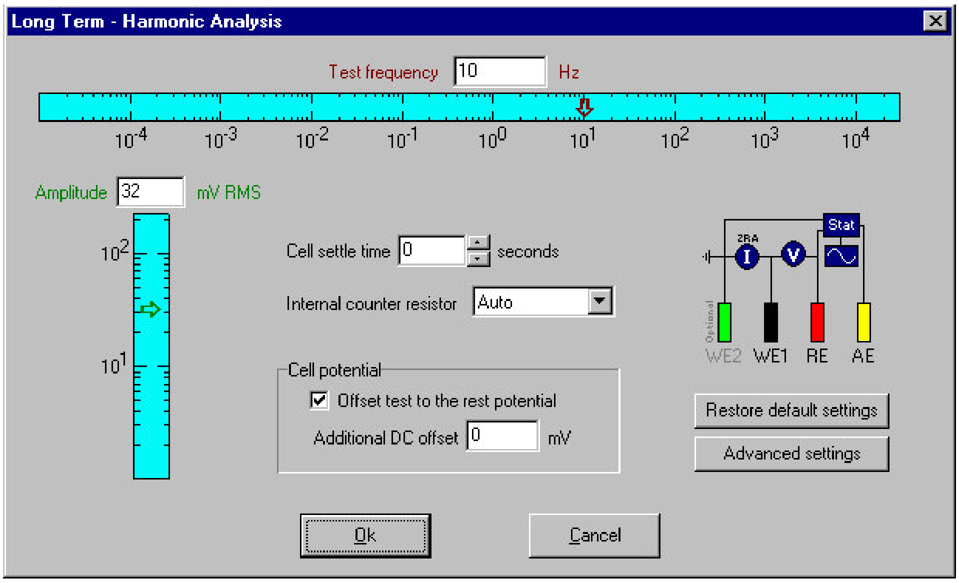
Fix on this CR ☐

Measurement averaging: Normal

Galvanic Measurement should only be placed within a long term folder. Measurement averages are the same as the potential measurement table above.

*Couple Aux Electrode to WE2* allows 3 electrode cells to record galvanic measurements, the AE connection normally used to polarise the test can be switched to act as WE2, so WE2 does not have to be connected.

# Harmonic Analysis



Long Term - Harmonic Analysis

Test frequency 10 Hz

Amplitude 32 mV RMS

Cell settle time 0 seconds

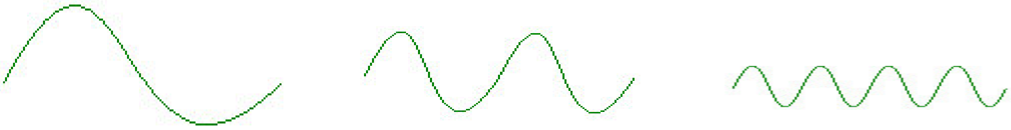
Internal counter resistor Auto

Cell potential

☑ Offset test to the rest potential

Additional DC offset 0 mV

Harmonic analysis applies a sine wave at a predefined frequency. From the collected results first, second and third harmonics are extrapolated. A corrosion current and Tafel slope are then calculated from the three harmonic results.



First Harmonic                Second Harmonic
Third Harmonic

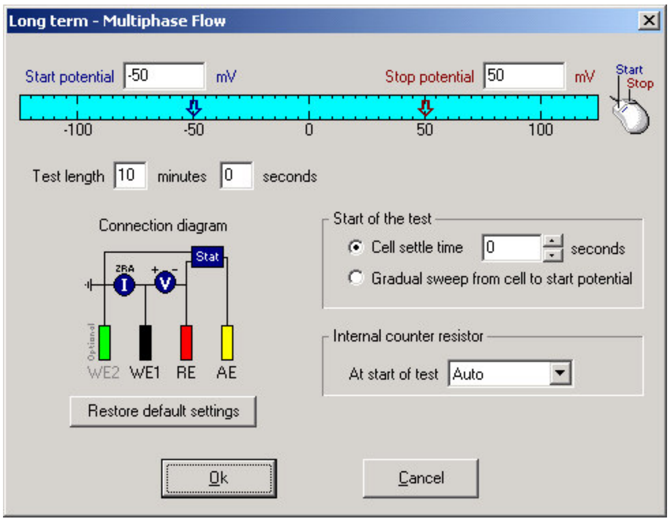
The *Amplitude* used for the AC signal plays an important role in obtaining the best possible results, always use the highest amplitude possible, a test at 30mV AC will yield better results than a test at 4mV AC.

The cell potential applied to the cell is affected by two options, firstly is the option *Offset test to the rest potential* that biases the test around the rest potential if checked, the second option allows a DC Offset to be applied (with respect to RE). An example of both options, a cell has a rest potential of 133mV, if *Offset test to the rest potential* is checked and there is a DC Offset of 200mV to be applied then the test would be done at 333mV.

Advanced Settings

Each instrument undergoes a very extensive calibration procedure over a series of 8 decades of cell load, 10 amplitudes and the full range of frequencies to produce a calibration matrix we call the P.A.I.R. table (Phase and Impedance Reduction). This is then applied to the raw data to reduce cable and instrument induced errors. The PAIR option is on by default, it can be accessed on the Advanced options page.

# Long Term – Multiphase Flow



Long term - Multiphase Flow

Start potential -50 mV

Stop potential 50 mV

Test length 10 minutes 0 seconds

Start of the test

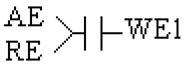
● Cell settle time 0 seconds

○ Gradual sweep from cell to start potential

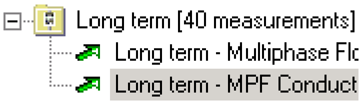
Internal counter resistor

At start of test Auto

A *Long Term – Multiphase Flow* and it's sister test *Long Term – MPF Conductive Solution CR (Corrosion Rate)* can be used together to give a Corrosion Rate and a Solution Corrosion Rate result. This test should be performed with two electrodes only, wired as:



To correctly setup a Multiphase Flow Test, first place the *Long Term – Multiphase Flow* into a *Long Term* Folder, followed by a *Long Term – MPF Conductive Solution CR technique:*



Long term [40 measurements]

Long term - Multiphase Flc

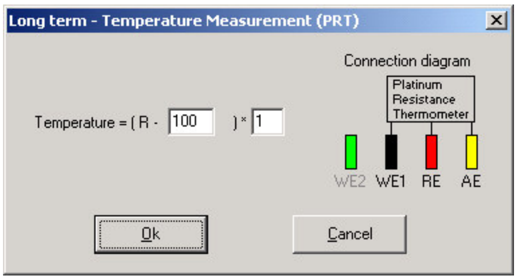
Long term - MPF Conduct

The *Long Term – MPF Conductive Solution CR* technique does not run a test, it uses the data provided by the proceeding *Long Term – Multiphase Flow* technique to calculate a Solution Corrosion Rate.

Test Notes

As with 2 electrode LPR type tests the polarisation is effectively halved, the current has to pass through two interfaced layers associated with each electrode. To mathematically compensate for this, half the electrode area can be used. Selection of the counter resistor is more critical for this type of test, as the current value can quickly flip from a practical zero, to a significant value. It is best therefore to use a fixed resistor which will cover the larger magnitude of currents required. "Auto" endeavours to do this, however if 'flat' results are returned (indicative of over-range), select a fixed resistor, 1Kohms may well be a good starting point. Lower resistor values should be used for higher corrosion currents, higher ones for lower corrosion currents.

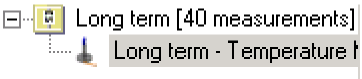
# Long Term – Temperature Measurement (PRT)



Long term - Temperature Measurement (PRT)

Temperature = ( R - 100 ) * 1

A Platinum Resistance Thermometer (PRT) is used to accurately measure temperature. The PRT test can be placed inside a Long Term Folder, to collect data over time:



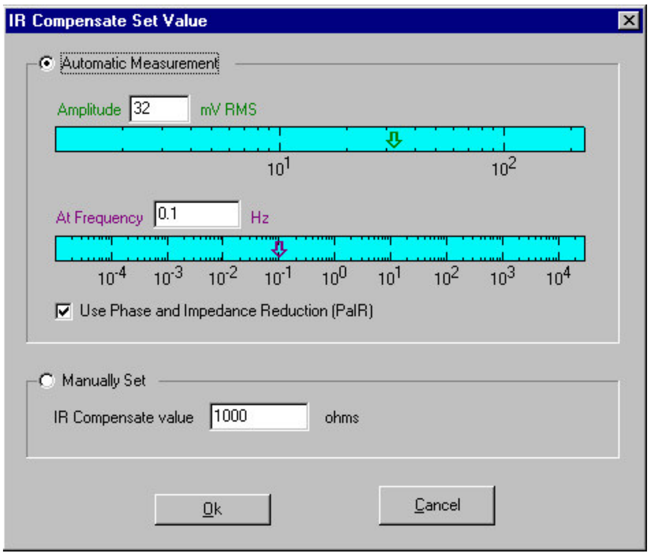
Long term [40 measurements]
Long term - Temperature I

Should calibration of the final temperature be required, calibration values can be entered on the setup page. A PRT module outputs it’s temperature as impedance, with a 100 ohms offset. Each 0.385 ohm increase above 100 ohms counts for 1 Degree Celsius.

# IR Compensation

Polarisation tests (cyclic sweep, custom sweep, LPR sweep & LPR step) may be compensated by a measured, or a manually set IR compensation value. IR compensation is typically used with high impedance test subjects, such as concrete samples.

Before IR compensated techniques can be used, a compensation value has to be specified, this is done by adding the technique *IR compensate – set value*, before any IR compensation techniques:



IR Compensate Set Value

● Automatic Measurement

Amplitude 32 mV RMS

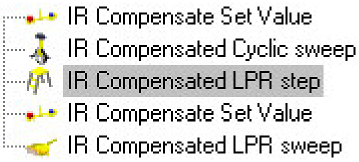
At Frequency 0.1 Hz

☑ Use Phase and Impedance Reduction (PaIR)

○ Manually Set

IR Compensate value 1000 ohms

After *IR compensation set value* has been added to the sequence list, subsequent IR compensated tests will use the last set IR compensation value, for example:



IR Compensate Set Value
IR Compensated Cyclic sweep
IR Compensated LPR step
IR Compensate Set Value
IR Compensated LPR sweep

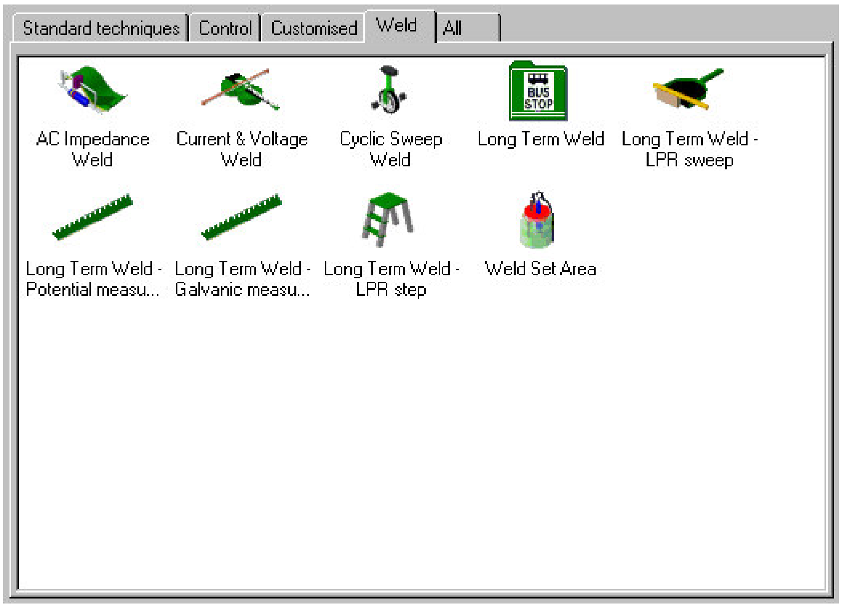
The *Cyclic sweep* and *LPR step* techniques will be compensated by the *IR compensate set value* in position 1, whilst *LPR sweep* will be compensated by the value set in position 4.

### IR Compensated Techniques

The various IR compensated techniques are identical in all but two respects to their standard counter parts:

1. No over sample option is available for IR compensated techniques,
2. There is no gradual delay sweep option at the beginning of the test.

# Weld Option



Weld techniques perform tests simultaneously on multiple ZRAs (one to five). The above weld techniques, as found on the *Weld* tab in the sequencer are identical to their counter parts in *Standard techniques*, with two exceptions:

1. Areas – Before using any Weld techniques a *Weld Set Area* technique must be placed somewhere in the sequence list before the weld technique. The *Weld Set Area* sets the area for subsequent Weld type tests. The area setting on the sequencer page is not used by weld techniques.

2. Oversampling – There is no oversampling option for weld techniques, as a result any data collected will be less smooth than if collected by one of the standard techniques.

It is also worth noting that Weld Long Term techniques and Standard Long Term techniques are not interchangeable with each other.

### WE2(s) and the Weld Tester

On a Gill 12 Weld tester there are two WE2's, each one performs a different role. A normal Gill 12 instrument has only one WE2 (WE2 on the Gill8/12 connector on a Weld instrument), it is the instrument ground and is used to take galvanic measurements.

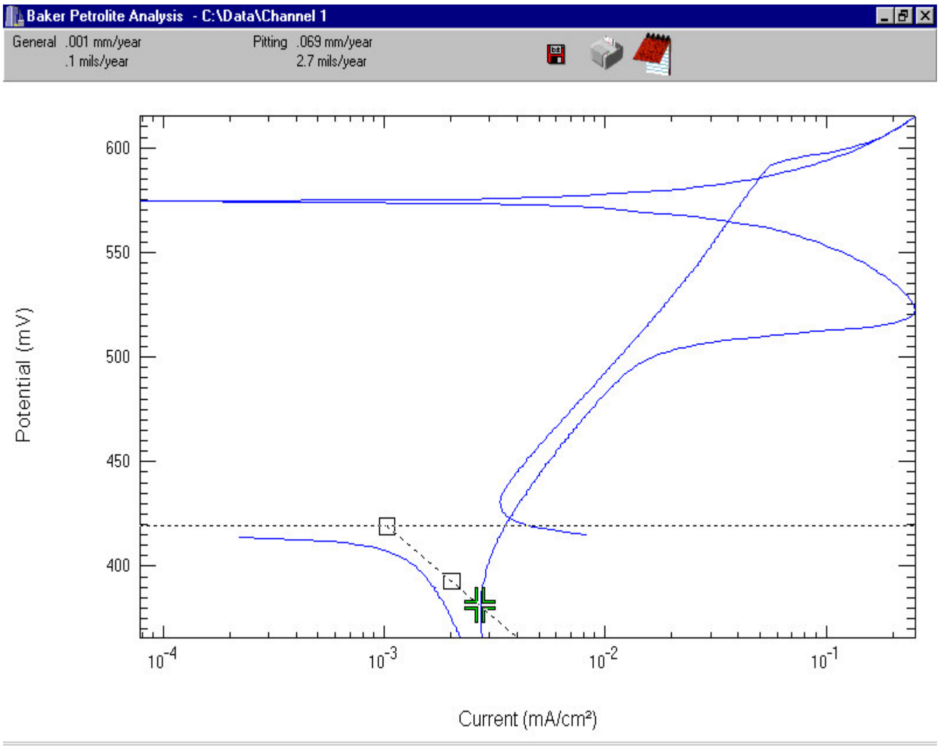
On a weld tester there is also a WE2 on the Weld connector, this is ZRA 2's equivalent of ZRA 1's WE1.

## Long Term – LPR Corrosion Rate Step

Long Term – LPR Corrosion Rate Step can be found in the Customised Tab, place this test within a Long Term folder to collect data against time. Use the DC Analysis analyse Corrosion Rate Step data.

## Baker Petrolite Sweep

Each time this test completes an analysis page is automatically launched, to view the data at a later date, load the sequence into the DC Analysis, and double click on one of the mini graphs.

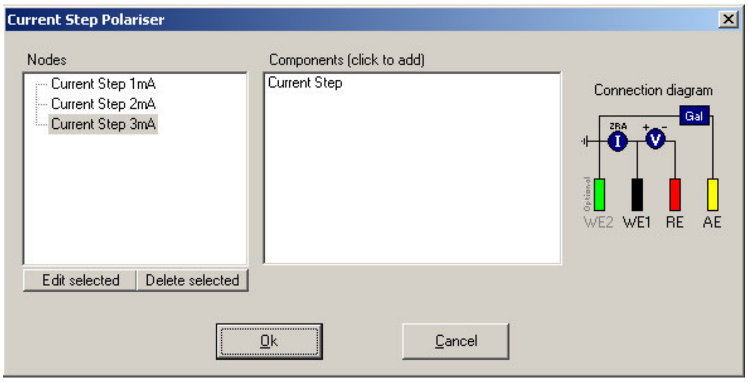

Baker Petrolite Analysis - C:\Data\Channel 1

General .001 mm/year
.1 mils/year

Pitting .069 mm/year
2.7 mils/year

Position the mouse above the Rest Potential line, and drag it up or down to move it. The intersection point is highlighted by the green cross, the intersection line can be positioned by clicking and dragging the mouse in one of two positioning squares shown on the line.

