

Trace₂o

METALYSER HM5000 BENCHTOP

Laboratory Heavy Metals Analyser



CONTENTS

| | |
|---------------------------------------|----|
| 1. INTRODUCTION | 4 |
| 2. BOX CONTENTS | 5 |
| 3. GETTING TO KNOW YOUR METALYSER | 6 |
| 4. INSTRUMENT INSTALLATION | 8 |
| 5. SENSOR MODULE | 10 |
| 6. CONNECTION SETUP | 16 |
| 7. INTRODUCING METAWARE | 18 |
| 8. USING THE METAWARE | 21 |
| 9. GRAPH TAB | 25 |
| 10. SPREADSHEET TAB | 28 |
| 11. MEASUREMENT TECHNIQUES | 31 |
| Linear Sweep | 31 |
| Differential Pulse | 32 |
| Square Wave | 33 |
| Cyclic Voltammetry | 34 |
| Table of Electrochemical Parameters | 35 |
| Method sequence | 36 |
| User configurable parameters & ranges | 37 |
| 12. CARE AND MAINTENANCE | 38 |
| 13. SPECIFICATIONS | 40 |
| APPENDIX 1: POWER SUPPLY FREQUENCY | 42 |
| APPENDIX 2: ACCESSORIES & CONSUMABLES | 44 |

1. INTRODUCTION

The Metalyser HM5000 Benchtop heavy metals analyser is the professional level product in Trace2o's popular Metalyser range. The instrument is a compact yet powerful voltammetric analyser, capable of running a variety of the most commonly used anodic and cathodic stripping techniques. Bespoke advanced PC-based software has been specifically designed to provide a simple yet comprehensive user interface, providing the analyst with complete control over the operation of the instrument, enabling optimisation of the performance of electrochemical methods. The following instructions explain how to install, set up and use your analyser.

The HM5000 performs potentiometric analysis where the voltage is controlled and current is measured. Typically an ASV method parameter file is loaded or creating which sets up the instrument. The sample is prepared and the analysis is performed. Usually the analysis will consist of a deposition phase, followed by a stripping phase for the element of interest. This will produce a graphical (voltammogram) of the current output from the cell. The voltammogram will feature peaks, the heights of which are proportionally related to the element(s) being analysed and can be measured in order to calculate the sample concentration.

Trace2o's expert team of Research and Development scientists are continually working on developing new methods for heavy metals analysis, and enhancing and improving existing methods. The latest application notes for these methods are available to registered customers on the members-only section of our website, trace2o.com.

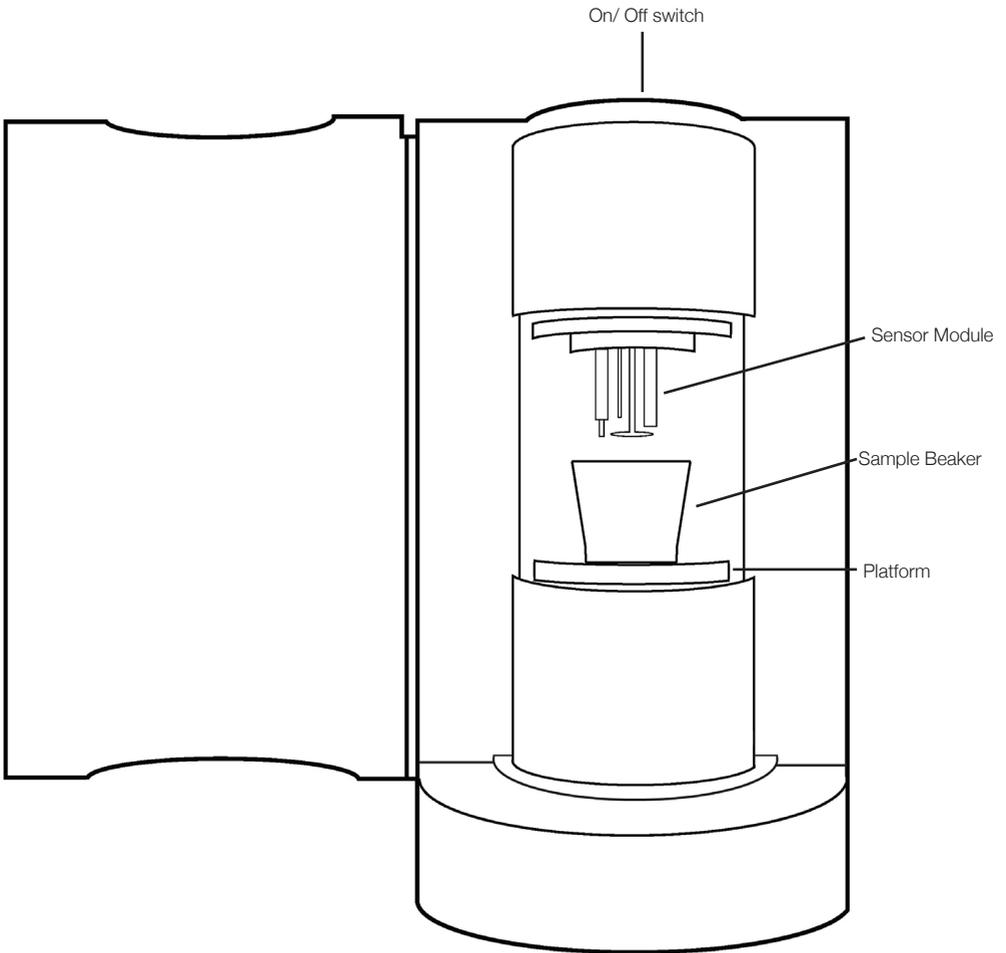
Customers with a handheld instrument from the Metalyser range can use measurement parameter files developed on the HM5000 on the handheld instrument. The measurement parameter file must be submitted to the Trace2o technical department. The file will then be converted for use on the handheld instruments by one of our team, who will liaise with you to ensure complete compatibility with your instrument. For more information on our file conversion service, contact after-sales support: customer.service@trace2o.com