# Current Step Polariser

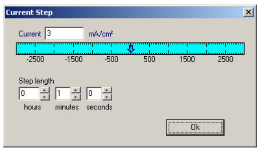


Nodes

- Current Step 1mA
- Current Step 2mA
- Current Step 3mA

Components (click to add)

- Current Step

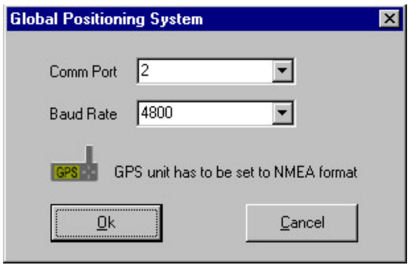


Current: 3 mA/cm²

Step length: 0 hours, 1 minutes, 0 seconds

The current step polariser is constructed from *Nodes*, each node represents a current step for a length of time. To add a *Node* click one of the available *Components*:

## Global Positioning System



**Global Positioning System**

Comm Port: 2

Baud Rate: 4800

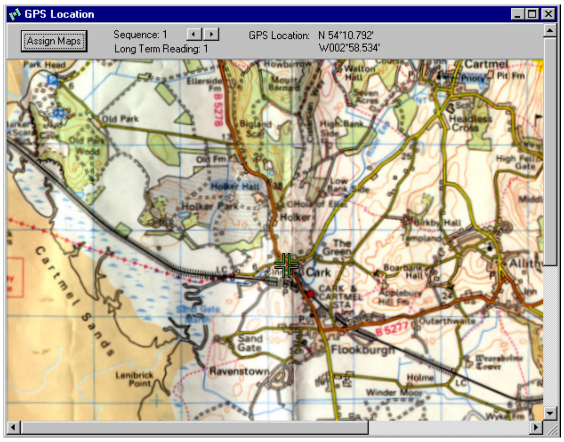
GPS unit has to be set to NMEA format

With GPS, a location (longitude and latitude) can be stored prior to commencing other techniques. Later on when analysing the data the location can be extrapolated, and optionally plotted on a map.

The majority of GPS units available will be compatible with the core running, the GPS unit has to be able to connect via a serial link to the PC, also the GPS output format be set to **NMEA**. When adding the GPS technique to the sequence list, Comm Port and Baud Rate (usually 4800) should be set to match the GPS unit.

### Analysing Locations

A Map can be scanned into the computer (Bitmap - .Bmp or Jpeg - .Jpg) and be used to plot the location:



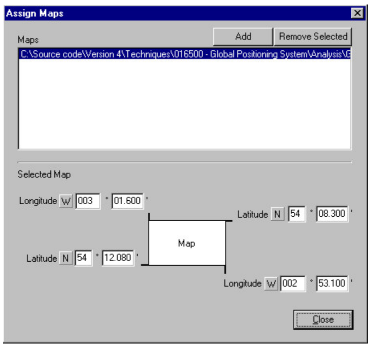
**GPS Location**

Sequence: 1

Long Term Reading: 1

GPS Location: N 54°10.792' W002°58.534'

To add a Map, click on the ‘Assign Maps’ button, and press the ‘Add’ button on the *Assign Maps* page. When a map has been added select it in the *Maps* list section and set the maximum / minimum longitude and latitude settings for the edges of the map. After map(s) have been added the program will automatically search them to find one suitable to plot the location onto (location is shown as a slowly flashing green cross). Maps only have to be added once and are remembered through subsequent analysis loads.



**Assign Maps**

Maps

C:\Source code\Version 4\Techniques\016500 - Global Positioning System\Analysis\G

Selected Map

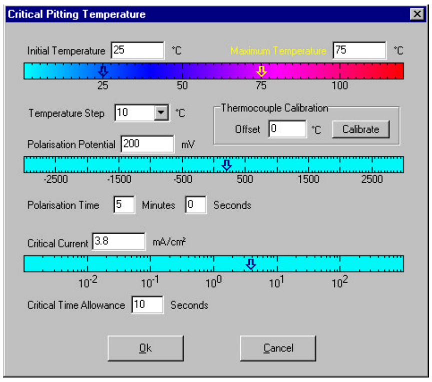
Longitude W 003 ° 01.600 '

Latitude N 54 ° 08.300 '

Latitude N 54 ° 12.080 '

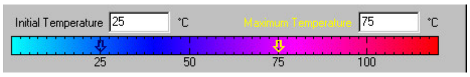
Longitude W 002 ° 53.100 '

# Critical Pitting Temperature



Initial Temperature 25 °C
Maximum Temperature 75 °C
Temperature Step 10 °C
Offset 0 °C
Polarisation Potential 200 mV
Polarisation Time 5 Minutes 0 Seconds
Critical Current 3.8 mA/cm²
Critical Time Allowance 10 Seconds

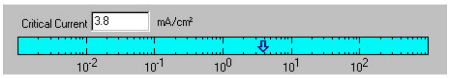
By stepping the temperature, a critical pitting temperature can be discovered. Before using the Critical Pitting Temperature test, make sure that the external Critical Pitting Temperature hardware is connected correctly to the instrument.



Initial Temperature 25 °C
Maximum Temperature 75 °C

At the top of the page is where the test temperature is set, an *initial temperature* and a *maximum temperature*. Below the temperature scale is *Temperature Step*, in the above example the test would go 25, 35, 45, 55, 66, 75 degrees C.

When the initial Temperature is reached the test can commence, the cell is offset to the *Polarisation Potential* for the *Polarisation Time* specified, it is worth noting that the *Polarisation Potential* is an absolute potential and is not offset by the rest potential.

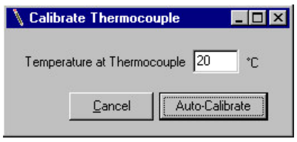


Critical Current 3.8 mA/cm²

A *Critical Current* (area corrected) is set along with a *Critical Time Allowance*, this ends the test if the current limit is reached for the set time.

Thermocouple Calibration

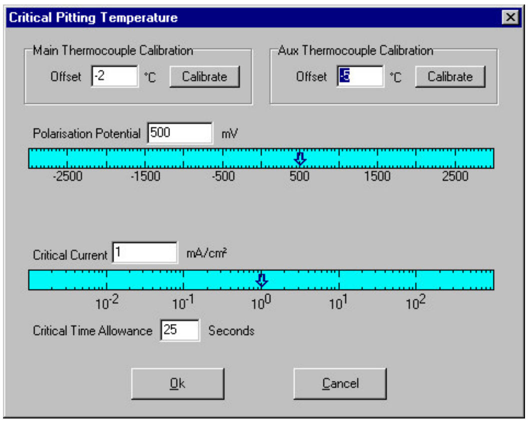
Thermocouples can have slight calibration errors, to correct this there is a calibration section accessible with the *Calibrate* button.



Temperature at Thermocouple 20 °C

To calibrate the thermocouple, first measure the temperature at the thermocouple with a thermometer, type the measured value in to the text box and press the *Auto-Calibrate* button, the instrument will calculate the exact offset value.

# Dual CPT



Main Thermocouple Calibration: Offset -2 °C

Aux Thermocouple Calibration: Offset -5 °C

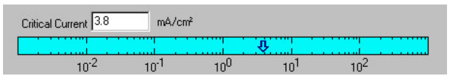
Polarisation Potential 500 mV

Critical Current 1 mA/cm²

Critical Time Allowance 25 Seconds

By stepping the temperature, a critical pitting temperature can be discovered. Before using the Critical Pitting Temperature test, make sure that the external Critical Pitting Temperature hardware is connected correctly to the instrument.

At the start of the test, the cell is offset to the *Polarisation Potential*, it is worth noting that the *Polarisation Potential* is an absolute potential and is not offset by the rest potential. The test continues until the critical current is passed, or the Finish Test button is pressed.

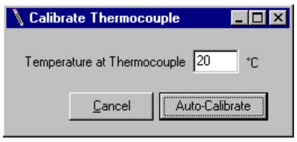


Critical Current 3.8 mA/cm²

A *Critical Current* (area corrected) is set along with a *Critical Time Allowance*, this ends the test if the critical current limit is reached for the set time. *Critical Time Allowance* is accumulative, for example, if this value was set to 10 seconds and the *Critical Current* was passed for 7 seconds, then fell below the critical current, it would only require an additional 3 seconds over the critical Current to finish the test.

Thermocouple Calibration

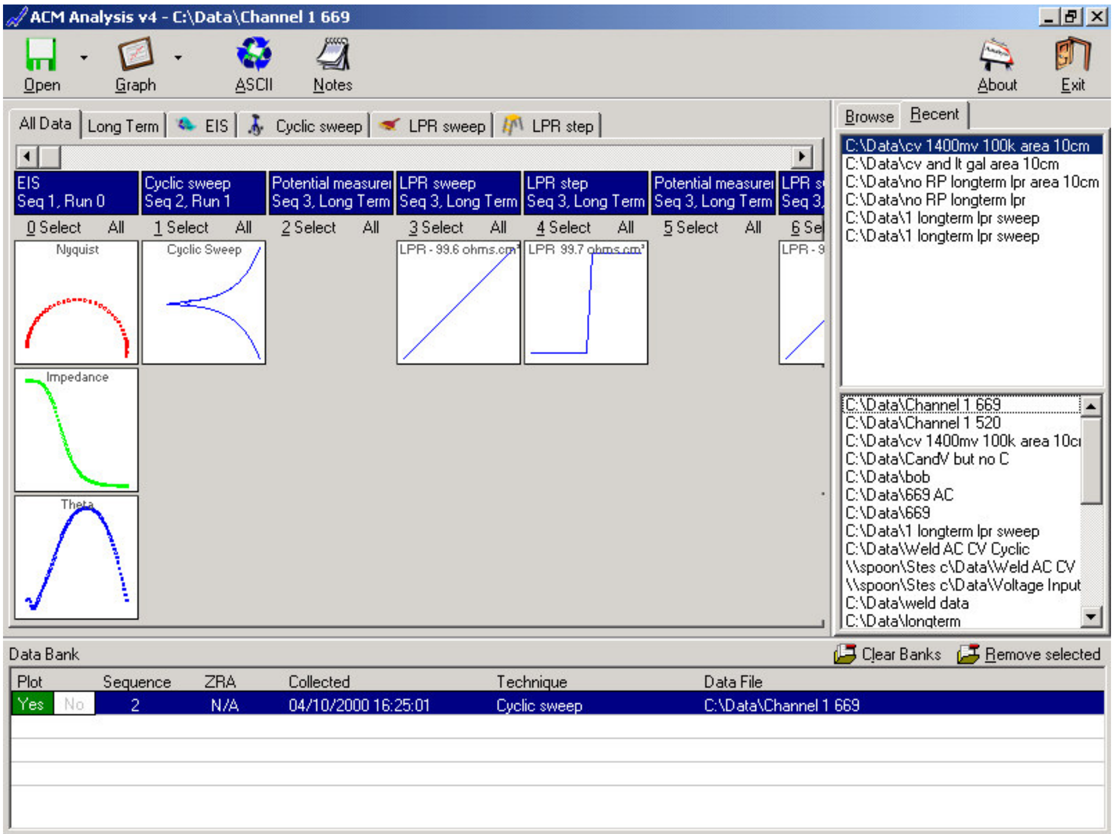
Thermocouples can have slight calibration errors, to correct this there is a calibration section accessible with the *Calibrate* button. There are two separate calibration sections at the top of the page, one for the main thermocouple and another for the auxiliary thermocouple.



Temperature at Thermocouple 20 °C

To calibrate a thermocouple, first measure the temperature at the thermocouple with a thermometer, type the measured value in to the text box and press the *Auto-Calibrate* button, the instrument will calculate the exact offset value.

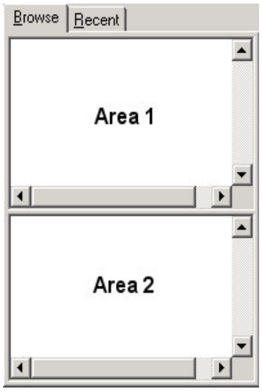
# Analysis



| Plot | Sequence | ZRA | Collected | Technique | Data File |
|---|---|---|---|---|---|
| Yes No | 2 | N/A | 04/10/2000 16:25:01 | Cyclic sweep | C:\Data\Channel 1 669 |

Above is the analysis data explorer window; it is split into 4 sections. At the top is a Tool bar, right side is the data file selection area, centre of screen is where the contents of a data file are displayed and at the bottom are data banks where individual tests are placed ready for analysis.

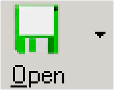
First we will look at the data file selection (right side). This is partially hidden until the mouse cursor is moved over it:



Here you will see two tabs, Browse and Recent, and two Areas.

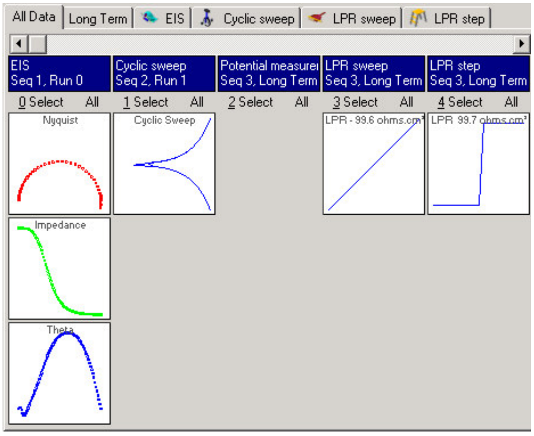
The Browse tab will display a tree view of available Drives and Folders in Area 1, Area 2 shows the contents for the presently selected folder in Area 1. To open a data file select a folder in Area 1 and click on the required file in Area 2

The Recent tab displays the last files stored by the Core running in Area 1 and a history of files previously opened in Area 2. To open a file displayed in Area 1 or 2 simply click it.



The 'Open' button on the tool bar can also be used to select a file. Unlike the Browse option above, network drives do not need to be mapped for data to be accessed. Next to the open button is a drop down arrow that produces a list of the last data files recorded.

Once a data file is opened its contents are displayed:



At the top there are several tabs showing different types of data contained within the file.

Data from individual tabs can be viewed all together by using ‘All Data’ Tab or a specific technique can be displayed by selecting the required Tab.

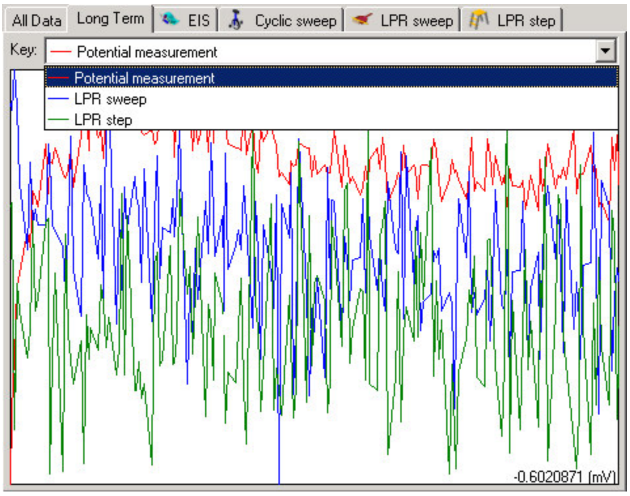
Below these Tabs is a scroll bar for when the number of tests within a data file exceeds the displayable number.

Individual tests are displayed in columns. At the top of the column is a text area showing information about the test. Each test can contain up to three thumbnail graphs previewing data stored. Each column also contains selection buttons, ‘Select’ which enters the data from the column into data bank and ‘All’ which places all data from the same technique into data banks. However, some techniques are treated differently with the ‘All’ option, techniques that contain data which can not produce a graph individually (Harmonic or a single potential or current measurement, etc.) will produce a measurement Vs time graph for any values stored. For Current and Voltage data the ‘All’ option adds all individual tests end to end and also allows trend graphs to be drawn (e.g., Delta I Vs time, Delta V Vs time, etc).

Data may also be entered into data banks by dragging one of the thumbnail graphs down to one data bank.

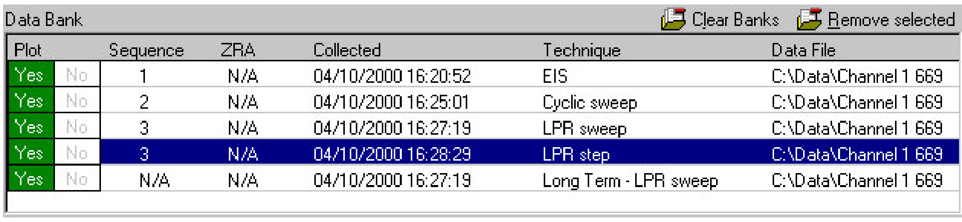
Some Custom techniques have their own analysis software; in these cases double clicking one of the thumbnail graphs will launch the Custom analysis software. Normally double clicking a thumbnail graph will place it into the data banks.

Long term and Voltage input data are treated differently:



Selecting long term or voltage input tabs displays a graph containing all the relevant data. A drop down list containing a colour key is used to identify each type of data and also decides which data type is added to the data bank. To select data, first select the type of data from the Key and then drag the graph into the data bank area.

Data banks contain the data to be displayed:



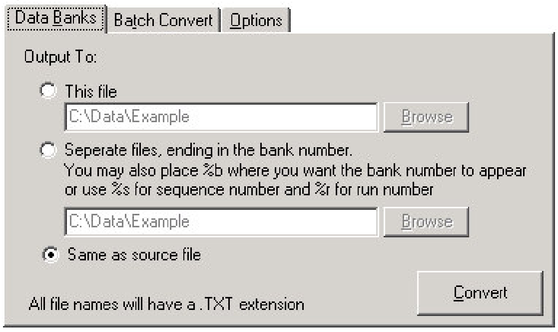
| Plot | | Sequence | ZRA | Collected | Technique | Data File |
|---|---|---|---|---|---|---|
| Yes | No | 1 | N/A | 04/10/2000 16:20:52 | EIS | C:\Data\Channel 1 669 |
| Yes | No | 2 | N/A | 04/10/2000 16:25:01 | Cyclic sweep | C:\Data\Channel 1 669 |
| Yes | No | 3 | N/A | 04/10/2000 16:27:19 | LPR sweep | C:\Data\Channel 1 669 |
| Yes | No | 3 | N/A | 04/10/2000 16:28:29 | LPR step | C:\Data\Channel 1 669 |
| Yes | No | N/A | N/A | 04/10/2000 16:27:19 | Long Term - LPR sweep | C:\Data\Channel 1 669 |

To remove a single test, select it and click ‘Remove Selected’.

To remove all tests from the data bank select ‘Clear Banks’ option.

The tool bar contains the available options:

ASCII allows data to be converted to a file suitable for spread sheets and other analysis software. This option has three sections:


Data Banks | Batch Convert | Options

Output To:

○ This file
C:\Data\Example
Browse

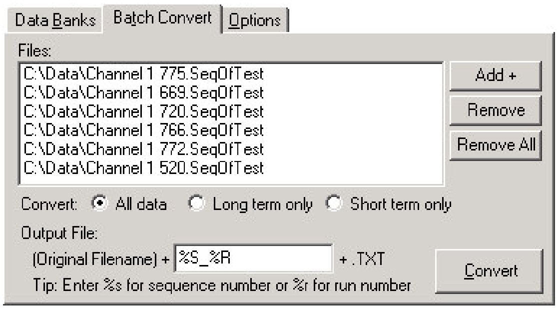
○ Seperate files, ending in the bank number.
You may also place %b where you want the bank number to appear or use %s for sequence number and %r for run number
C:\Data\Example
Browse

● Same as source file

All file names will have a .TXT extension

Convert

**Data Banks** convert tests currently in the data banks. All files created will have the file extension of .TXT. There are three options which decide the filename. **This File** where all data is stored in the same file, **Separate files** where the filename ends in the bank number or bank number (%b), sequence number (%s) or run number (%r) can be placed anywhere in the file name. Same as source file uses the source filename (but with the .TXT extension). Select **Convert** to start conversion.


Data Banks | Batch Convert | Options

Files:
C:\Data\Channel 1 775.SeqOfTest
C:\Data\Channel 1 669.SeqOfTest
C:\Data\Channel 1 720.SeqOfTest
C:\Data\Channel 1 766.SeqOfTest
C:\Data\Channel 1 772.SeqOfTest
C:\Data\Channel 1 520.SeqOfTest

Add +
Remove
Remove All

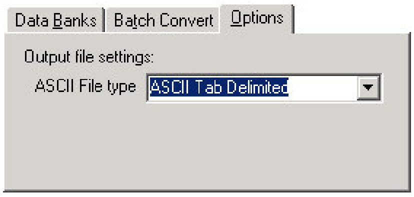
Convert: ● All data ○ Long term only ○ Short term only

Output File:
(Original Filename) + %S_%R + .TXT

Tip: Enter %s for sequence number or %r for run number

Convert

**Batch convert** enables large amounts of data to be converted to ASCII. Use **Add** to add files to the list, select unwanted files and click on **Remove** or **Remove all** to clear the list of files.
There is the ability to convert just Long term or just Short term or all types of data. Output file is the original filename with the file extension of .TXT and the ability to include sequence number (%s), run number (%r) and additional text.


Data Banks | Batch Convert | Options

Output file settings:
ASCII File type: ASCII Tab Delimited

**Options** contains settings for the output file. ASCII file types are Tab delimited (used by most packages) and Comma delimited (sometimes known as CSV).

NB, More data conversion options are available from the File and Edit menus once the data graph has been drawn.

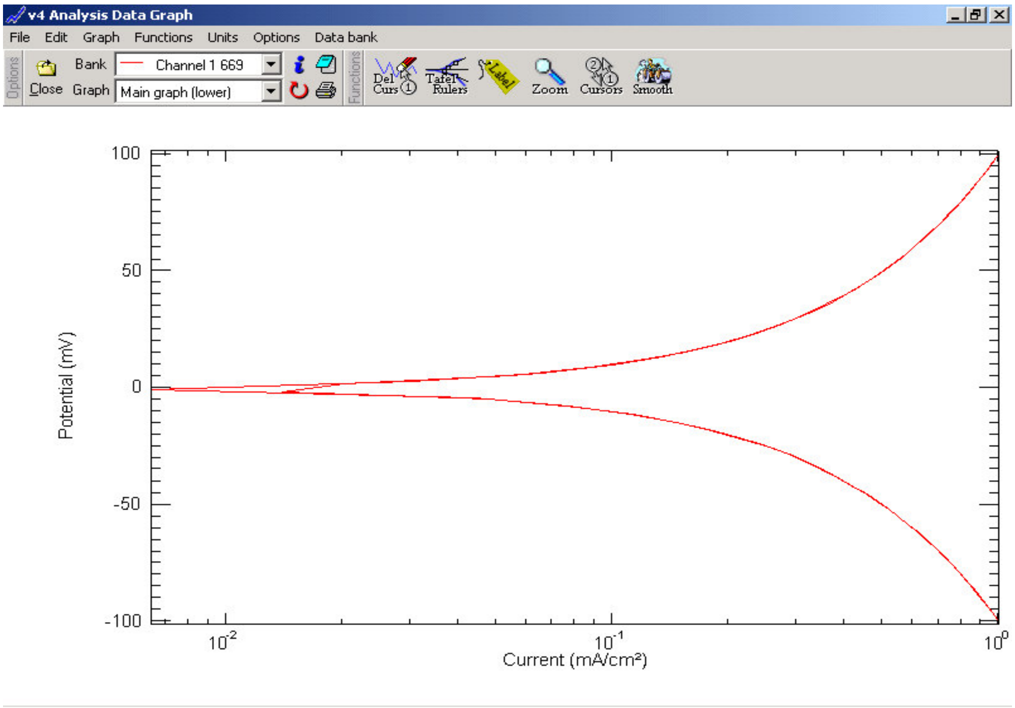
Notes displays the note book for the open data file (see the notebook section of the manual).

Graph draws the default graph data in the data banks. Selecting the down arrow next to graph displays a list of all possible graphs.

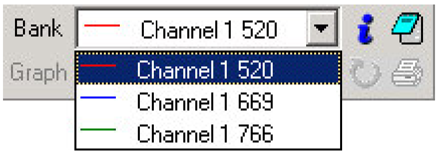
About shows the analysis version info.

Exit quits the analysis software

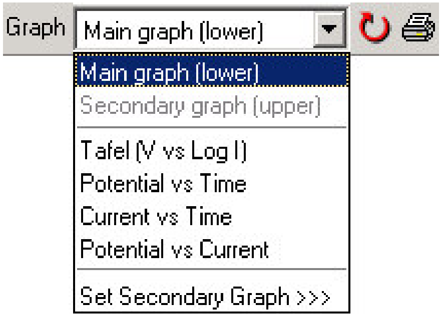
# Data Graph



A data graph is made up of three sections, a menu where functions and options can be selected, info bar where functions, information and results are shown, and the graph itself.



On the info bar is a Bank key, this can be used to select an active data bank and to identify a data set by it's colour. Clicking on the word 'Bank' allows banks settings to be edited (i.e., title, colours and markers). Clicking on ℹ displays all information associated with the selected data bank. Selecting 🗒 will display the note book for the active data bank.

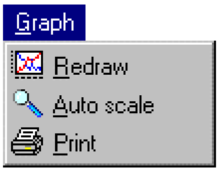


Graph contains a list of standard graphs that may be drawn. Certain standard graphs types will include two separate graphs, however on graphs which don't, a second graph may be added via the **Set Secondary Graph >>>** option. Clicking on the word **Graph** allows graph settings to be edited (i.e., axis types, colours, fonts, etc.). Clicking on the ↻ button re-draws the graph. 🖶 prints the present graph.

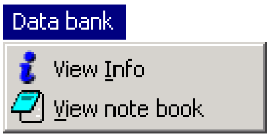
Menus:

**Save As** allows data or graph information to be saved to various formats, e.g., text, bit map, HTML, etc.
**Close graph** returns to the data file explorer page.
**About** displays the about form.
**Exit** quits the analysis software

**Copy As** copies data to the clipboard to be pasted into other programs such as MS Word or Excel.
**Revert** reloads data from disk, therefore removing any modifications to the data such as smoothing.



**Log X axis** and **Log Y axis** allow the axis to be displayed in linear or logarithmic.
**Redraw** refreshes the graph after any changes.
**Auto scale** re-scales the graph to the maximum scaling.
**Print** prints the graph to the default printer. If a special function, such as tafel rulers, is selected they will also be printed.

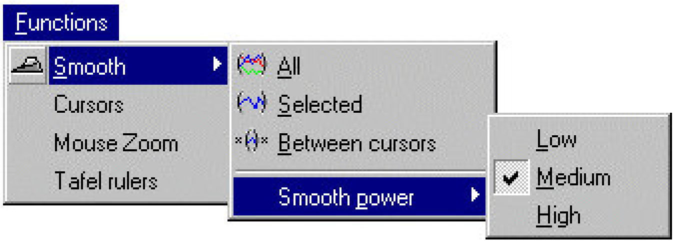


**View Data info** displays all relevant information on the selected data bank including analysis results such as Icorr and Corrosion rates.
**View Note book** displays the note book for the selected data bank.

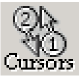
**Units** menu can be used to change displayed units. For example potential may be set to V, mV, µV or pV. Certain data types such as voltage inputs are unaffected by the units.

**Bank Settings** allows the selected banks settings to be edited (i.e., title, colours and markers).
**Graph Settings** edits graphs settings such as axis, colours, fonts, etc.
**Join gaps in data** when checked draws lines between gaps in the data. Gaps appear when data points are deleted, cyclic sweeps are reversed or long term data is appended to.
**Zero plot time axis** when checked starts all data banks from Zero on the time axis. When un-checked zero plot start each bank from its relative start time to all other data graphs start times.

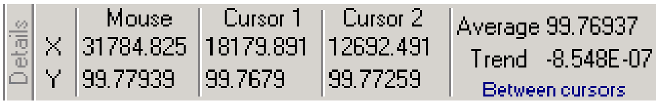
**Functions menu:**



The Smooth function can be applied to **All** data on a graph, just the **Selected** data bank or **Between** the cursors for the selected data bank.



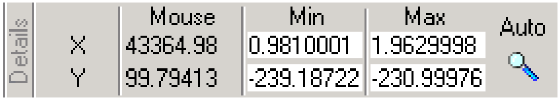
**Cursors** function places two cursors onto the selected data bank. When **Cursors** is selected, X and Y positions of both cursors and the mouse pointer are displayed on the info bar. Also shown is information relative to the present graph, usually average and trend between the cursors.



| Details | Mouse | Cursor 1 | Cursor 2 | |
|---|---|---|---|---|
| X | 31784.825 | 18179.891 | 12692.491 | Average 99.76937 |
| Y | 99.77939 | 99.7679 | 99.77259 | Trend -8.548E-07 |
| | | | | Between cursors |

Cursor 1 is be moved by clicking the left mouse button when the mouse pointer is over the required point or by using the left and right cursor keys. Cursor 2 is moved by clicking the right mouse or using the up and down cursor keys.

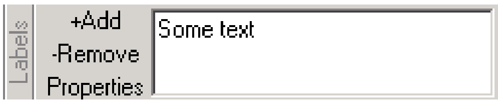
**Mouse Zoom** function is used to re-scale the graph. Simply click the mouse pointer at the top left corner of the required area and drag the mouse pointer to draw a box around the selected area. Displayed in the details section of the info bar is current X, Y, Max & Min positions of the mouse pointer. Values for the max and min X and Y axis may be typed directly into these boxes. 🔍 re-scales the graph to the new values. To scale the graph around the data select Auto followed by 🔍.



| Details | Mouse | Min | Max | Auto |
|---|---|---|---|---|
| X | 43364.98 | 0.9810001 | 1.9629998 | 🔍 |
| Y | 99.79413 | -239.18722 | -230.99976 | |

**Del Curs (1)** deletes the data point at cursor 1. To recover deleted data points select Revert under the Edit menu. Only data in memory is deleted and not data in the data file.

Label enables labels to be placed onto the graph. Click on the graph where the label is required and type some text. A yellow background highlights the selected label. Labels may be ‘dragged’ anywhere on the graph.
On the info bar a labels section is shown. Here text can be edited, label removed or new labels added. Select the **Properties** option to change the font, size and colour of the selected label.



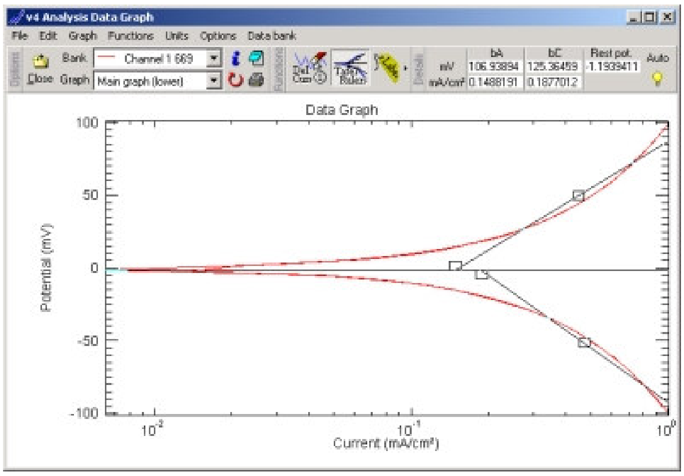
Some text

Other functions are available which are data/graph type specific. These include:

**Point to point** is available with current and voltage data only. Selecting point to point calculates the difference between one point and the next and displays the result. To revert back to the original data select Revert under the Edit menu.

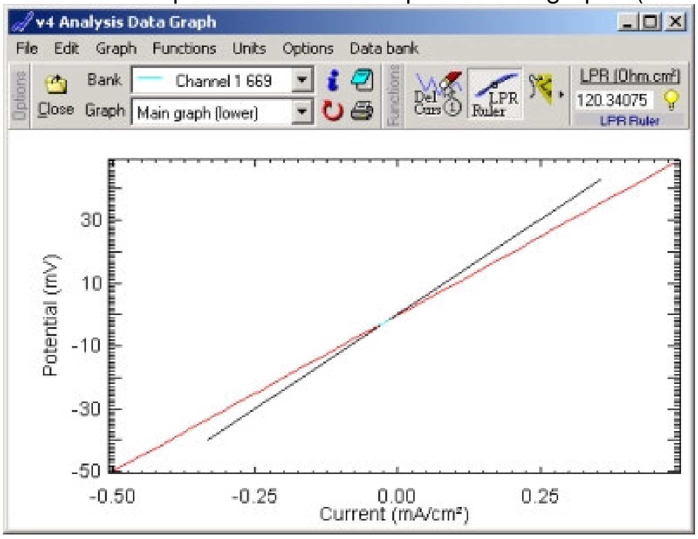
**Load Cur(1)** is available for Long term LPR/Corrosion rate graphs recorded with the Long term LPR Sweep or Step techniques. When selected the LPR Sweep or Step for the data point at cursor 1 is loaded and displayed.

**Tafel Rulers** function is specific to Custom, Cyclic, Galvanic and LPR Sweeps. Tafel rulers can only be used on potential Vs current graphs where the current axis is Logarithmic. Three rulers are placed on the graph, a horizontal ruler (vertical if the X axis is potential) which identifies the rest potential. This can be moved by placing the mouse pointer over the line until it changes to a ⇕ cursor (⇔ if the X axis is potential), press and hold the left mouse button down and drag the line to its new position. The other rulers indicate tafel slopes. These can be adjusted by moving the mouse pointer over a square until the mouse pointer changes to ⇔( ⇕ if the X axis is potential), then press and hold the left mouse button and drag the line to its new position. Use the 💡 option to remember these tafel slopes. Corrosion rate will be calculated and displayed in the Data Info section (see **Data bank** menu, **View Info**). Auto automatically fits rulers to the slopes.



| | bA | bC | Rest pot. |
|---|---|---|---|
| mV | 106.93894 | 125.36459 | -1.1939411 |
| mA/cm² | 0.1488191 | 0.1877012 | |

**LPR Ruler** is specific to LPR sweep and Tafel graphs (on linear axis only). A Ruler is drawn that can be positioned by use of the mouse pointer and the left & right mouse buttons. The result is displayed in real time. Use 💡 to remember the LPR result (for the selected data bank). Corrosion rate will be calculated and displayed in the Data Info section (see **Data bank** menu, **View Info**).

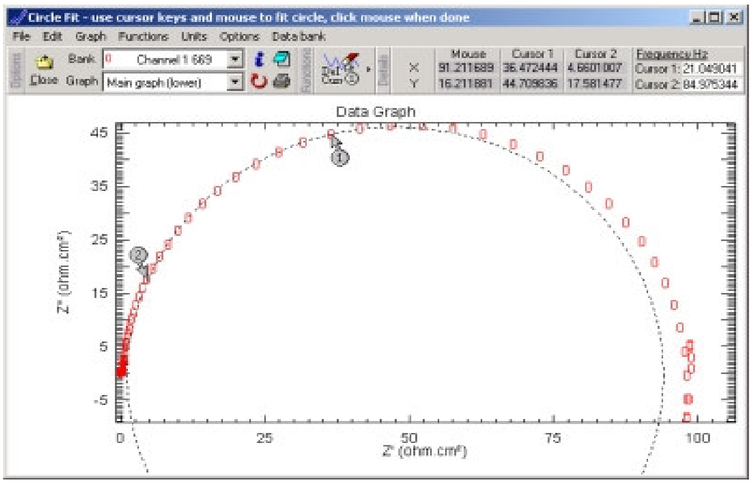


Bank: Channel 1 669

Graph: Main graph (lower)

LPR (Ohm.cm²): 120.34075

**Circle fit** is specific to Nyquist graphs. First select **Cursor** and place two cursors on the arc, then select **Circle fit**, use the mouse pointer as the third point of the circle adjusting it until the circle best fits the data and click the mouse button to calculate the result. While fitting you may reposition cursor 1 and 2 by using the cursor keys.