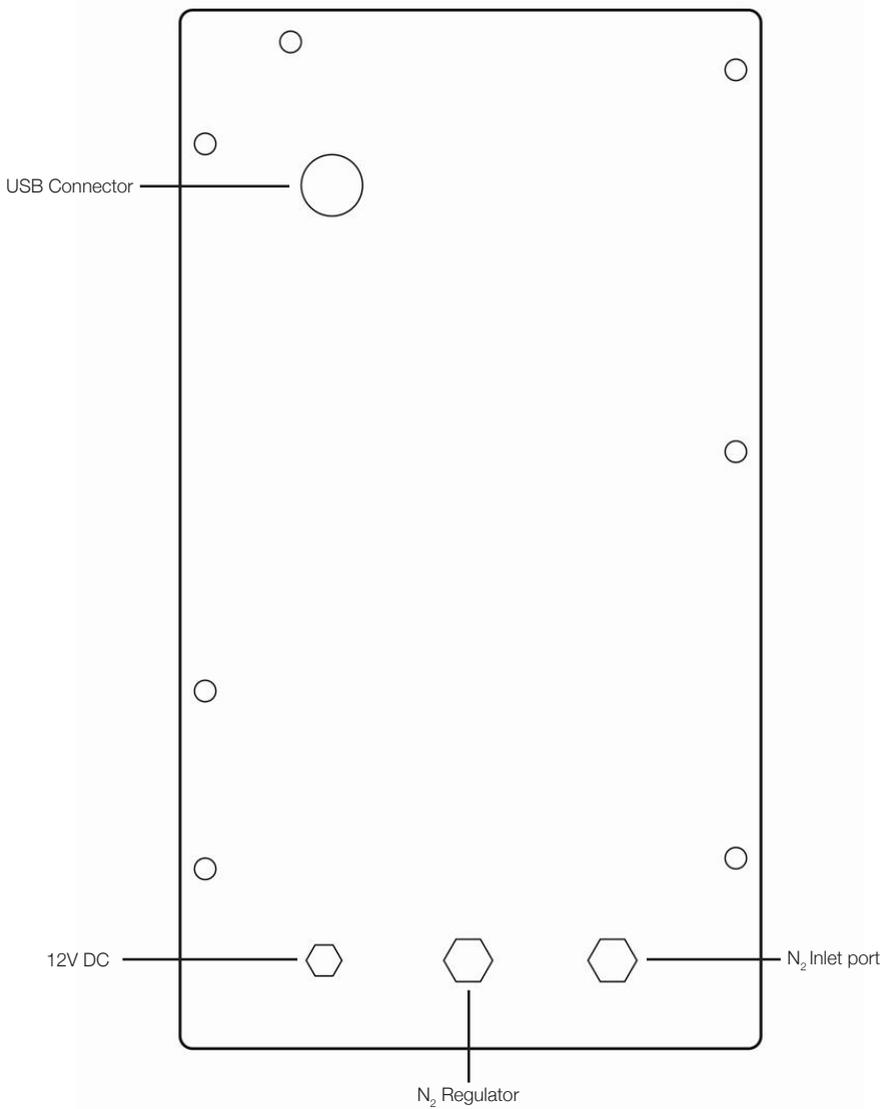
2. BOX CONTENTS

- Metalyser HM5000 Benchtop analyser
- 2 x GC working electrode
- 1 x Counter electrode
- 1 x Reference electrode
- 1 x Polishing platen
- 5 x Polishing cloth
- 1 x Bottle of polishing slurry
- 1 x Reference electrode fill solution bottle
- 1 x Bluetooth USB dongle
- 1 x USB lead
- 5 x Graduated sample analysis beaker
- 0.5 metres flexible N₂ tubing
- 20cm 4mm I/D rigid N₂ tubing
- Adapters and hose barb set
- Mains power adapter
- USB to 12V car charger adapter

In the unlikely event that any of these items are missing, contact Trace2o or your local distributor at your first opportunity.

3. GETTING TO KNOW YOUR METALYSER





4. INSTRUMENT INSTALLATION

Remove all of the packaging from the HM5000 instrument and ensure that all of the contents are present. If any items appear to be missing, please contact your local distributor or Trace2o.

Set the instrument on a firm level surface.

Connect the lead of the mains adapter to the socket marked 12V DC on the back of the instrument, and plug into a wall socket.

Nitrogen supply

If a nitrogen supply is to be connected then there are several connection options depending on the supply available, but the supply must have an external in-line regulator or adjustable tap.

Option 1: 4mm O/D Rigid tubing

4mm O/D rigid tubing can be connected directly to the N₂ inlet port on the back of the HM5000 without the use of additional tubing or adapters.



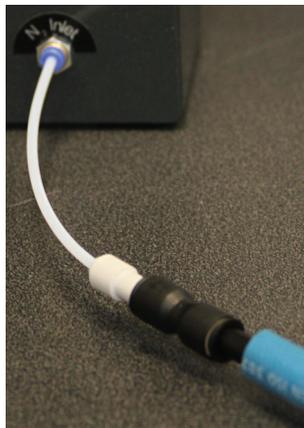
Option 2: 10mm O/D Rigid tubing

The supplied 20cm length of 4mm O/D rigid tubing should be connected to the N₂ inlet port on the back of the HM5000. The supplied adapter can then be used to connect this to 10mm O/D rigid tubing.



Option 3: 10mm I/D Flexible tubing

The supplied 20cm length of 4mm O/D rigid tubing should be connected to the N₂ inlet port on the back of the HM5000. The supplied adapter and hose barb can then be used to connect this to 10mm I/D flexible tubing. Secure the tubing to the hose barb with the clamp provided.



Priming the nitrogen system

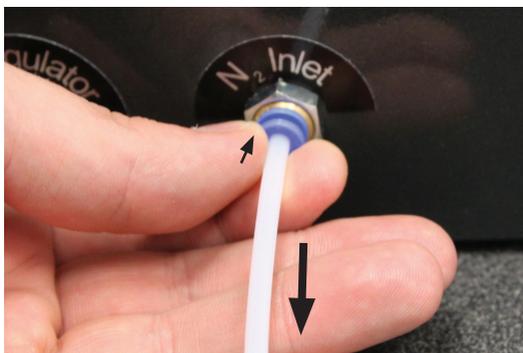
The nitrogen system must be primed to ensure that the built-in nitrogen regulator can effectively control the flow of nitrogen into the instrument.

- Close off the built-in nitrogen regulator on the back of the HM5000, by turning clockwise until it is finger tight.
- Slowly open the external in-line regulator or adjustable tap, until there is a small level of audible gas flow through the nitrogen nozzle in the sensor module.
- Slowly close the external in-line regulator or adjustable tap, until the audible gas flow stops.
- The nitrogen flow can now be controlled by the HM5000 built-in nitrogen regulator.

Removing the 4mm rigid tubing from the HM5000

The nitrogen inlet port has a locking mechanism to prevent accidental dislodging of the tubing from the instrument.

If the tubing needs to be removed for any reason push the surrounding locking ring towards the instrument and simultaneously pull on the tubing. The tube should be released.



5. SENSOR MODULE

Introducing the sensor module

The sensor module is covered with a protective cardboard sheath for transportation. Remove this sheath before use.

The sensor module comprises three removable electrodes:

Working electrode (W)



Counter electrode (C)



Reference electrode (R)



The working electrodes and reference electrode are supplied with protective rubber caps. These are intended to protect the electrode surfaces. They should be removed during analysis, and replaced when the instrument is not in use.

Trace2o recommends removing the reference electrode from the sensor module for storage. When replacing the protective cap for the reference electrode, fill with deionised water, as this will prevent the reference electrode from drying out.