Circle Fit - use cursor keys and mouse to fit circle, click mouse when done

Bank: 0 Channel 1 669

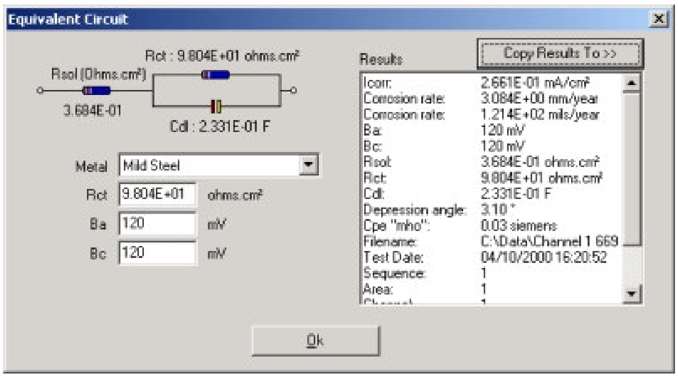
Graph: Main graph (lower)

| | Mouse | Cursor 1 | Cursor 2 |
|---|---|---|---|
| X | 91.211689 | 36.472444 | 4.6601007 |
| Y | 16.211881 | 44.709836 | 17.581477 |

Frequency Hz

Cursor 1: 21.049041

Cursor 2: 84.975344



Equivalent Circuit

Rsol (Ohms.cm²) 3.684E-01

Rct : 9.804E+01 ohms.cm²

Cdl : 2.331E-01 F

Metal: Mild Steel

Rct: 9.804E+01 ohms.cm²

Ba: 120 mV

Bc: 120 mV

Results

| | |
|---|---|
| Icorr: | 2.661E-01 mA/cm² |
| Corrosion rate: | 3.084E+00 mm/year |
| Corrosion rate: | 1.214E+02 mils/year |
| Ba: | 120 mV |
| Bc: | 120 mV |
| Rsol: | 3.684E-01 ohms.cm² |
| Rct: | 9.804E+01 ohms.cm² |
| Cdl: | 2.331E-01 F |
| Depression angle: | 3.10 ° |
| Cpe "mho": | 0.03 siemens |
| Filename: | C:\Data\Channel 1 669 |
| Test Date: | 04/10/2000 16:20:52 |
| Sequence: | 1 |
| Area: | 1 |

Once calculated, **Equivalent circuit** is displayed. The results can then be copied to Printer, Excel file, Text file or to the Clipboard.

**Nyquist** functions are specific to Nyquist Graphs. **Isotropic Scale X and Y** uses the X or Y axis to keep the aspect correct. Isotropic scaling is useful if the nyquist plot has been zoomed and the aspect of the X and Y axis are incorrect. **Zero Plot XY** sets the minimum values for the X and Y axis to Zero (Isotropic should be used after Zero plot).

# Analysis How To

1) How to store a graph for duplication at a later date.

   Once you have created your graph, save the graph as a Metafile (see **File** menu - **Save as** - **Metafile**). To print the graph again load the meta file into a package which support them (e.g., Microsoft's Word) and print the file.

2) How to include a graph in a report.

   Draw the graph and select **Copy As** – **Metafile** form the **Edit** menu. In your report package (e.g., Microsoft's Word) create a new document or open an existing one and select **Paste** from its **Edit** menu. For packages that don't support meta files use **Copy as** – **BitMap**.

3) How to include raw data in a report.

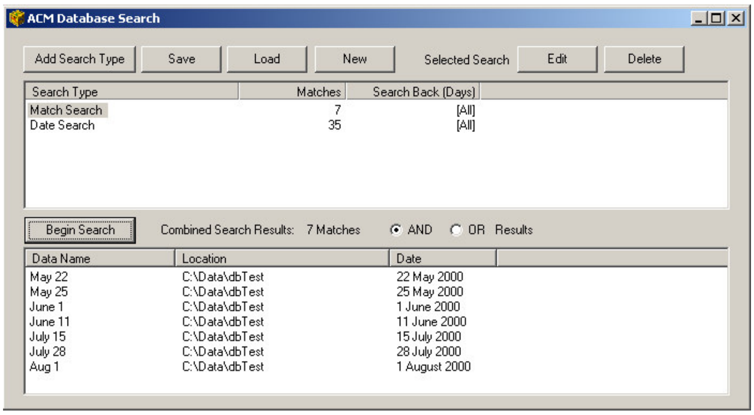
   As 2) but select **Copy as** - **Tab separated text** for spread sheets or **Copy as** – **Rich Text** for word processors.

4) How to save a graph for use in a web page.

   For just the graph select **Save as** – **BitMap** form the **File menu** and then import this file into your Web page editor. For the raw data select **Save as – HTML Grid**, and for both the graph and raw data select **Save as – HTML Report**.

# Notebook Database Search

Database searches can be performed across notebooks using previously collected data. Each time a sequence is started, the notebook for the test is stored in a database. The *ACM Database Search* program is able to search the database, making finding data easier:

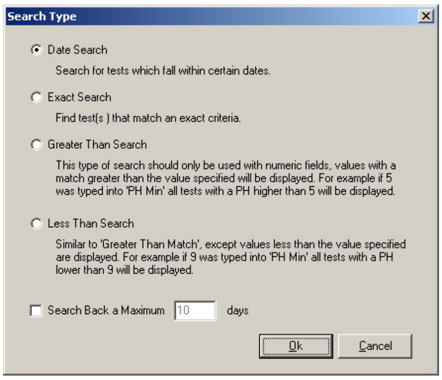


| Search Type | Matches | Search Back (Days) |
|---|---|---|
| Match Search | 7 | [All] |
| Date Search | 35 | [All] |

Combined Search Results: 7 Matches ● AND ○ OR Results

| Data Name | Location | Date |
|---|---|---|
| May 22 | C:\Data\dbTest | 22 May 2000 |
| May 25 | C:\Data\dbTest | 25 May 2000 |
| June 1 | C:\Data\dbTest | 1 June 2000 |
| June 11 | C:\Data\dbTest | 11 June 2000 |
| July 15 | C:\Data\dbTest | 15 July 2000 |
| July 28 | C:\Data\dbTest | 28 July 2000 |
| Aug 1 | C:\Data\dbTest | 1 August 2000 |

Searches can be built up from more than one search type, and the results combined across searches. A new search is added with the *Add Search Type* button:



● Date Search

Search for tests which fall within certain dates.

○ Exact Search

Find test(s ) that match an exact criteria.

○ Greater Than Search

This type of search should only be used with numeric fields, values with a match greater than the value specified will be displayed. For example if 5 was typed into 'PH Min' all tests with a PH higher than 5 will be displayed.

○ Less Than Search

Similar to 'Greater Than Match', except values less than the value specified are displayed. For example if 9 was typed into 'PH Min' all tests with a PH lower than 9 will be displayed.

☐ Search Back a Maximum 10 days

There are 4 different search types:

| | |
|---|---|
| *Date Search* | - data collected between a specified start and end date is be found. |
| *Exact Search* | - After selection an empty 'Notebook' will appear, edit the field(s) in this notebook that will be sought in the database (leave the rest blank). When searching for text, short key words can be used. |

*Greater & Less Than Search* – Numeric fields can be searched on a greater than, or less than basis. Using 2 searches (a Greater Than and a Less Than search) enables records that fall in a certain range to be found, for example: doing a Greater Than search on a Temperature of 16, and a Less Than search on a Temperature of 26 will find records with a temperature greater than 16, but less than 26.

Once the search type(s) have been selected the search can be carried out by selecting *Begin Search*, results from the search are be displayed in the lower list. Double clicking on a search result loads that data into the Analysis. Right clicking on a result gives the option of displaying / editing the Notebook. Results from each search type can be combined in two ways *And / Or*, when *And*ing results, only records present in each search are displayed, whereas *Or*ing will display each unique result.

# Calculating corrosion rates

Corrosion rates are calculated from icorr (corrosion current) multiplied by a Metal factor. The following formulas are used :

$$\text{mm / year corrosion rate} = \frac{\text{Metal factor x icorr}}{1000}$$

$$\text{mils / year corrosion rate} = \frac{\text{Metal factor x icorr}}{25.4}$$

where Icorr is calculated from:

$$\text{icorr (A/m}^2\text{)} = \frac{\text{ba x bc}}{2.3 \text{ x Rp x (ba + bc)}}$$

ba is anodic Tafel slope in Volts
bc is the cathodic Tafel slope in Volts
Rp is the polarisation resistance in $\Omega/m^2$

Metal factor is calculated from:

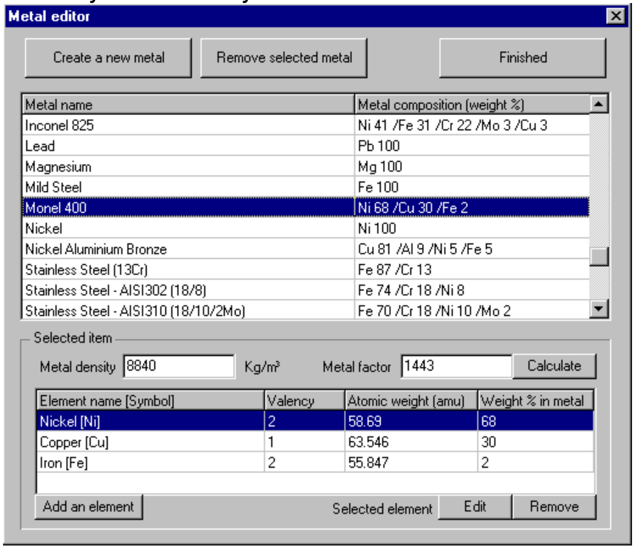
$$\text{Metal factor} = \frac{\text{t x K}}{\rho}$$

t (seconds in year) = 365.2422454 * 24 * 60 * 60 = 31556930 seconds
ρ is the metal density in $g/cm^2$
K is the electrochemical equivalent in g / coulombs, K is obtained from

$$\sum \quad K = \frac{\text{atomic \% of element x atomic weight of element}}{96487 \text{ x Valency of element}}$$

## Metal editor

The metal editor enables metal factors to be created from known compositions of metals or alloys, properties of the most popular metals and alloys are already included:



| Metal name | Metal composition (weight %) |
|---|---|
| Inconel 825 | Ni 41 /Fe 31 /Cr 22 /Mo 3 /Cu 3 |
| Lead | Pb 100 |
| Magnesium | Mg 100 |
| Mild Steel | Fe 100 |
| Monel 400 | Ni 68 /Cu 30 /Fe 2 |
| Nickel | Ni 100 |
| Nickel Aluminium Bronze | Cu 81 /Al 9 /Ni 5 /Fe 5 |
| Stainless Steel (13Cr) | Fe 87 /Cr 13 |
| Stainless Steel - AISI302 (18/8) | Fe 74 /Cr 18 /Ni 8 |
| Stainless Steel - AISI310 (18/10/2Mo) | Fe 70 /Cr 18 /Ni 10 /Mo 2 |

Metal density 8840 Kg/m³ Metal factor 1443

| Element name (Symbol) | Valency | Atomic weight (amu) | Weight % in metal |
|---|---|---|---|
| Nickel (Ni) | 2 | 58.69 | 68 |
| Copper (Cu) | 1 | 63.546 | 30 |
| Iron (Fe) | 2 | 55.847 | 2 |

## Creating a new metal

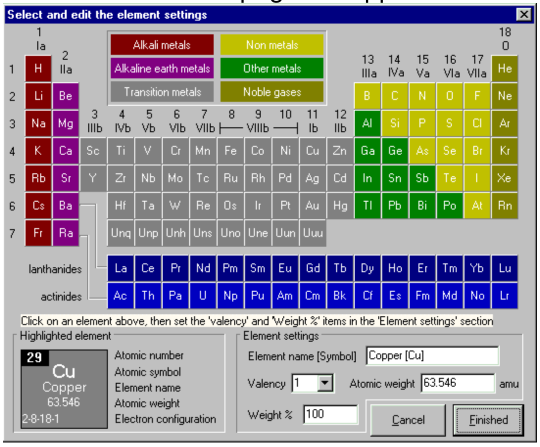
If a metal is not listed it can be created, the following illustration will create Brass 70/30. Firstly a new blank metal must be created, this can be done by clicking on the Create a new metal button.

Type the name of the metal, and click *Accept*:


Create a new metal - please enter a new metal name

Metal name: Brass 70/30

The new metal is then transferred onto the main page, in amongst all the existing metals and is automatically highlighted. The *Selected item* will have no entries, yet. From this point there are two ways of setting a metal factor, the first and easiest way is to just type the metal factor if it is known into the metal factor box. The second way is to build the metal up from its elements, and then let the program calculate the metal factor. Brass 70/30 is composed of 70% Copper and 30% Zinc, to add elements firstly start with the largest Cu. Click the Add an element button and the *Element selection* page will appear:

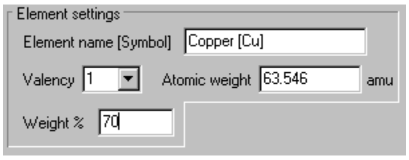

Select and edit the element settings

Click on an element above, then set the 'valency' and 'Weight %' items in the 'Element settings' section

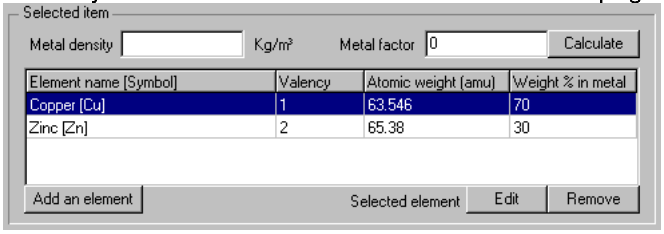
Highlighted element: 29 Cu Copper 63.546 2-8-18-1

Element settings:
Element name [Symbol]: Copper [Cu]
Valency: 1
Atomic weight: 63.546 amu
Weight %: 100

Move the mouse over to element Cu (29). The *Highlighted element* section in the bottom left of the page will change to show the element parameters for Cu. Click the left mouse button, to transfer all the element parameters to the *Element settings* section in the bottom right. There are two items which have to be checked, first is *Weight %*. Initially when an element is selected, the program calculates the remaining % needed to fill the metal. For the first element, the weight % is set to 100, this value should be reduced to 70. The other item which requires attention is the *Valency* box. The most popular valencies for the selected element are shown in the *Valency* box. Copper has two typical valencies of 1 and 2. For the example of Brass a Valency of 1 is normally used. If a Valency is not listed, then just type the number required into the Valency box. Before *Finish* is selected, the settings for copper should resemble:


Element settings
Element name [Symbol]: Copper [Cu]
Valency: 1
Atomic weight: 63.546 amu
Weight %: 70

Next 30% Zinc has to be added, this is done in exactly the same way as for copper, the Valency for Zinc is normally 2. The *Selected item* section on the main page should now resemble:


Selected item

Metal density: Kg/m³
Metal factor: 0

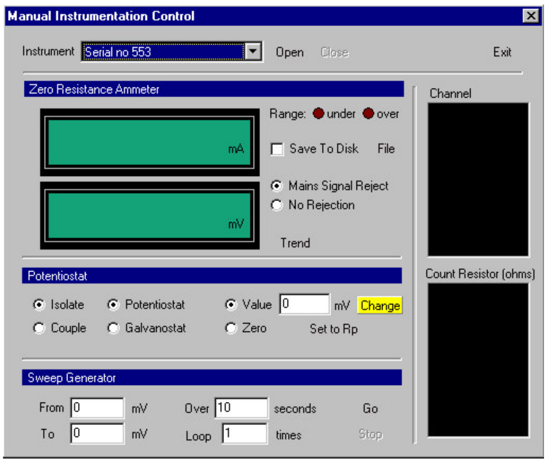
| Element name [Symbol] | Valency | Atomic weight (amu) | Weight % in metal |
|---|---|---|---|
| Copper [Cu] | 1 | 63.546 | 70 |
| Zinc [Zn] | 2 | 65.38 | 30 |

Finally the metal density for Brass is set to 8550Kg/m³ and when in the above example the Calculate button is pressed, a Metal factor of 2083 is calculated. Interestingly for selective corrosion such as dezincification, the Valency of Cu should be set to 0.The program is able to automatically suggest a density if there is only one element which is set to 100% composition.

## Editing an existing metal

Existing metals can be edited by first choosing the metal. The element to be edited is selected, and the *Edit* button clicked. On the *Element* page the settings can then be changed, remember to re-click the *Calculate* button to get the new metal factor.

# Manual Instrumentation Control



Instrument: Serial no 553

☐ Save To Disk

● Mains Signal Reject
○ No Rejection

● Isolate
○ Couple

● Potentiostat
○ Galvanostat

● Value 0 mV
○ Zero

From 0 mV
To 0 mV
Over 10 seconds
Loop 1 times

Having precise manual control over instrumentation, as possible with a Manual instrument such as a ZRA, or Potentiostat can be a useful tool in an educational environment. *Manual Instrumentation Control* enables a computer controlled instrument to be controlled manually.