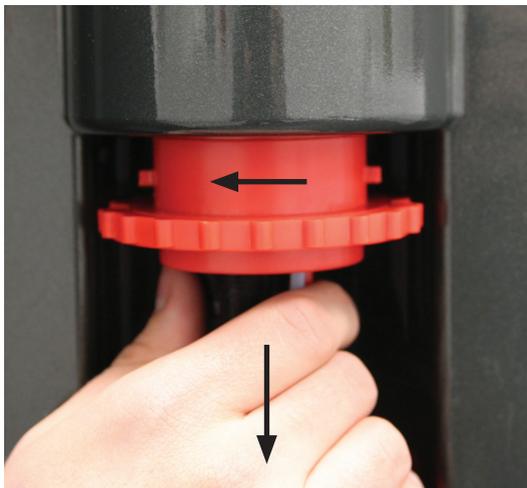
The sensor module also includes:

- Stirrer
- Temperature probe
- Nitrogen nozzle

Connecting/disconnecting the sensor module

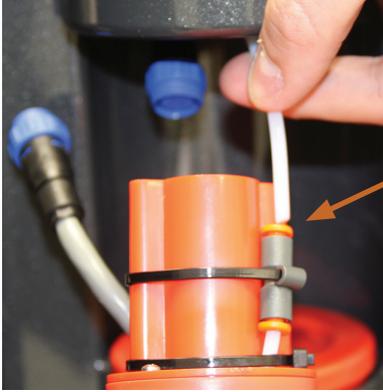
The sensor module can be disconnected and removed if necessary to facilitate cleaning of the HM5000 instrument.

To disconnect the sensor module from the body of the instrument, turn the sensor module cogwheel counter-clockwise to unlock, and carefully pull gently downwards.



To remove the sensor module completely, disconnect the 4mm nitrogen tubing from the in-line connector on the side of the sensor module, and unscrew the blue cable connector. The sensor module may now be completely removed.

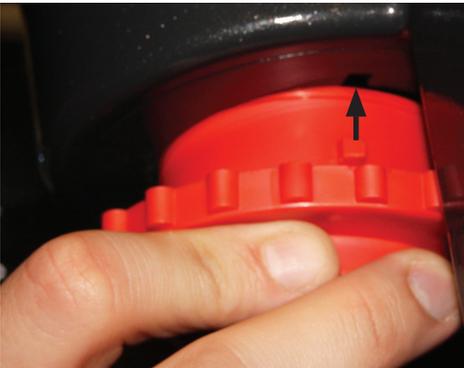
To reconnect the sensor module, plug the 4mm nitrogen tubing into the in-line connector.



Connect plug A to socket B of the blue cable connector and tighten the locking ring.



Ensure that the working electrode socket is located towards the front of the sensor module, then align the twist-lock pins with the slots on the instrument, slide the sensor module into place, and twist clockwise to lock.



Connecting/disconnecting the electrodes

The electrodes are all pre-fitted, but may need to be removed during the course of analysis, for example when switching between the two working electrodes.

To fit the electrodes, simply align the white arrow on the black connector shroud with the arrow on the socket and gently push the electrode. A quiet double click should be heard. Gently pull on the top of the black holder of the electrode to ensure it is fully connected.



To remove the electrodes, pull back on the black connector shroud and the connector will pull off.

Avoid knocking the stirrer whilst attaching or removing electrodes.

Insert the electrodes into the appropriate sockets as indicated by the electrode letters printed on the sensor module.



The working electrode has a 6-pin connector and will only fit into the W position.

The counter and reference electrodes both have 3-pin connectors.

The instrument requires all three electrodes in the correct positions to function correctly. Do not mix up the counter and reference electrodes.

Working electrode usage

The HM5000 is supplied with two working electrodes to prevent cross-contamination of samples.

Working electrodes should be properly cleaned between analyses. Cleaning instructions are provided at the back of the manual.

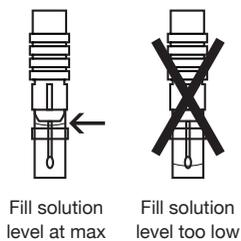
The working electrode comprises a disc of glassy carbon. Although supplied in a polished, ready-to-use state, gradual fouling of the electrode surface will occur by adsorption of buffers, analytes, or organic surfactants, which will reduce the sensitivity of the analysis. The electrode should therefore be regularly polished to ensure a smooth mirror-like surface using the supplied glass platen, polishing cloth and polishing solution.

See care and maintenance section for further details.

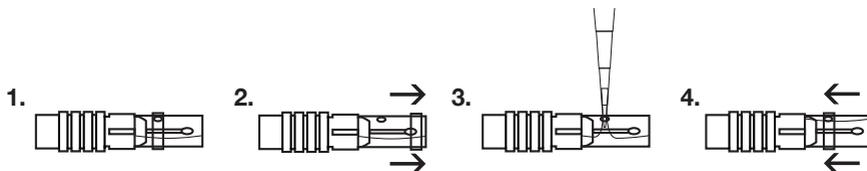
Reference electrode usage

The HM5000 has a custom-built reference electrode that has been designed to eliminate the need to drain and refill the reference chamber on a regular basis. The only maintenance required is to top up the fill solution in the reference chamber from time to time. The reference chamber should be kept at least 1/3rd full.

When necessary, replenish from the Reference Electrode Fill Solution bottle supplied with the unit. Slide the band down from the hole and use a micropipette to add fill solution to the reference chamber. Once topped up, replace the band to prevent the electrode fill solution escaping. Following addition of fill solution to the reference chamber, the reference electrode should be allowed to equilibrate for 24 hours before use.



Only use the supplied reference electrode fill solution.



Remember to remove the protective rubber cap before use, and to replace the cap after use to prevent the reference electrode from drying out. Add a few drops of deionised water to the cap before placing on the reference electrode.



Cleaning and maintaining the instrument, sensor module, and electrodes

Cleaning and maintenance instructions for the instrument, sensor module and all of the electrodes are provided at the back of this manual.

6. CONNECTION SETUP

Connecting a PC to the Metalysers HM5000

The Metalysers HM5000 can connect to a PC via either USB or Bluetooth. If, after initial setup the user later decides to connect via another method, initial setup will need to be repeated to re-establish the connection.

Initial Setup

USB Driver Installation

Your HM5000 instrument is compatible with most computers with a USB socket, but first the USB drivers may need to be installed. The drivers are located on the HM5000 CD ROM and are available for all versions of Windows post-95. Follow the procedure to ensure that the USB drivers are correctly installed.

- Insert the CD ROM into the CD drive.
- Ensure that the HM5000 instrument is connected to a power supply using the 12V DC adapter.
- Unscrew the blue protector cap on the rear of the HM5000, connect the waterproof USB cable to the HM5000 instrument, and tighten the screw cap.
- Connect the other end of the USB cable to an available USB port on your PC.
- Turn on the HM5000 by depressing the green power switch on the top of the instrument. The green indicator LED should turn on.
- Windows will run the 'install new hardware wizard'.
- When asked if Windows can connect to Windows update to search for software, select 'No, not this time'.
- Select 'Install from a list or specific location (Advanced)'
- Check 'Include this location in search' if applicable, and navigate to the CD ROM drive.
- Windows should install the drivers.
- Once complete, the wizard will run again. Use the same settings as before.
- Once Windows has installed the drivers, reboot the computer.