Starting *Manual Instrumentation Control*: begin by selecting the correct instrument serial number and click the *Open* button, this will open communications with the instrument (NB other programs will not be able to access the instrument at the same time).

### Zero Resistance Ammeter

The *Zero Resistance Ammeter* section begins operating as soon as communication is opened to the instrument, Over / Under range LED's are provided as a guide for selecting the correct count resistor. A *Show Trend* button can be pressed to show a small trend graph of potential and current.

### Potentiostat

Potential offsets can be applied by changing the *Value* text. *Value* can also be changed by moving the mouse up and down, click and hold the left mouse button down on the yellow *Change* square and move the mouse up and down. The mouse gives an analogue movement that is similar in many ways to a turn-pot found on manual Potentiostats. *Set to Rp* offsets *Value* to the rest potential, it will only function correctly when the instrument is *Isolated* from the cell.

### Sweep Generator

The Sweep Generator section allows very simple sweeps to be constructed. Enter your *From* and *To* potentials and the length of time the sweep will be *Over*. A *Loop* can be specified, for example if *Loop* were set at 3 the sweep would go From-To-From-To. To start a sweep press *Go*, the current value of the sweep is shown in *Value* (Potentiostat section).

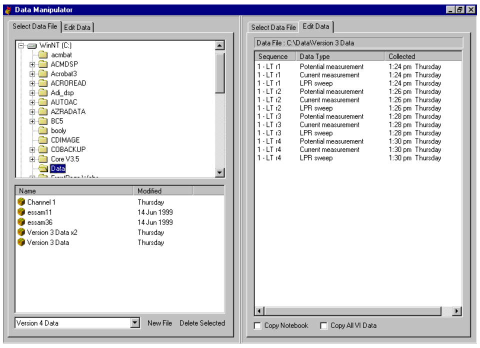
### Advanced Options

Save To Disk – When checked, all collected data will be saved to disk (a filename will be requested) in an Excel compatible text file. Data will be continually saved until the box is unchecked.

Mains Signal Rejection – Ordinarily the instrument will automatically remove any mains signal interference from the collected data, with *No Rejection* the mains rejection can be switched off.

# Data Manipulator

The *Data Manipulator* has many uses:



| Name | Modified |
|---|---|
| Channel 1 | Thursday |
| essam11 | 14 Jun 1999 |
| essam36 | 14 Jun 1999 |
| Version 3 Data x2 | Thursday |
| Version 3 Data | Thursday |

Data File : C:\Data\Version 3 Data

| Sequence | Data Type | Collected |
|---|---|---|
| 1 - LT r1 | Potential measurement | 1:24 pm Thursday |
| 1 - LT r1 | Current measurement | 1:24 pm Thursday |
| 1 - LT r1 | LPR sweep | 1:24 pm Thursday |
| 1 - LT r2 | Potential measurement | 1:26 pm Thursday |
| 1 - LT r2 | Current measurement | 1:26 pm Thursday |
| 1 - LT r2 | LPR sweep | 1:26 pm Thursday |
| 1 - LT r3 | Potential measurement | 1:28 pm Thursday |
| 1 - LT r3 | Current measurement | 1:28 pm Thursday |
| 1 - LT r3 | LPR sweep | 1:28 pm Thursday |
| 1 - LT r4 | Potential measurement | 1:30 pm Thursday |
| 1 - LT r4 | Current measurement | 1:30 pm Thursday |
| 1 - LT r4 | LPR sweep | 1:30 pm Thursday |

- Look inside data files and show test information,
- Copy individual sequence data from one data file to another,
- Convert *Version 3.xx data to V4* data format,
- Copy a complete data set,
- The main screen of *Data Manipulator* is split into two halves, each half is identical to the other half with one exception, only the left side can *convert Version 3.x data to Version 4*.

## Look inside data files and show test information

Information such as Test parameters and Test Environment (i.e. Rest Potential) can be viewed with Data Manipulator. To view sequence information, use the explorer display to select a folder where your data resides. Data files contained within the selected the folder view are listed at the bottom, right click on a data file and select **Edit Data**. The page will change to show individual tests within the data file, right clicking on any one of these tests and select **Show Sequence** Information reveals detailed information about the test. To return to select another data file click the **Select Data** File tab at the top.

## Copy individual sequence data from one data file to another

It will become apparent why the data viewer is split into to halves in this short tutorial, how one individual test is copied from one file into another:

> Using the left half of *Data Manipulator,* right click on a data file, and select **Edit Data**. Now move your attention over to the right hand pane, select a folder and click the **New File** button at the bottom, give this empty data file a name. Next, right click on the new data file and select **Edit Data** also. A piece of data can be copied to this new data file by dragging it from the left pane and dropping it in the right pane.

## Convert *Version 3.xx data to V4* data format

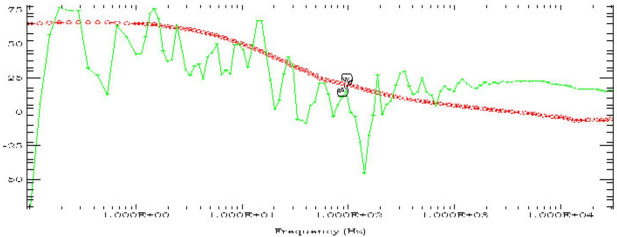
Data file conversion can only be done using the left pane. Select a folder in which V3 data exists, the select **Version 3.x** Data in the combo box at the bottom of the screen (next to **New File** button), all version 3 data will be shown below the folder view. Right click on the file to convert and select **Convert Selected to Version 4**. The existing V3 data is left untouched, Version 4 data files are placed along side in the same folder. Multiple files can be converted by holding CTRL down whilst selecting.

## Copying a complete data set

This is achieved by selecting a folder containing data and right clicking on a data file, select. Next select a different folder where the data is to be copied to, and right click in the pane where the files are normally displayed and select **Paste**.

# Reduction of noise during AC Impedance

Modifications have been made to the GillAC to dramatically reduce noise in AC impedance curves. Systems



using remote reference electrodes, salt bridges and lugin probes are prone to very high levels of mains pick up, causing large random scatter of data points. ACM sell a unique noise reducing electrode for this job, contact our sales department for details. This does not change the rest potential as the NR electrode is purely AC coupled with no DC component.
For example the bode plot showing phase v frequency above shows the result using a SCE and lugin only in green, and in red the SCE, lugin and a Pt electrode connected to NR. All run parameters were the same.
In many cases the Noise Reducing electrode will not be needed, but if the data looks like the green curve above it is strongly recommended.

# Field Machine Transportation

The following information relates to the internal battery inside a field machine:

1. **Air Transport**
   Under IATA Dangerous Goods Regulations 38th Edition effective 1st January 1997, the above battery is classified as – “Class 8, Group III UN No. 2800 Batteries, wet, non-spillable, electric storage, special provision A67”
   This battery is classified as non-spillable because it has been shown to meet the requirements of the above regulations as stated in Packing Instruction 806 on page 406.
   According to the “Technical Instructions for the Safe Transport of Dangerous Goods by Air” 1995-1996 Edition of the International Civil Aviation Organisation (ICAO), special provision states “Non-spillable batteries are not subject to these Instructions (Packing Instruction 806) if, at temperature of 55 deg C, the electrolyte will not flow from a ruptured or cracked case and there is no free liquid to flow, and if, when packaged for transport the terminals are protected from short circuit.”
   This battery is classified as meeting the A67 requirement after testing by PIRA in 1985, who stated that the batteries may be regarded as non-dangerous. Several other independent internationally recognised laboratories are also in agreement that these batteries are non-dangerous.
   **Transport by Road and Sea Freight**
   The IMDG code states that to be classified as non-spillable, “The battery must be able to satisfactorily withstand without leakage the vibration and attitude tests described…” Ref IMDG Code page 8121. These battery types have satisfactorily completed these tests as certified be Giltspur Packaging Ltd. Certificated of Test Serial No RTD3330/45/2 Job No 4000/0775 issued under Ministry of Technology Authority Ref No 30260. Packaging and labelling are as in 1.
   **TREM cards are not required.**

**Contact Information**

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Fax: 015395 58562
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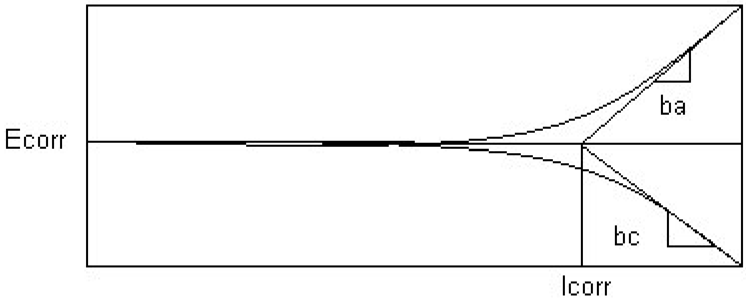
www: www.acminstruments.com

# Applications

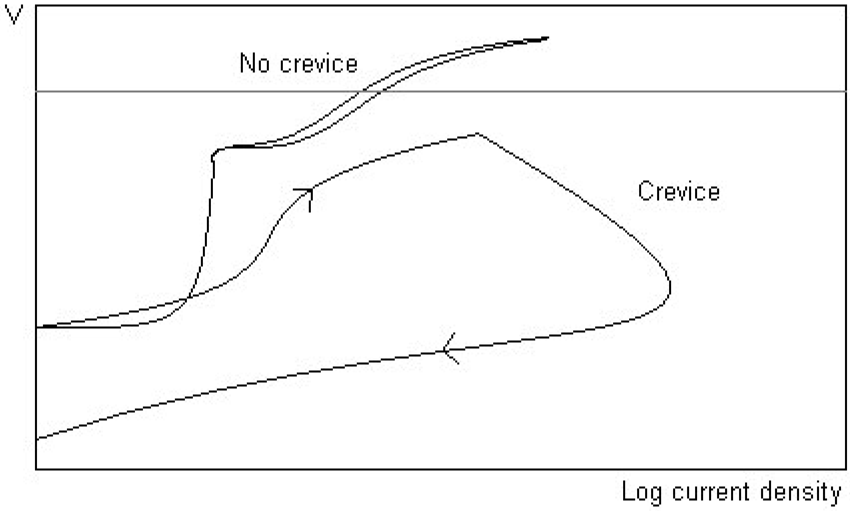
## Test Types

### Cyclic Voltammetry

Cyclic sweeps are used to measure corrosion that proceeds at about the same rate all over the metals surface (uniform) and corrosion at discrete sites on the surface, e.g. pitting, crevice and stress corrosion cracking (localised). A typical experiment comprises an electrochemical cell containing the electrolyte, a reference electrode, a platinum auxiliary and a working electrode of the metal under test. The instrument is connected RE to reference electrode, AE to the platinum and WE1 to the working electrode. This applies to the Gill AC, the Gill 8 and 12, the Field Machine and the manual potentiostat. A sweep is programmed into the sequencer, say –1000 mV to +1000 mV @ 20 mV/min and the test started. The results can be interpreted in the Analysis package. To measure uniform corrosion the method of Tafel extrapolation is used, the Tafel slope been the slope of the straight line portion of the semi-logarithmic polarisation curve. To determine the degree of localised corrosion the amount of hysteresis between the positive going sweep and the negative going sweep is calculated.



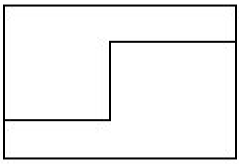
The diagram above shows a plot of voltage versus log(current). At large perturbations away from Ecorr the reaction measured becomes almost totally oxidation (when going positive) or almost totally reduction (when going negative). The equations that describe the reactions at large overpotentials can be simplified to a linear relationship, i.e. Anodic overpotential = ba.log(iapp/icorr), Cathodic overpotential = (-bc.log(iapp/icorr)). This allows an extrapolation of iapp from either the anodic or cathodic Tafel region to the open circuit potential, and hence to obtain the corrosion current.



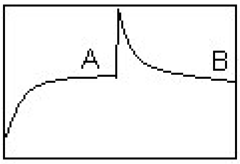
This is an example of a cyclic polarisation on high chrome steel in 1M NaCl. Two samples are scanned and the results show that one has no crevice while the other has a crevice. The large hysteresis is typical of localised corrosion.

## Linear Polarisation Resistance

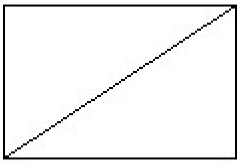
The LPR technique is most frequently used, been quick and easy to use. A small sweep from typically –10 mV to +10 mV at 10 mV/min around the rest potential is performed. The resulting current/voltage plot usually exhibits a straight line, the inverse slope of which is proportional to the corrosion rate. The Gill AC, Gill 8 and 12, the Field Machine, the Pocket Machine, the LPR meter and the bubble test software all use this method.



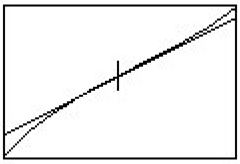
Voltage step



Current response



Voltage sweep

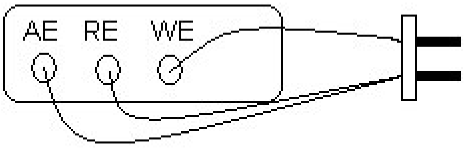


Current response

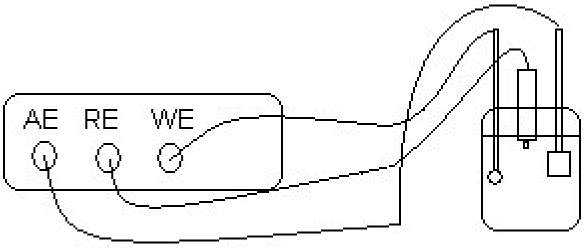
The step method is used in hand held instruments, for example the pocket machine. The current is measured at points A and B once the initial current surge has steadied. The voltage sweep results in a response shown above, a best fit straight line gives the charge transfer resistance. A variant of the LPR test is the pitting index. This is a measure of the asymmetry between anodic and cathodic current response, this feature is built into to the portable LPR meter where it is available as a switched option between corrosion rate and pitting index. The LPR method is ideal for plant monitoring offering an almost instantaneous indication of corrosion rate, allowing for quick evaluation of remedial action and minimising unscheduled downtime. As an example and guide to allow the new operator to obtain a feeling for the numbers involved the table below gives a qualitative classification of corrosion rates of carbon steel in a cooling water system.

| Corrosion rate mm/year | mils/year | Classification |
|---|---|---|
| <0.03 | 1.2 | Excellent |
| 0.03 – 0.08 | 1.2 – 3.2 | Very good |
| 0.08 – 0.13 | 3.2 – 5.2 | Good |
| 0.13 – 0.2 | 5.2 – 8 | Moderate |
| 0.2 – 0.25 | 8 – 10 | Poor |
| >0.25 | >10 | Very poor |

To convert a corrosion current in mA/cm$^2$ to a corrosion rate in mm/year multiply the current by 12. The sources of error in LPR tests include uncertainty in the parameter B used in the Stern and Geary equation where icorr = B/Rp, B = (ba.bc)/(2.3(ba+bc)), choice of a scan rate that is too fast, neglect of the solution resistance and non linearity. In practice a value of 20 mV usually works well for B, a scan rate of 0.2 mV/sec is often adequate, the solution resistance can be compensated by positive feedback and the non-linearity error is only a small percentage of the result. Linear polarisation resistance can be done either three or two electrode. The two electrode method relies on both electrodes been similar so that when they are coupled and offset the test is still in the linear region. This matching of electrodes is not needed when using the three electrode method as the potentiostat measures the rest potential and offsets the test around that.



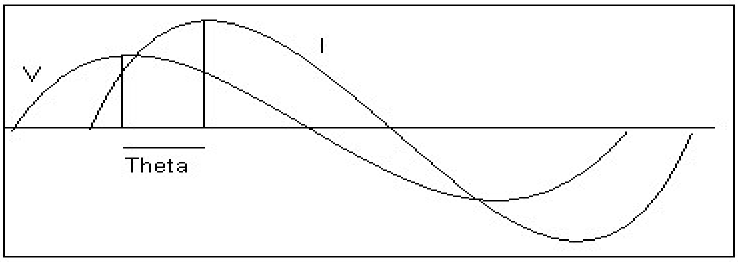
Two electrode wiring of Gill AC



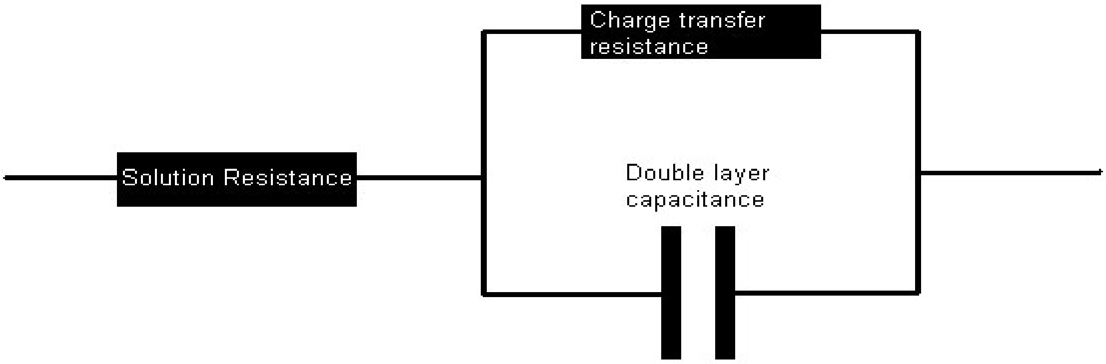
Three electrode wiring of Gill AC

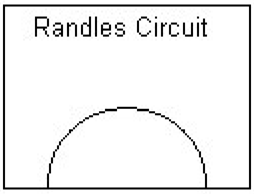
## AC Impedance

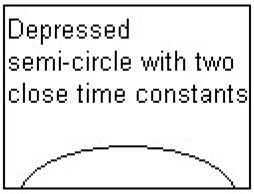
The Gill AC, Gill 8 and 12 and the Field Machine are used for this test. A typical experiment sweeps from 10 kHz to 0.01 Hz with a 10 mV perturbation around the rest potential. The usual result is an nyquist impedance plot of half a semi-circle, the high frequency part giving the solution resistance and the width of the semi-circle giving the corrosion rate in the same manner as LPR. The analysis of this data is performed by circle fitting in the analysis software. One useful benefit of AC is the ability to measure the solution resistance at high frequency. This allows any instrument that incorporates AC to perform automatic IR compensation during DC tests.