Bluetooth Installation

Your HM5000 instrument is able to communicate with a PC through Bluetooth. Most newer laptops and some desktops will have built-in Bluetooth connectivity; however, a Bluetooth adapter dongle is supplied with the instrument. Follow the procedure to correctly establish a connection to the instrument via Bluetooth.

- If use of the Bluetooth dongle is required, insert the Bluetooth dongle installation CD and follow the on-screen instructions to correctly install the Bluetooth software and drivers.
- Turn on built-in Bluetooth, or plug the supplied Bluetooth dongle into an available USB port on your PC.
- Turn on the HM5000 instrument by depressing the green power switch on the top of the instrument. The green indicator LED should turn on.
- Double-click the Bluetooth icon, found in the system tray.
- Depending on previous use of Bluetooth, the configuration wizard may load. Follow the on-screen instructions.
- Select 'Add device'.
- 'Metalyser 5XX-XX-XXX' should be displayed in the window, where the X values correspond to the serial number of your instrument. If it is not, turn the instrument off, wait five seconds, then turn the instrument on again.
- Select 'Metalyser 5XX-XX-XXX' and click 'Next'.
- 'Installing device driver software' may be displayed.
- You may be asked to enter the PIN for the instrument. This is pre-set as 1234.
- 'Your device is now ready to use' will be displayed.
- A Bluetooth connection between the Metalyser and the computer has now been established.

Software Installation

The HM5000 instrument is controlled through the Trace2o Metaware Benchtop software, which is provided on the HM5000 CD ROM.

- Load the CD ROM into the CD/DVD drive of the computer.
- Browse to the 'Metaware Benchtop Install' folder.
- Double click setup.exe
- Follow on-screen instructions.

7. INTRODUCING METAWARE

Loading Metaware

- Go to **Start Menu**
- Select **Programs**
- Select **Trace2o**
- Click **Trace2o Metaware Benchtop**.

The screenshot shows the Trace2o Metaware Benchtop software interface. The main window is titled "Trace2o Metaware - test.msdf" and contains a "Graph Tab" with a plot area. The plot area is currently empty, showing only axes labeled "Current (uA)" on the y-axis and "Volts (V)" on the x-axis. The plot area is labeled "Method Parameter Setup Tab".

On the right side of the window is a "Control Panel" with the following controls:

- Stirrer: Start, Stop buttons
- Speed: 100 (with a slider)
- Platform: Raise, Lower buttons
- Analysis: Start Analysis button
- Deposition Time: 60 sec (with a spinner)
- Analysis Options:
 - Repeats: 1 (with a spinner)
 - Use Calibration:
 - Plot Results:

At the bottom of the window is a "Table of Analyses" with the following columns: File name, Date and Time, Comments, Line colour, Scan method, Sample No., T/C, Baseline, and R.

At the very bottom of the window is a status bar with the following information:

- Connected
- Id: HM5000
- Ver: E020202-2.0L
- Benchtop
- 08/05/2012 15:25
- 20.2°
- Stirrer: Off
- Platform: Up
- ...

Labels with arrows point to the following components in the image:

- File Menu
- Tools Menu
- SpreadsheetTab
- Control Panel
- Graph Tab
- Method Parameter Setup Tab
- Voltammogram
- Table of Analyses
- Instrument ID
- Instrument Type
- Stirrer Status
- Platform Status
- Connection Status
- Date/Time
- Probe Temperature
- Potentiostat Status
- Firmware Version

Connecting to HM5000

If connecting via the USB cable:

Select **Tools**, then **Communications**, then **Metalyser HM5000**.

The connection status panel on the info pane at the bottom of the screen will change from 'Disconnected' to 'Connecting.....'

Disconnected

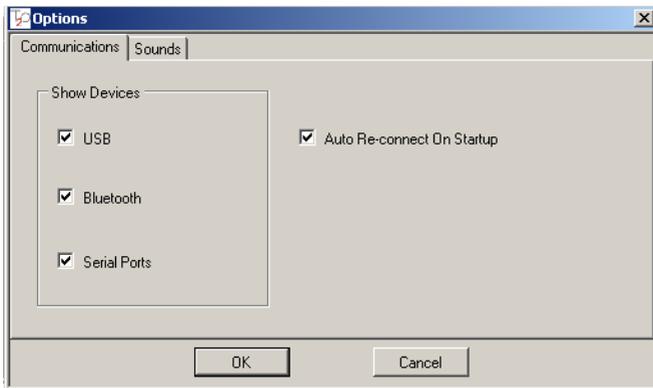
Connecting.....

When a connection has been successfully established, the connection status panel will read 'Connected'

Connected

and the serial number of the instrument will be displayed on the instrument ID panel.

If the Metalyser cannot be seen in the **Connections** sub-menu, then select **Tools**, then **Options**, and check the **Serial Ports** checkbox on the dialog box. Click **OK**, then look for **Metalyser HM5000 (COMX)** (where X is the numerical value of the COM port to which your Metalyser is connected), in the **Communications** sub-menu.



If connecting via Bluetooth

Select **Tools**, then **Communications**, then **Metalysers 5XX-XX-XXX** (where the X values correspond to the serial number of your instrument).

The connection status panel on the info pane will change from 'Disconnected' to 'Connecting.....'



When a connection has been successfully established, the connection status panel will display 'Connected'

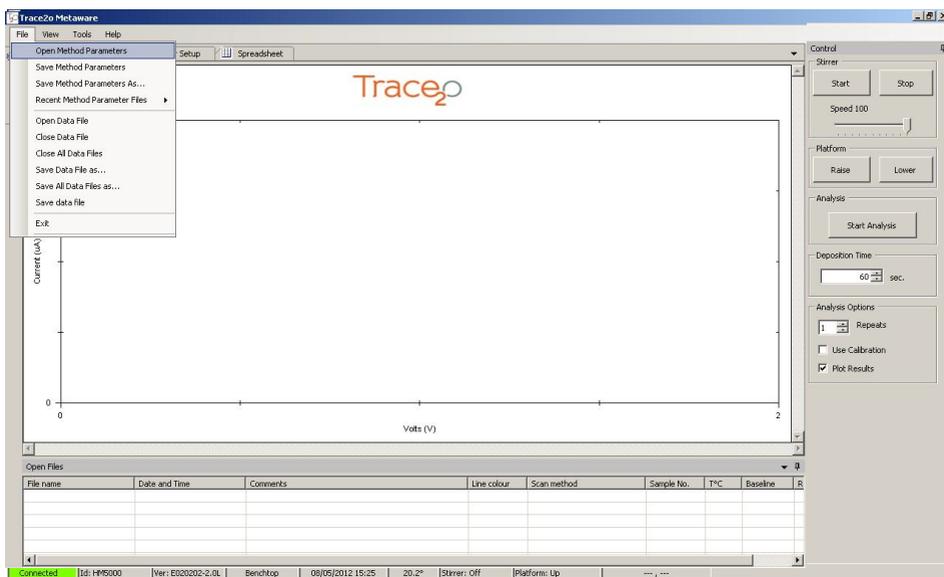


and the serial number of the instrument will be displayed on the instrument ID panel.