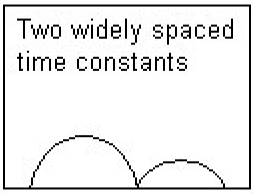


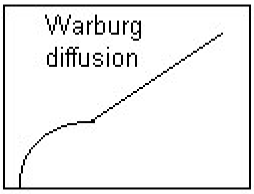
At each frequency a sine wave is generated and fed into the potentiostat. This wave is then imposed on the cell and the cell potential and current flow measured. The measured values of current and voltage are compared for amplitude and phase and an impedance calculated. This is repeated for the rest of the frequencies and a plot generated. The standard starting point with AC impedance is the basic Randles circuit below.

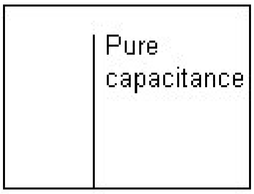


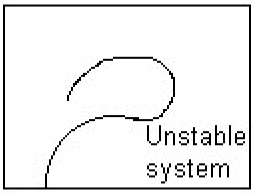








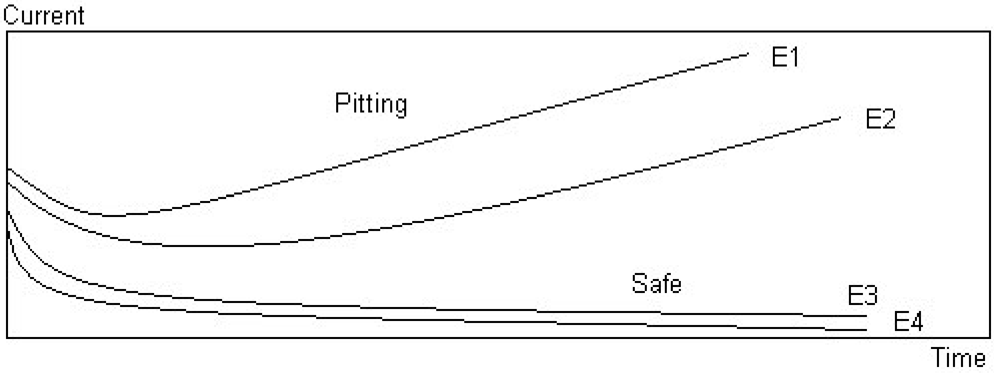




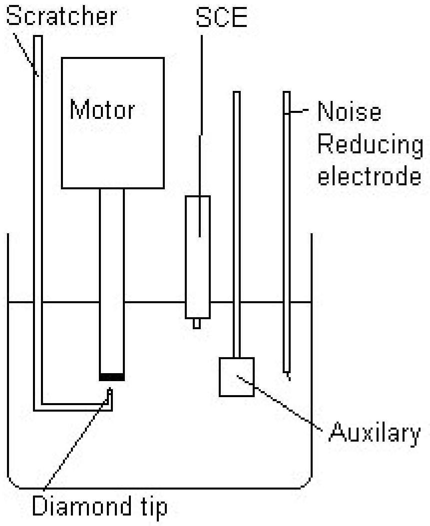
An alternate name for AC impedance is Electrochemical Impedance Spectroscopy (EIS).

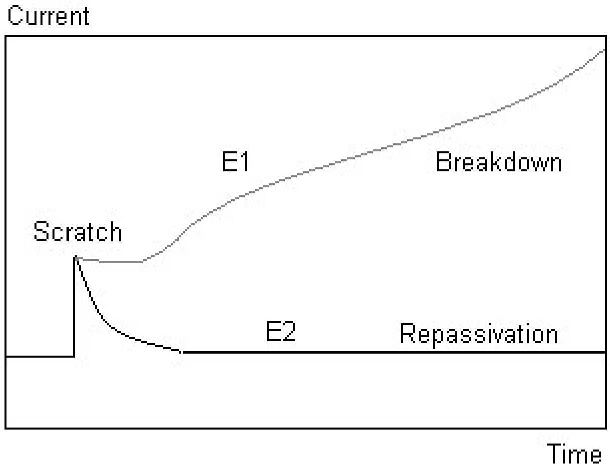
## Potentiostatic

The potentiostatic test is usually performed to determine resistance to pit initiation at a given potential and to simulate galvanic situations using any of Gill AC, 8, 12, Field Machine and manual potentiostat set to a stable potential whilst recording the current. In the study of pitting the cyclic sweep method can have problems related to sweep rate and too much pitting propagation before sweep reversal. The use of individual samples held at potentials around the suspected pitting potential will allow the correct determination of the pitting potential. For example the graph below shows current versus time for samples polarised at different potentials.



In this case potentials E3 and E4 were below the protection potential and E1 and E2 were above the protection potential, allowing pits to form. A modification of the potentiostatic method has been built for a customer by ACM. A rig to scratch a rotating electrode with a diamond knife was made and the resulting currents recorded at different potentials.





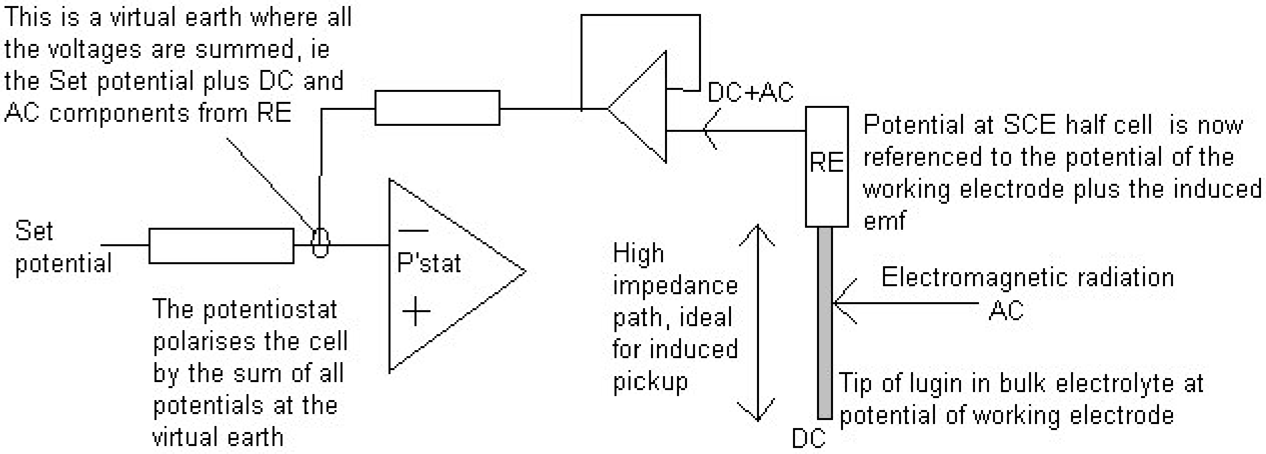
At potential E2 the mechanically induced scratch repassivated. At potential E1 the induced scratch did not repassivate indicating the potential is above the critical potential. Rapid logging of the current in the first milliseconds after scratching gives kinetic information about the mechanism of repassivation.

## Noise Induction

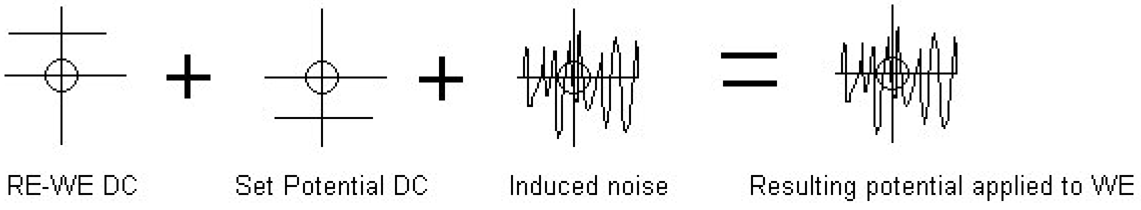
All good electrochemical test equipment is designed to reject mains noise, giving very tidy data as a result. Unfortunately this massaging of the raw data can mask a disturbing phenomena which is usually overlooked. Testing here at the ACM laboratories has found that induced power line pickup within salt bridges, lugin probes and reference electrode bodies causes very high levels of voltage at 50 Hz (in England) to be present at the reference electrode lead, summed with the reference electrode DC voltage. Most experimenters are completely unaware of this as the measuring instrument removes this noise and present the underlying data. Problems occur in two cases.

First, during current and voltage noise monitoring using a ZRA the voltage measured is a composite of the true DC voltage and any remaining 50 Hz, provided the DC analysers are not swamped by the AC component.

Secondly, when the cell is connected to a potentiostat the picked-up mains frequency acts as an additional polarising signal for the potentiostat. The potentiostat has no way of detecting that the spurious mains noise is not a desired polarising voltage and consequently polarises the cell by exactly the induced voltage. The working electrodes response is indicated by the measured current. Equipment makers remove this 50 Hz component from the result but the true unfiltered value of current shows that the working electrode has been polarised by often 100's of milliVolts at 50 Hz. This additional polarisation, often on top of a small +/- 10 mV LPR sweep, can completely upset the electrodics of the system and make a nonsense of the results. This phenomena of Self Polarisation Activated by Mains is a very serious problem and because the induced voltage and the corresponding current response is hidden from the experimenter by the instrument makers (ACM included) the scientist does not appreciate the working electrode is swung wildly around the desired potential at 50 Hz.



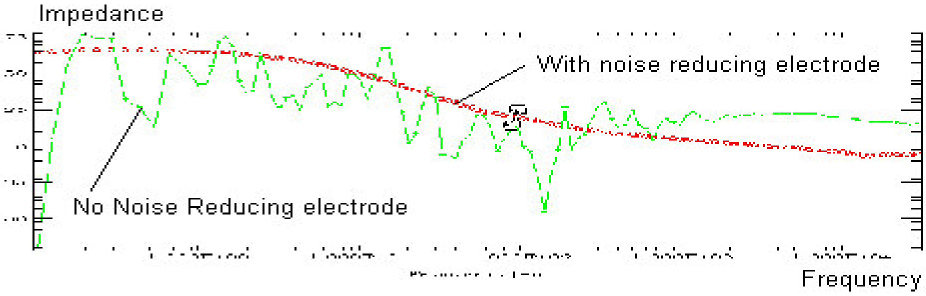
The electromagnetic radiation has two components, the easily screened out electrical component and the much more difficult magnetic component. It is the magnetic component that penetrates Faraday cages and causes problems even in well screened systems.



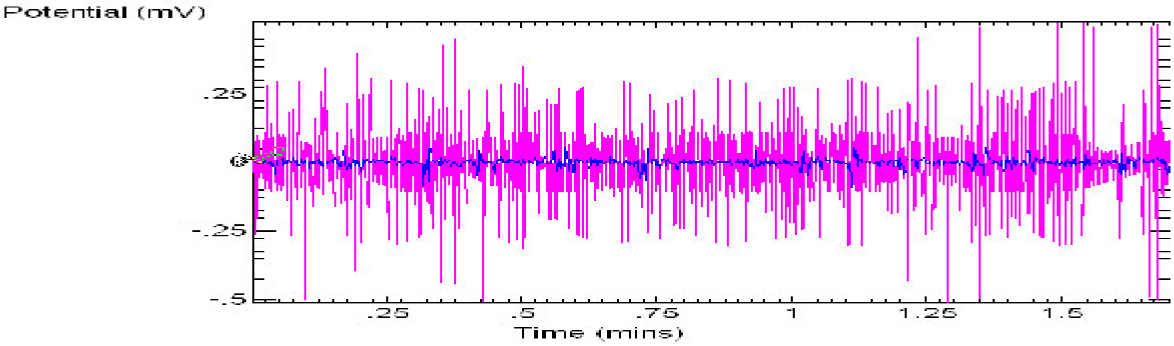
At ACM we have discovered that the way to solve the problem of induced pickup is to provide an AC coupled low impedance path between the input of the reference electrode buffer and the bulk electrolyte. This path is easily achieved using an ACM Noise Reducing Electrode as a fourth electrode in the cell. Because this electrode is AC coupled the DC component of the reference is not effected.

# Noise Reducing Electrode

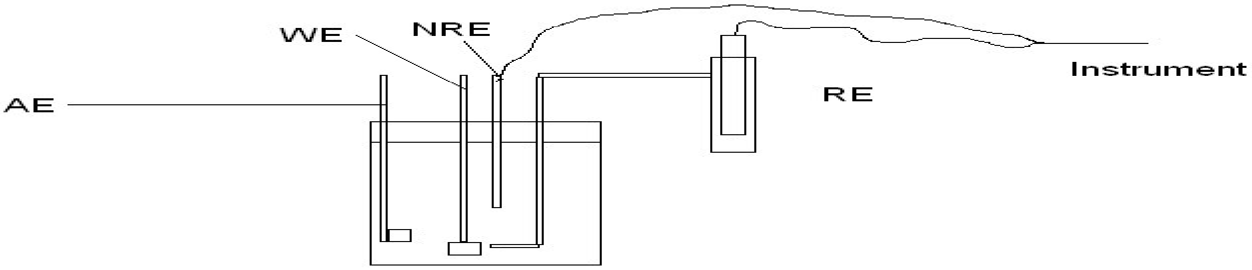
The ACM Noise Reducing Electrode (NR) is designed to overcome the serious problem of induced mains pickup causing unwanted polarisation of the test cell (see the section on induced noise). It also benefits ZRA experiments allowing more gain to be applied to the measurement of potential. Two examples are shown below.



In the example above a working electrode of titanium is immersed in tap water with a lugin probe and SCE. Three electrode AC impedance was performed and the resulting Bode plot shown in green. For the second run using identical parameters a Noise Reducing electrode was added. The Bode plot is shown in red. It is apparent that the red curve is much smoother, and, because less averaging was needed by the instrument, it was faster to obtain.



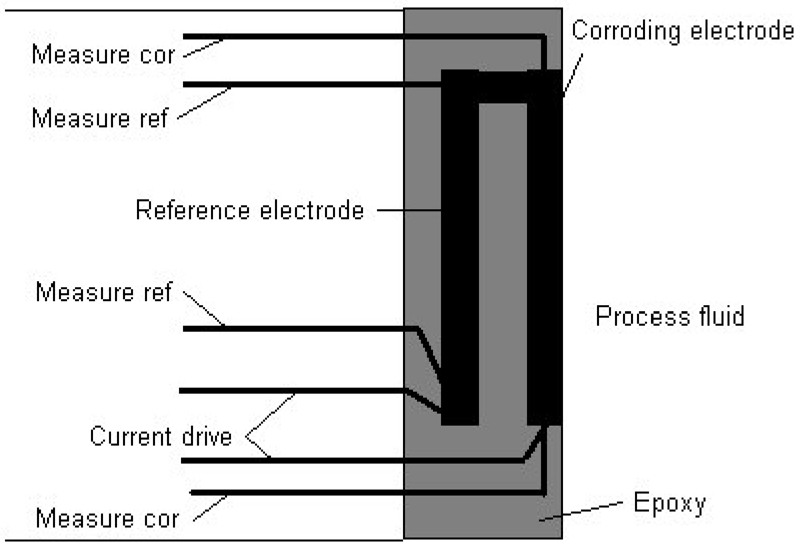
This plot is taken from a current and voltage noise test on tin in 1M NaCl. Shown in purple is the point to point voltage trace for the system not using a Noise Reducing Electrode and shown in blue is the same experiment using a Noise Reducing electrode. The use of a NR electrode allowed much finer features of the noise trace to be seen.



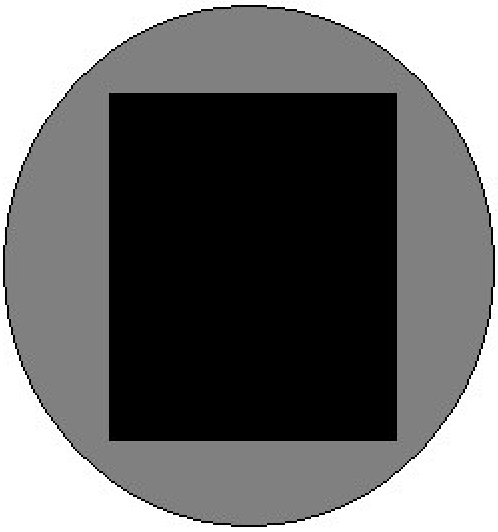
All ACM Noise Reducing electrodes are made of pure platinum set in a glass tube with the active component sealed in the glass. They are used to capacitively couple the input of the reference electrode buffer to the bulk electrolyte, thus providing a low impedance path to ground for voltages induced in the lugin probe/salt bridge. This then ensures that the reference electrode is measured with respect to ground (the potential of WE) and not with respect to ground plus the induced voltage. Some systems in the past such as those involving mains heaters have had so much mains induced polarisation that the results were not correct. The use of a NR electrode stops this unwanted polarisation.

## Electrical Resistance

This technique is not electrochemical, but relies on the increase in electrical resistance of a metal sample when the metal sample is thinned as a result of corrosion compared to a reference element of the same material. Commercial probes are usually used but laboratory constructed ones can work well. The slow metal loss is recorded against time. To measure Electrical Resistance using a Gill AC the optional ER box is plugged into a small connector on the back of the instrument. This conditions the signal across the sample and non-corroding reference applying enough gain to send a very strong signal to the twin 21 bit analysers within the Gill AC. ER may be used for virtually all environments, gases liquids or solids, whether conductive or non-conductive. The effect of pitting on probes completely destroys the uniform corrosion result and can be a problem. The art of good probe design is to keep the corroding and reference elements at the same temperature.

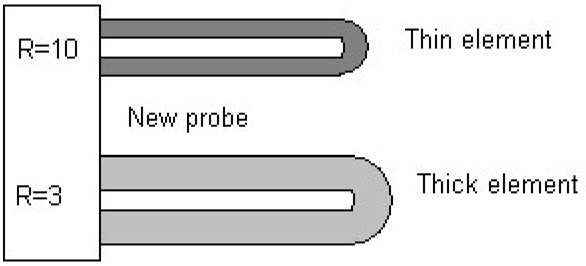


Typical flush probe cross-section

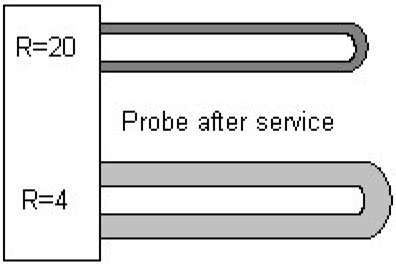


Front view of flush probe

In this case the reference and corroding elements are sandwiched together and rapidly reach the same temperature. A novel electrode design uses both elements in the process stream, and relies on the ratio of surface area to volume been larger on one element than the other. As both experience the same corrosion rate at the surface the thinner element will show a different rate of change of resistance. The metal loss for the thinner element can then be calculated. This allows for almost perfect thermal accuracy. In the example below the ratio changes from 3.3 to 5 after exposure, allowing metal loss to be calculated using software supplied free by ACM.



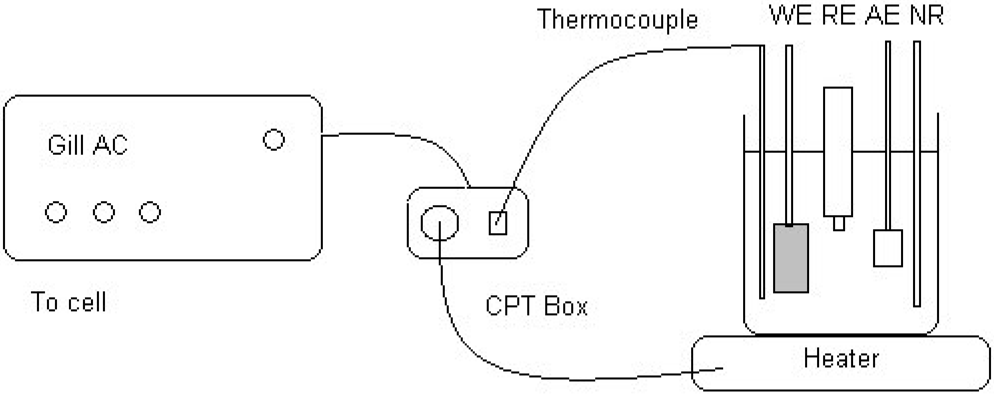
Thin/Thick ratio = 3.3



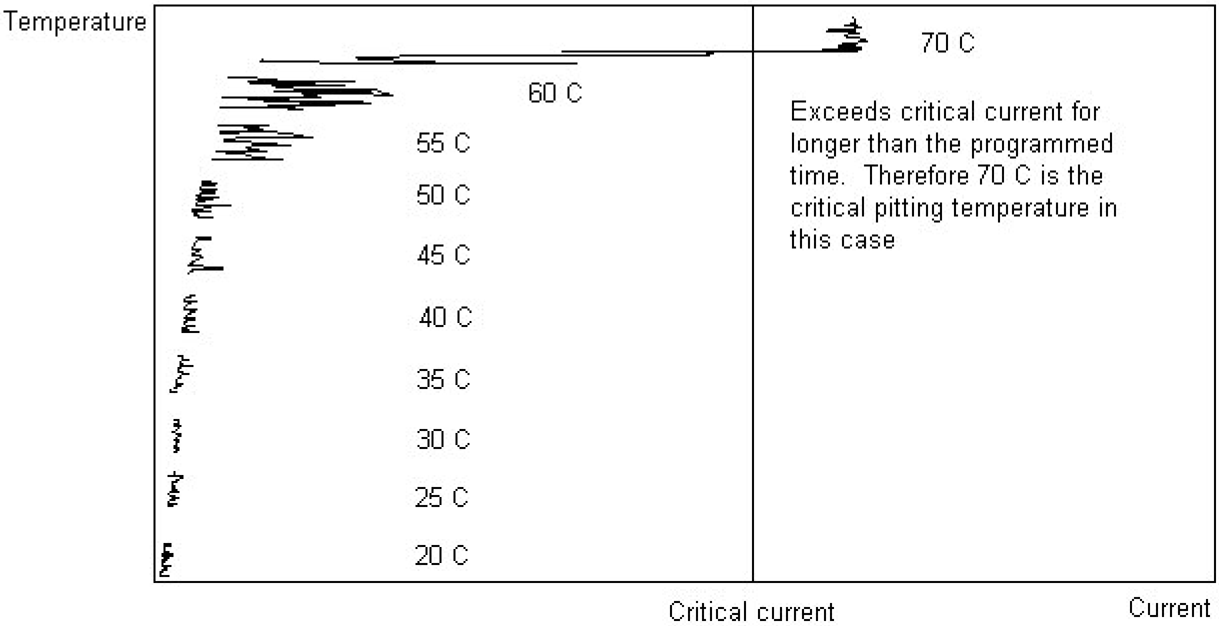
Thin/Thick ratio = 5

## Critical Pitting Temperature

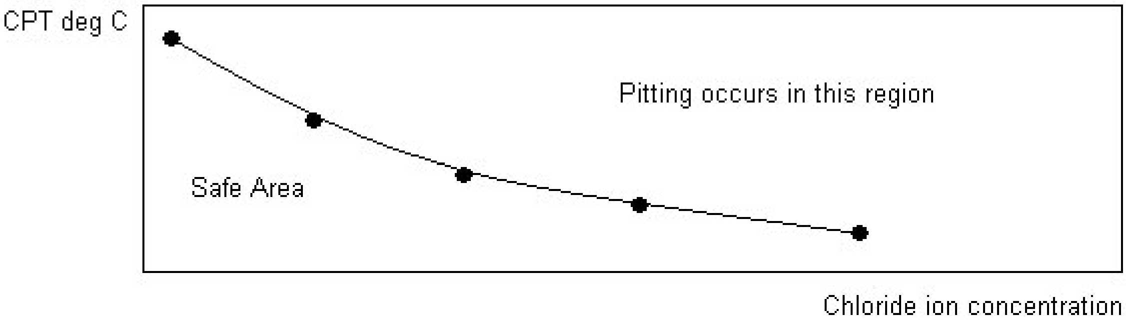
This test involves determining the temperature at which initiation of localised corrosion occurs. It is performed using a Gill AC with the optional CPT box plugged into a small connector on the back. The CPT box accepts a thermocouple input and controls a mains powered heater. The basis of this simple test is to hold the test sample at a required anodic potential using the Gill AC. The temperature is then increased in steps by the Gill AC until the current recorded by the Gill AC reaches a set current, this temperature is the critical pitting temperature.



A typical temperature current graph from the equipment above is shown in the diagram below.



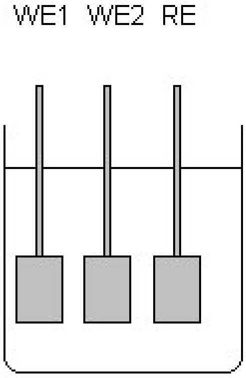
This can then be repeated at different chloride levels, pH's etc to obtain a graph of critical pitting temperature versus the altered parameter, e.g.



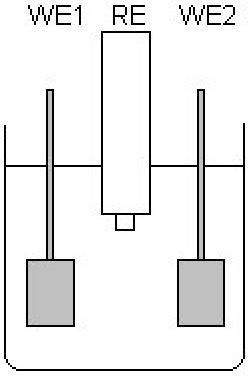
## Current and Voltage Noise

This is one of the simplest tests, usually three identical electrodes are immersed in an electrolyte and two of them are coupled by a zero resistance ammeter whilst the third is connected to a voltmeter to monitor the potential between the couple and this third electrode. The ZRA ensures both electrodes in the couple are at exactly the same potential. The Gill AC, 8, and 12 and the Field Machine along with the dedicated Galvo Gill 12 all incorporate ZRA's. In practice a set length test is set up, say 1000 points recorded every 1 second and the resulting data analysed.

3 identical electrodes for immersion in process stream

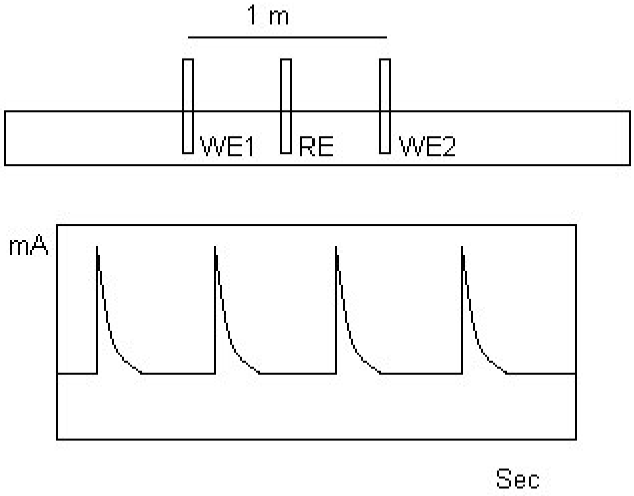


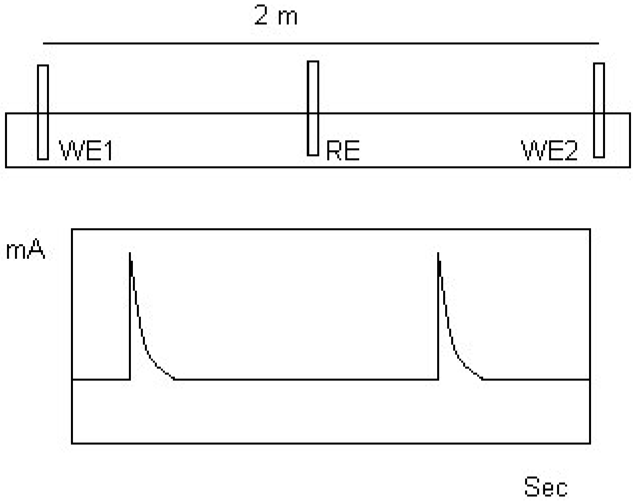
3 identical electrodes



2 identical electrodes and SCE

Electrochemical noise is appealing because it can be conducted at open circuit without perturbing the corroding system. Currently no real consensus exists as to the test details, each experimenter usually develops their own procedure for testing known products and builds an experience base for comparative purposes. Attempts have been made to find a relationship between uniform corrosion rate and measured electrochemical noise but this universal equation has not yet been found and consequently no ASTM standard exists for noise testing. When used for long term field monitoring in aqueous systems an analysis of fluctuations in the current record has proved useful. One system was installed at 13 points in a plant and known conditions reproduced. Calibrating the current pulses against known corrosion rates allowed the system to monitor the plant. One of the original uses for electrochemical noise in the late sixties was for monitoring localised corrosion. The transient development of bare metal at newly formed pit or cracking sites as a result of temporary propagation and repassivation can result in potential noise, current noise or both. Cell geometry is often overlooked during noise tests but is just as important as for standard polarisation tests. Consider the experiment below, performed using a Gill AC.



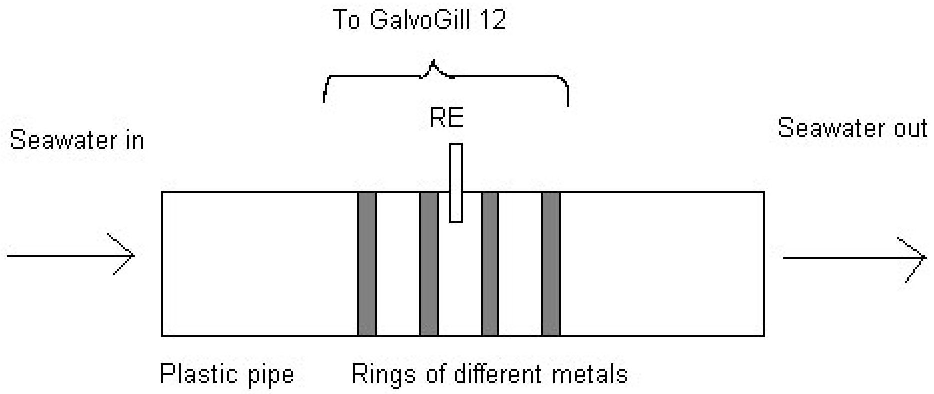


Three identical tool steel electrodes were immersed in tap water. Test 1 had WE spacing of 1m and test 2 had WE spacing of 2m. A regular large pulse was observed in the current trace with a frequency inversely proportional to the electrode spacing.

## Environment

Sea Water

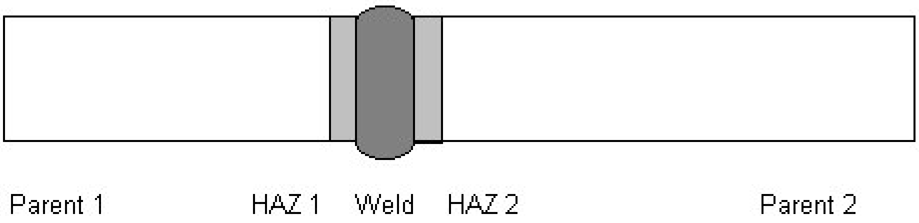
This electrolyte has the virtue of high conductivity, making electrochemical tests a natural choice. In-situ testing has the advantage of allowing the natural bio-film to form. The Field Machine is used in this case to monitor galvanic currents, Tafel slopes, LPR and AC Impedance. In the laboratory the same tests are undertaken with the Gill range, possibly using man-made sea water. Laboratory testing is usually short term with the advantage that specific environmental conditions can be controlled. The disadvantage of laboratory testing is that the actual service environment and conditions are not been evaluated. The special case of coated samples in sea water relies on testing with AC impedance to overcome the paint resistance and 'look through' to the metal surface. Galvanic coupling in seawater systems can be a problem, the use of multiple electrode tests can help diagnose this. As an example consider the test rig below connected to a GalvoGill 12 for testing torpedo parts in seawater.



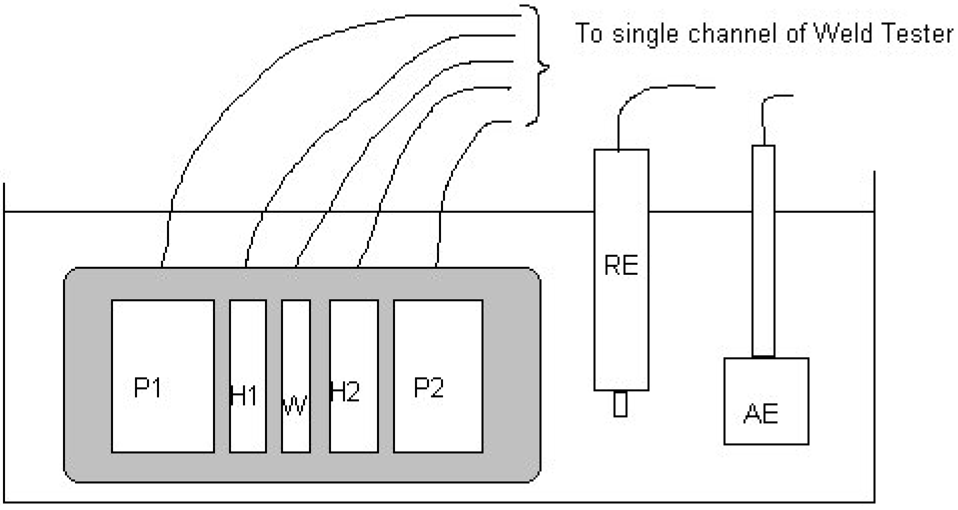
The effect of temperature in real seawater is not as straightforward as with industrial electrolytes. As a general rule the corrosion rate in seawater increases as the temperature is increased. This rule holds true when only the effect of temperature on the corrosion rate is considered. However, other variables such as oxygen concentration and biological activity must be accounted for. The solubility of oxygen decreases as temperature is increased. Biological activity generally increases with increasing temperature, and calcareous deposits and other protective scales are also more likely to form/deposit on metal surfaces at higher temperatures. The preferred environment for laboratory testing is natural seawater, but synthetic seawater is often used. Natural seawater is fresh water piped straight to the laboratory, once it is removed from it's natural environment and stored in bowsers the composition changes and this can have a large impact on the resultant metal corrosion rate. Synthetic seawater solutions that are typically used include 3.5 wt.% sodium chloride and ASTM D 1141 substitute ocean water mix. The results from this synthetic seawater may not be the same as for real fresh seawater. One advantage of using the easily made up 3.5% NaCl is that calcareous deposits will not form under cathodic polarisation conditions in the solution. Although seawater has high electrical conductivity there are cases when the IR drop should be considered. In system with large current flow, such as testing sacrificial anode material, the I component could be of the order of Amps. Even with an R component of say, 100 mOhm, this would lead to a drop of 100's of mV between RE and WE. This may be minimised by good cell geometry and compensated for by using IR compensation after measuring the drop with AC Impedance.