8. USING THE METAWARE

Loading a method parameter file

The HM5000 Installation CD includes a selection of method parameters and template files to get you started. Refer to the application notes provided with the instrument for information on how to carry out an analysis using these files. To load, click **File**, then **Open Method Parameters**. Navigate to the location of the method parameter file (.mxf file) and select the method parameter of choice. Click **Open** to open.



This will populate the Method Parameter Setup Tab. For more information on method parameter setup, see section X.

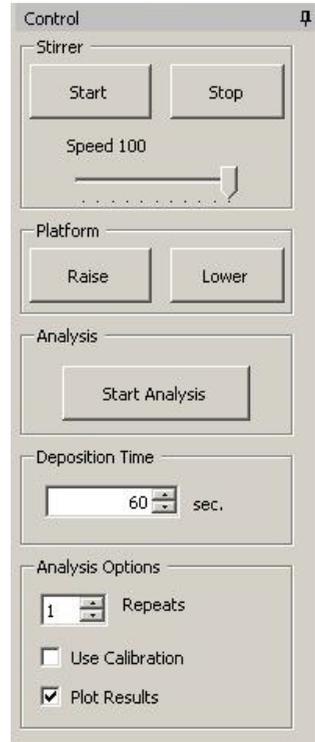
To load another saved method parameter file, repeat the process.

Only one method parameter file can be open at any given time.

On switching to a new method parameter file, the Metaware will ask the user to save any changes if amendments have been made to the file. Amendments to the method parameter files can also be saved using the **Save Method Parameters** option (overwrites previous saved file) or **Save Method Parameters As** option (creates a new file) on the **File** menu.

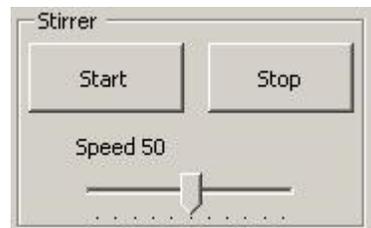
The control panel

The control panel is used for controlling the stirrer and the height of the adjustable platform, as well as setting the number of repeats, and starting the analysis.



Stirrer

The stirrer will run automatically throughout the conditioning, deposition and equilibrium steps of an analysis. It can also run independently of the potentiostat. To start the stirrer, click the **Start** button. To stop the stirrer, click the **Stop** button.



The stirrer has a variable speed setting (0-100) which is controlled by clicking and dragging the slider. The stirrer speed must be adjusted when the stirrer is turned off. It is recommended that the speed is set to no less than 5.

At full speed the stirrer runs at 4000rpm, so a setting of 5 would equate to 200rpm. If you are not sure whether the stirrer is running, the 'Stirrer' panel of the info pane at the bottom of the Metaware window will display 'On' or 'Off', dependent on the current status of the stirrer.

Stirrer: Off

Platform

The sample elevation platform must be lowered to facilitate insertion or removal of a sample, or addition of a standard. The platform must be raised during analysis so that the electrodes are submerged in the sample.

To raise the sample platform, click the **Raise** button.
To lower the sample platform, click the **Lower** button.



The current position of the sample platform is shown in the 'Platform' panel of the info pane at the bottom of the Metaware window. The panel will display either 'Down', signifying that the platform is lowered, 'Up', signifying that the platform is raised, or '?', indicating either that the platform is in motion, or that there is a fault.

Platform: Up

The HM5000 cannot run analyses with the sample platform in the down position.

If the 'Start Analysis' button is clicked and the sample platform is in the 'Down' position, the Metaware will inform the user by displaying the message:

Platform not raised. Raise platform and continue?

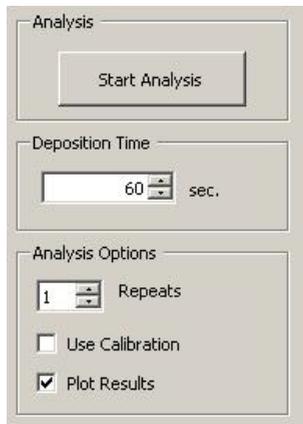
If **Yes** is clicked, the platform will be raised and the analysis will commence.

If **No** is clicked, the analysis will be cancelled.

Start Analysis

When **Start Analysis** is clicked, the HM5000 will run an analysis using the settings as currently specified in the Method Parameter Setup tab, for the number of repeats as specified in the **Repeats** box. The analysis may take a few seconds to start whilst the commands are sent to the instrument.

The deposition time can be set independently of the Method Parameter Setup tab, using the **Deposition Time** box.



The screenshot shows a software interface for the HM5000 instrument. It features three main sections: 1. 'Analysis' section with a 'Start Analysis' button. 2. 'Deposition Time' section with a numeric input field set to '60' and the unit 'sec.'. 3. 'Analysis Options' section with a 'Repeats' input field set to '1', a 'Use Calibration' checkbox which is unchecked, and a 'Plot Results' checkbox which is checked.

The Deposition Time box will ALWAYS override the Tdep settings in the Method Parameter Setup tab.

For more information on appropriate usage of the deposition time box, consult the relevant Trace2o application note for the analysis.

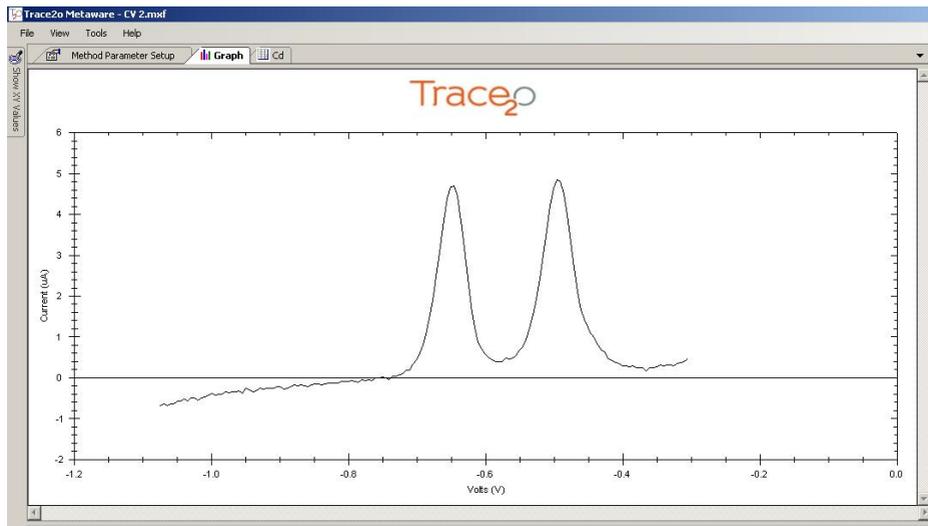
The **Plot Results** checkbox is intended for use when using the potentiostat for purposes other than analysis - for example, thin film plating of the working electrode. By default, the box is checked. If a graph is not required, uncheck the box.

Use Calibration

For information on the use of calibration, see section 10, detailing usage of the Spreadsheet tab.

9. GRAPH TAB

Once an analysis has run the voltammogram will be displayed and several functions can then be performed.



To manually find the cursor position for peak position and height, choose view from the top menu and then select cross hair value. A cross hair will appear which then shows the X value (peak potential) and the Y value (peak height) of the peaks on the left hand side of the screen

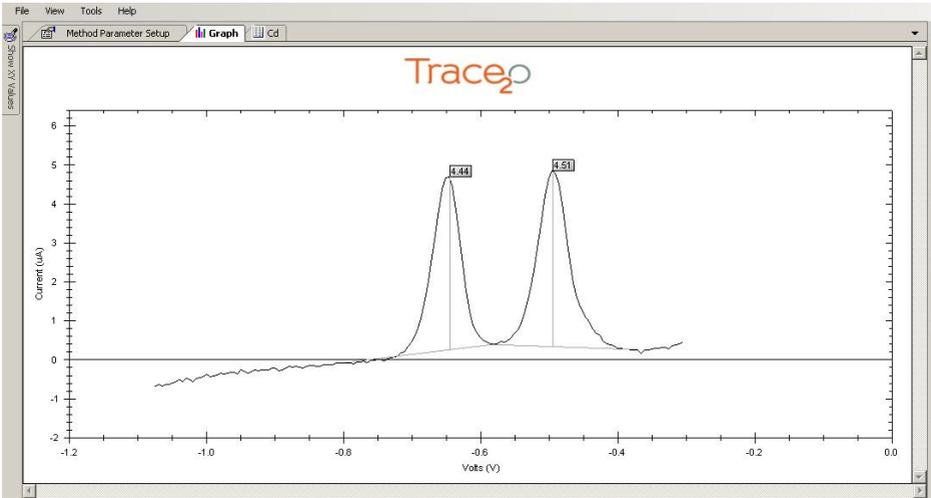
Right clicking the mouse whilst hovering over the graph will show these options:

- Copy: Copies the graph to the Windows clip board.
- Save Image As: Saves the image as a graphics file.
- Page Setup: Setup the printing options.
- Print: Prints the graph to a selected printer.
- Zoom Out: Zooms back out one zoom step.
- Reset Zoom: Restores the viewing window to the default setting.
- Set Scale to Default: Restores the axis to the default values.

Copy
 Save Image As...
 Page Setup...
 Print...
 Un-zoom
 Undo All Zoom/Pan
 Set Scale to Default
 Calculate Peak
 Abort Peak Calculation
 Remove all peak data
 Remove this peak data

Advanced

It is possible to measure the peak heights manually to provide raw current data which can assist with diagnostics and facilitate more accurate data analysis.



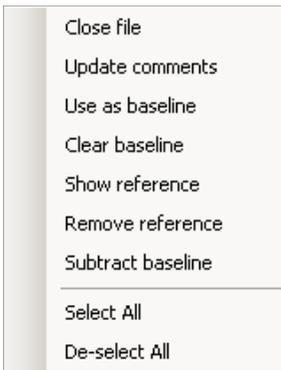
- Calculate Peak: To calculate a peak, first position the cursor over the data-point you wish to use as the right hand side of the peak then right click the mouse and click 'calculate peak'. If a valid point is not found this function will be greyed out. Move the mouse to the left hand side of the peak and left click the mouse. The baseline will be drawn in and the peak height shown, reported as a current (μA).
- Abort Peak Calculation: If an error is made during the selection of data points the calculation can be aborted.
- Remove all peak data: Removes all of the peak data from the graph.
- Remove this peak data: Used to remove result readings from voltammogram. Move the cursor over the peak result box and then select this option which will remove the peak data.

Show Data Points:

For more detailed information the data points can be displayed on the graph. Click the Tools option from the top menu and select the 'Show Data Points' option to toggle them off and on.

The table of analyses (open files) at the bottom of the screen displays all of the data currently loaded. The graphs can be toggled on and off using the tick box on the left for each individual graph. The file names of the graphs displayed are shown above the graph. The default filename is a date and time stamp in the format YYYYMMDD_HHMMSS. The filename can be altered by saving the files with a different name using the "File" menu and choosing "Save Data File As". If a file is saved with a different name the filename will not be displayed until the file is re-loaded.

Right-clicking on any of the open files gives the following options:



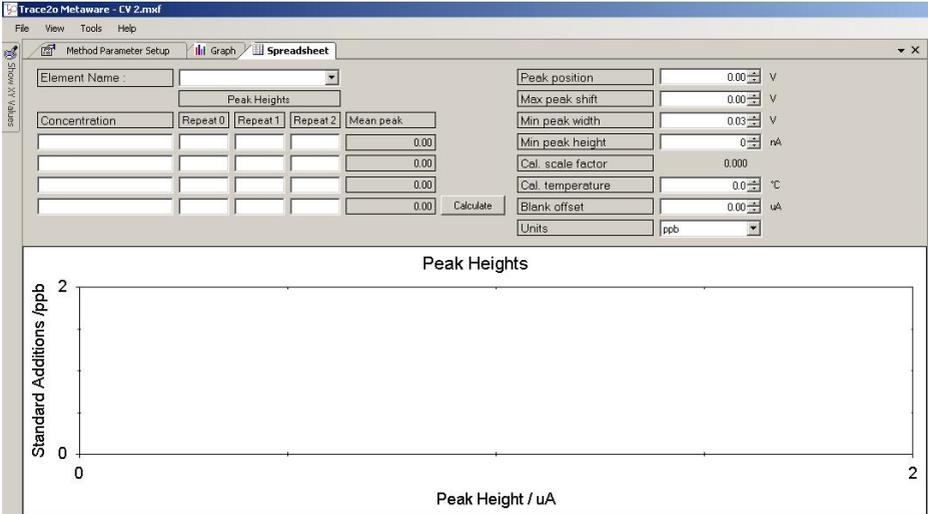
When a blank scan is run through the instrument, this can be designated as a baseline by right-clicking on the file and selecting 'Use as baseline'.

To subtract the baseline from subsequent analyses, right click on the file you wish to perform a background subtraction on, and click 'Subtract baseline'.

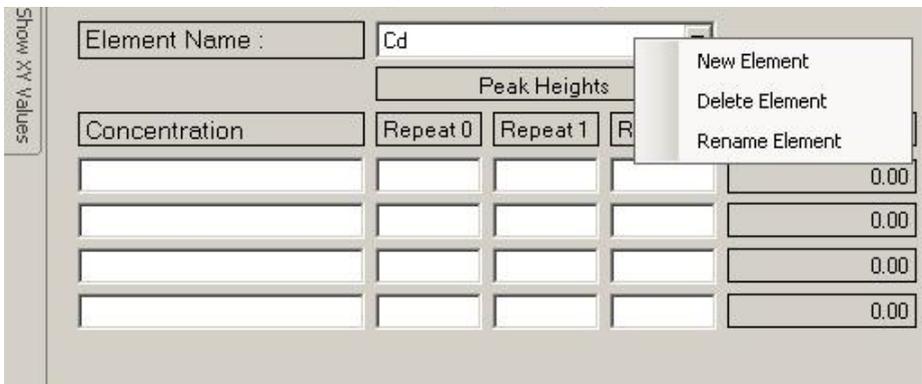
NB: The contents of the "File" menu will change according to which tab is currently in use.