## Welds

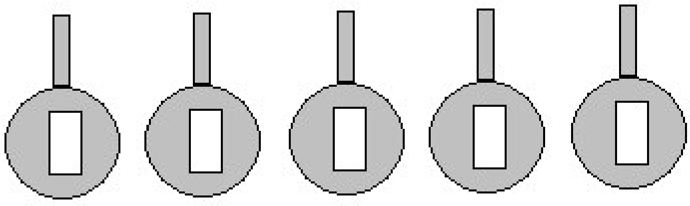
Welds pose a particular problem, each weld is a complex galvanic couple made up of parent metal (e.g. a pipeline), a weld metal and an area of heat affected metal. Under many circumstances the bulk of the anodic dissolution is within the heat affected zone (haz). This very thin anode and large cathode of parent metal is the worst possible case, rapidly leading to failure. Conventional electrochemical testing in which a sample of the weld is immersed and used as the working electrode will only reveal a low overall net rate not the high rate in the heat affected zone. The solution is to segment the sample weld, cutting out test electrodes of parent1, haz1, weld, haz2 and parent2. These can then be coupled together and the individual response from each section measured. The instrument to do this is called a Weld Tester. This instrument is a Gill 8 or 12 built to accommodate the extra four working electrodes per cell. Once the cell is connected the full range of DC and AC tests are used to determine the susceptibility of each component to corrosion.



This is then segmented, epoxy masked, and ground flat. All 5 electrodes are added to the test electrolyte and connected to the Weld Tester



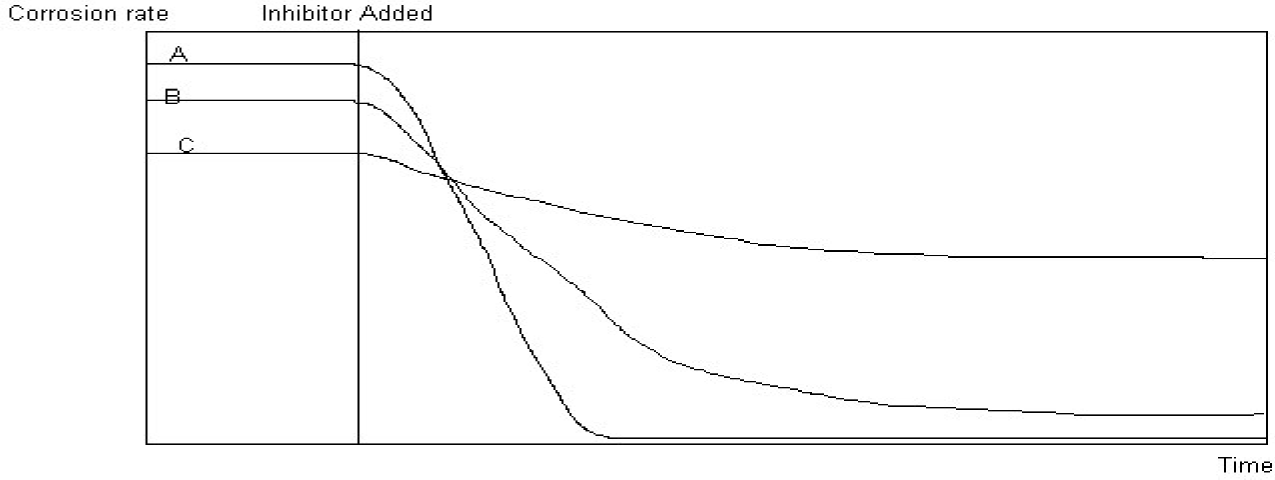
Segmented weld held in one block of epoxy



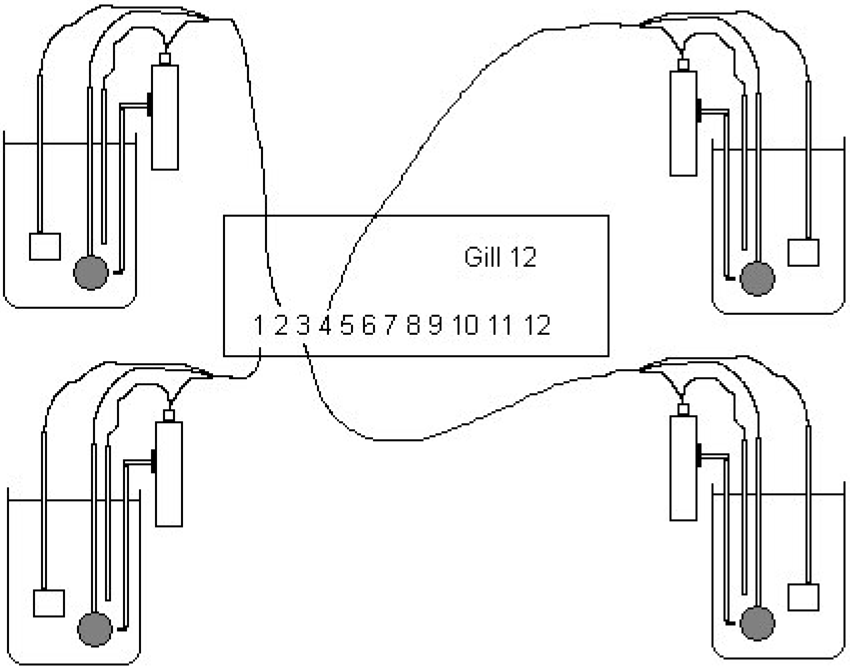
Alternative mounting method using standard samples

## Inhibitors

Testing of these chemicals is performed by makers and users. Makers set up initial screening to not reject any potential good ones. Users set up initial screening to reject any potential bad ones. Test metal should be the same metal as that to be protected, as should be the test fluid. The two most widely used tests are LPR and Tafel plots. Both are ideally performed using a Gill 8 or Gill 12. The use of a multiple channel instrument allows for replication to gain confidence in the results. The Bubble Test software automatically groups the tests and prints inhibition reports using the LPR method. After all the small LPR perturbations have been made an optional Tafel sweep is performed to obtain the Tafel slopes. AC impedance is less commonly used except with high resistance electrolytes where the IR drop in the solution needs to be measured and overcome.

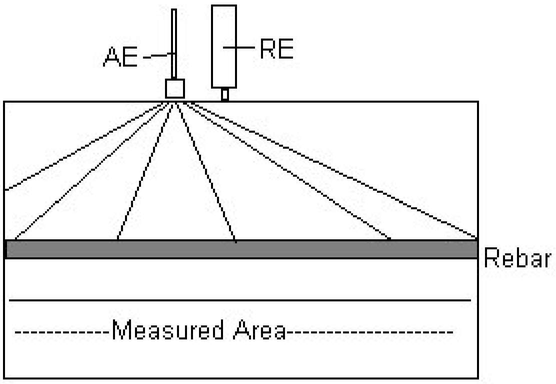


Typical results are shown above, metal A has the highest corrosion rate but on the addition of the inhibitor the rate drops to the lowest. The test should be repeated at least three times and the results compared for scatter. The percentage protection of an inhibitor is usually quoted, this is defined as
100*(uninhibited rate-inhibited rate)/uninhibited rate. The bubble test software will display this for all channels under test. The temperature of the test is important, a whole set of percentage protection values is displayed with respect to temperature.

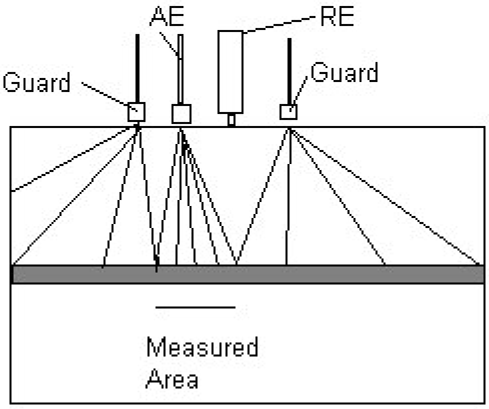


## Concrete

Steel encased in intact concrete with no chloride, carbonation, stray currents or cracks should experience a pH of 12.5 and passivate, remaining good for centuries. In practice problems do occur and finding the problem deep under the surface is no easy task. Some of the problems faced include; very high resistivity, difficult connection to the reinforcing bar, and the geometry of the test. Often the auxiliary and reference sit on the surface of the concrete with multiple strands of rebar some distance beneath. Knowing the extent of polarisation is often a matter of experience. The simplest electrochemical test is potential mapping, this gives an idea of corrosion activity but does not give corrosion rate data. More sophisticated tests use the Field Machine to determine corrosion rate from LPR, IR compensated LPR and AC Impedance. Variants of the Field Machine have been supplied incorporating a guard ring to focus the measurement on a better defined area of rebar.

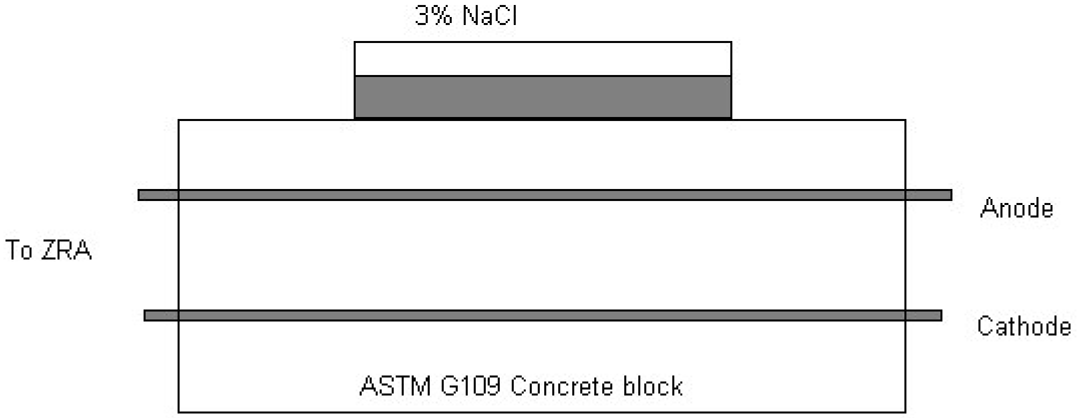


Unguarded Measurement



Guarded Measurement

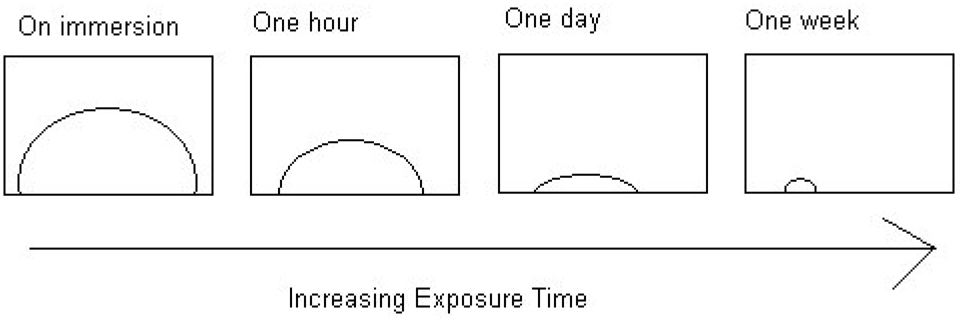
A test has been devised to measure chloride uptake in the laboratory. It consists of three bars mounted one above the other two in concrete and connected by a ZRA (as in Gill AC's etc). A bath of 3% NaCl is mounted on the top and when the chloride permeates through the top bar becomes and anode with respect to the bottom two, as indicated by the current shown from the Gill AC.



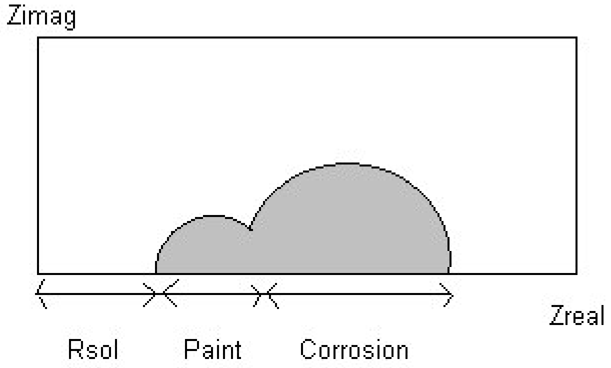
This test block can also be used to measure IR compensated LPR and AC Impedance, by applying a test frequency from 10000 Hz to 1 mHz, mechanistic information of the corrosion processes can be obtained by studying the time constants revealed. Some workers have used a galvanostat (e.g. Gill AC in galvanostat mode) and pulsed the concrete with known amounts of charge, the potential change is then recorded and the time constants of the cell calculated.

## Non-metallic Coatings

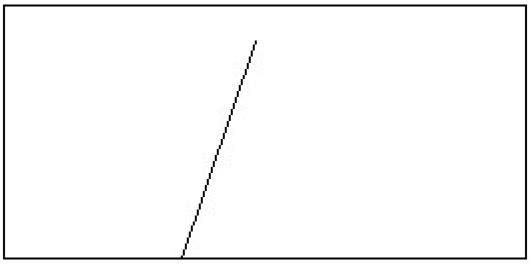
Electrochemical testing of coated samples is achieved using the Paint Buffer accessory for the Gill AC, 8, 12 and Field Machine. This performs four important functions when dealing with very high impedance systems: Increases input resistance of the RE buffer, reduces input bias current flowing from the sample and RE, shifts the current measuring ranges down by a factor of 100 and reduces noise. When studying very thin films or films with holes LPR is sometimes used but in most other cases AC impedance is often the technique of choice. Cells can be of the limpet type where a cell with a waterproof seal is clamped onto the painted surface or dipped immersion of painted samples. Performing regular AC impedance tests during the exposure time will also show aspects of coated samples other than corrosion, such as water uptake with respect to time. A forty channel instrument was built by us for a customer to study water uptake in coated pipelines and proved a valuable tool.



This represents a nyquist plot taken at the intervals shown for a painted sample that is suffering from water uptake. Note the diameter of the semi-circle decreases as the water is taken up.



This is the classical nyquist plot for a painted sample, the first high frequency points show the solution resistance, the diameter of the semi-circle is the paint and the large semi-circle the corrosion. In practice the nyquist curve can look like the plot below, indicating a good capacitive paint layer.



## Cell Design

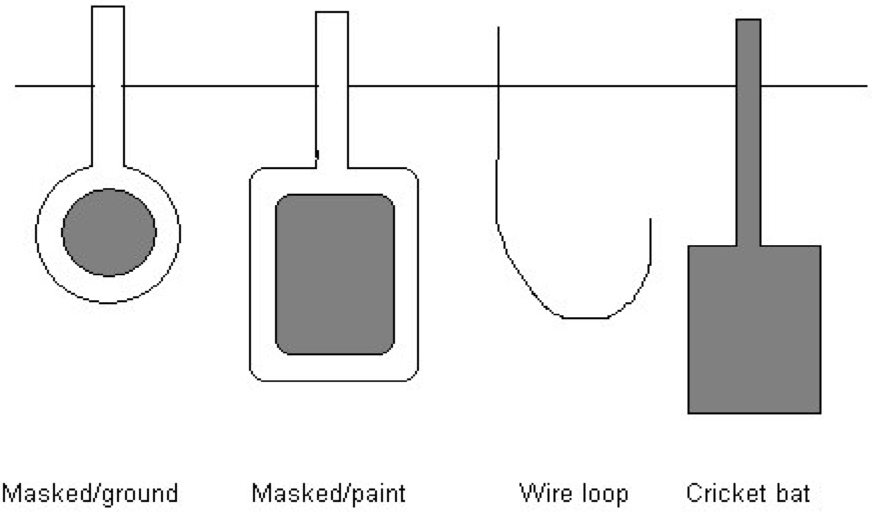
Many laboratory corrosion tests take place in flat bottomed glass reaction vessels with multi-access glass tops. These usually hold one litre of electrolyte and allow a thermometer, gas sparging tube, platinum auxiliary, lugin tube, and sample to be rigidly immersed in the electrolyte. The sample can take a few basic forms.

1 Fully submerged sample with electrical connection and edges masked using epoxy resin. After the resin has cured the sample face is ground down using a metallurgists polishing wheel to an acceptable finish and the cleaned.

2 The masked plate sample is also fully submerged with the back and sides covered in epoxy (or a beeswax\colophony mix) and an area of the front left clear. This is different to 1 above in that the sample is not ground down, making it suitable for painted samples.

3 The wire loop electrode is often used when testing materials like stainless steel to avoid crevices between the masking material and the sample. A thin loop of wire is immersed with very little in contact with the waterline.

4 The cricket bat style specimen is a modification of the wire loop for more solid pieces of metal. The sample is narrowed at the top so that the portion of the electrode in contact with the water line is small.



The reference electrode is often a commercial saturated calomel electrode (SCE) either directly dipped into the solution or connected remotely by a salt bridge and lugin probe. A small piece of platinum attached to the end of a glass rod makes the auxiliary electrode. A magnetic stirrer can be used along with a hot plate if required. If a lugin is used there is often a large amount of mains noise picked up between the test cell and the metallic part of the SCE, this noise acts as an additional reference voltage for the potentiostat, causing the cell to be polarised with a mains frequency voltage on top of the desired voltage. This additional signal can be of the order of 100's of mV so the working electrode is polarised by exactly this picked-up voltage. To remove this perturbing voltage use a noise reducing electrode in the cell.