10. SPREADSHEET TAB

The HM5000 has a built-in spreadsheet function enabling the user to perform linear regression on the analysis data as well as allowing the creation of a calibration curve for use on further samples.

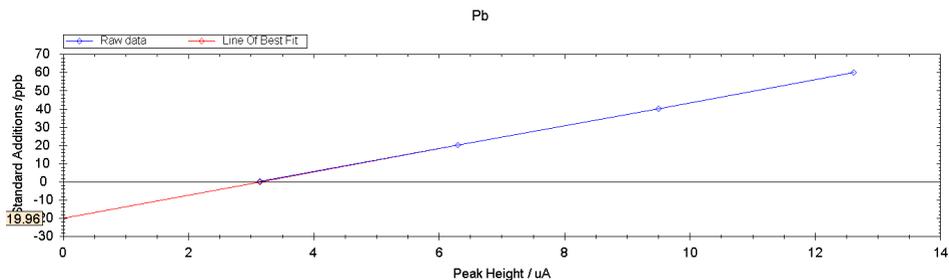


To create a spreadsheet, right-click on the drop-down menu and select New Element. Input an element name. Multiple spreadsheets can be created. Right-clicking on an existing element name offers the option to rename, or delete the spreadsheet as required.



| Peak Heights | | | | |
|---------------|----------|----------|----------|-----------|
| Concentration | Repeat 0 | Repeat 1 | Repeat 2 | Mean peak |
| 0 | 3.15 | 3.17 | 3.10 | 3.14 |
| 20 | 6.30 | 6.27 | 6.35 | 6.31 |
| 40 | 9.47 | 9.56 | 9.48 | 9.50 |
| 60 | 12.65 | 12.61 | 12.59 | 12.62 |

The table has four possible entries for the sample concentration. The first entry would normally be 0 as this is the unknown concentration of the sample that is trying to be established. The second, third and fourth entries would be the sample concentration after each addition of a standard. In the above example, three standard additions are performed, each of 20ppb, and the concentration is increased by 20ppb each time. The peak heights can be recorded for each of the additions, and up to three repeats for each addition can be entered. The table does not need to be filled. When all data is entered, click **Calculate** and the mean peak heights will be calculated and a graph plotted of concentration vs peak height. A line of best fit will be drawn through the data and the y-intercept shown, which corresponds to the unknown sample concentration for the metal of interest.



Calibration

The data calculated above is a calibration curve for the sample being analysed. If desired, this calibration can now be applied to new samples. To do this, some extra data is first required.

| | | |
|-------------------|-------|----|
| Peak position | -0.50 | V |
| Max peak shift | 0.08 | V |
| Min peak width | 0.03 | V |
| Min peak height | 0 | nA |
| Cal. scale factor | 6.331 | |
| Cal. temperature | 17.8 | °C |
| Blank offset | 0.00 | uA |
| Units | ppb | |

The calibration function requires the peak position (in V) to be set. Set the max peak shift (the maximum distance from the peak position where the peak is recognised), the min peak width for which the peak is recognised, and the min peak height for which the peak is recognised.

Set the calibration temperature (the temperature at which the calibration scans were carried out), and the blank offset (the peak height of the blank scan. This may be due to unavoidable contaminants in the buffer, for example natural levels of Pb in NaCl) and the units of the calibration.

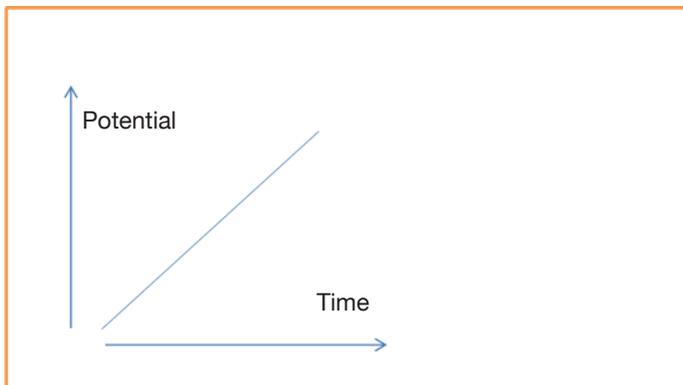
Click 'Calculate' and the HM5000 will use the data to create a calibration curve.

Thereafter, if 'Use Calibration' is checked, the HM5000 will use the calibration data that is stored and calculate the concentration of the sample in the units specified.

11. MEASUREMENT TECHNIQUES

The HM5000 is capable of running 4 different electrochemical voltammetric analysis techniques; Linear Sweep, Differential Pulse, Square Wave and Cyclic Voltammetry. The following gives a brief explanation of these techniques.

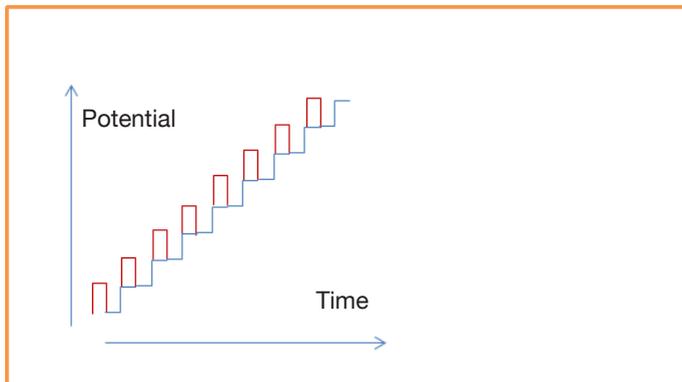
Linear Sweep



Linear sweep increases the potential from E_{begin} to E_{end} at the scan rate in V/s. In any given metal analysis, dependent on whether ASV or CSV is employed, the metal being examined is being either oxidised or reduced at the electrode at its redox potential. Linear sweep runs slowly and gives time for the reaction to occur, but the time at the redox potential is limited and the current generated is low.

| Technique | |
|--------------|--|
| Linear Sweep | |
| Linear Sweep | |
| Min Current | Max Current |
| 100uA | 100uA |
| E_{begin} | E_{end} |
| -1.10 V | -0.31 V |
| E_{step} | E_{cond} |
| 0.005 V | -0.05 V |
| E_{dep} | ScanRate |
| -1.10 V | 0.400 V/s |
| t_{cond} | t_{dep} |
| 20 s | 60 s |
| t_{eq} | Power supply frequency |
| 15 s | <input checked="" type="radio"/> 50 Hz <input type="radio"/> 60 Hz |

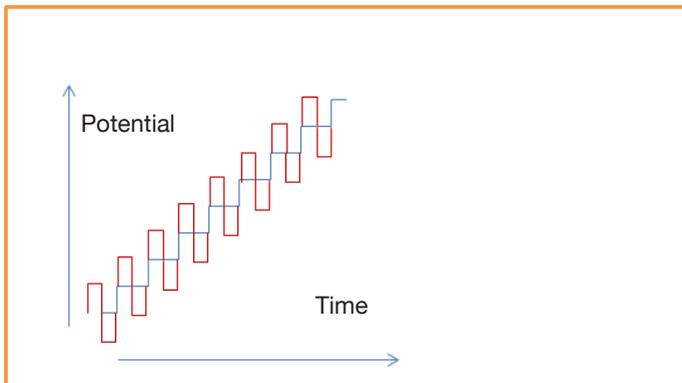
Differential Pulse



Differential Pulse is essentially a linear sweep scan with a voltage pulse at each of the voltage steps. The values of E_{pulse} and T_{pulse} set the amplitude and time of the pulses. The increased time at which the redox potential is held increases the current and provides a more sensitive technique. The instrument also takes measurements of current before and after the step and reports the difference which discriminates against the effects of capacitive current.

| | |
|-------------------|--|
| Technique | |
| DifferentialPulse | |
| DifferentialPulse | |
| Min Current | Max Current |
| 100µA | 100µA |
| Ebegin | Eend |
| -1.10 V | 0.00 V |
| Estep | Eamp |
| 0.005 V | 0.025 V |
| Econd | ScanRate |
| -0.05 V | 0.400 V/s |
| Edep | Power supply frequency |
| -1.10 V | <input checked="" type="radio"/> 50 Hz <input type="radio"/> 60 Hz |
| tcond | tdep |
| 20 s | 60 s |
| teq | tpulse |
| 15 s | 0.01 s |

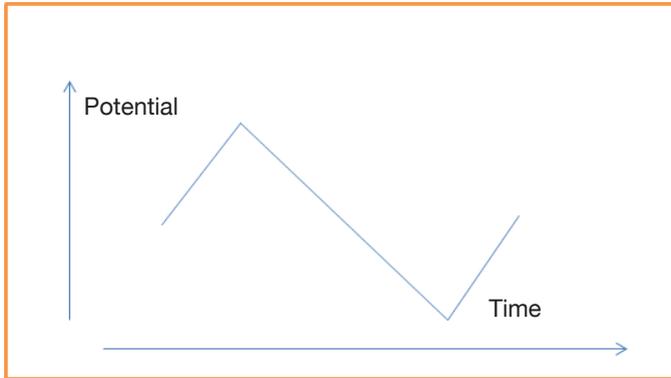
Square Wave



Square Wave voltammetry is also a derivative of linear sweep with a square wave super-imposed on the ramp but the centre of each square wave pulse is centred on the step potential. This method is the most effective against the effect of capacitive current and increases the signal to noise ratio giving lower detection limits.

| | |
|--|-------------|
| Technique | |
| Square Wave | |
| Square Wave | |
| Min Current | Max Current |
| 100uA | 100uA |
| Ebegin | Eend |
| -1.10 V | -0.31 V |
| Estep | Eamp |
| 0.005 V | 0.025 V |
| Econd | Edep |
| -0.05 V | -1.10 V |
| freq | tcond |
| 80 Hz | 20 s |
| tdep | teq |
| 60 s | 15 s |
| Power supply frequency | |
| <input checked="" type="radio"/> 50 Hz <input type="radio"/> 60 Hz | |

Cyclic Voltammetry



Cyclic Voltammetry is a reversible linear sweep. The scan starts at the potential E_{begin} and scans to E_{low} at which point it reverses and scans positive to E_{end} before reversing again and scanning back to the start potential.

| | |
|--|----------------------|
| Technique Cyclic Volts | |
| Cyclic Voltammetry | |
| Min Current 10nA | Max Current 100uA |
| Elow -1.00 V | Ehigh 1.00 V |
| Estep 0.050 V | Ebegin 0.00 V |
| Econd 0.00 V | Edep 0.00 V |
| ScanRate 0.051 V/s | tcond 0 s |
| tdep 0.0 s | teq 0 s |
| Power supply frequency <input checked="" type="radio"/> 50 Hz <input type="radio"/> 60 Hz | |

NB: Due to technical limitations of the instrument, the cyclic voltammetry function is limited to a maximum of 400 data points. If you are encountering problems with the cyclic voltammetry function, ensure that $E_{high} - E_{low} * 2 / E_{step} < 400$.

Electrochemical Parameters

The following table shows which method parameters are available with each of the four techniques outlined earlier. The drop down menu on the method parameter tab selects the technique and only those parameters available for that technique will be shown.

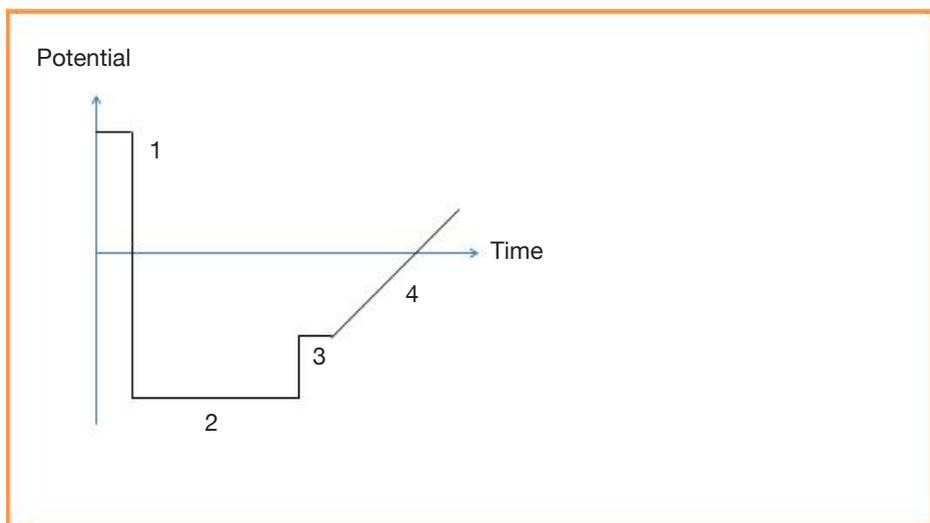
| | Units | Linear Sweep | Differential Pulse | Square Wave | Cyclic Voltammetry | Description |
|-----------|-------|--------------------------|--------------------------|--------------------------|--------------------------|---|
| Edep | V | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | | Deposition potential |
| Econd | V | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | | Conditioning potential |
| Estep | V | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | Step potential |
| Tpulse | s | | <input type="checkbox"/> | | | Time of pulse |
| Eamp | V | | <input type="checkbox"/> | <input type="checkbox"/> | | Pulse amplitude |
| Ebegin | V | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | Sweep start potential |
| Eend | V | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | | Sweep end potential |
| Elow | V | | | | <input type="checkbox"/> | Low potential at which sweep reverses |
| Ehigh | V | | | | <input type="checkbox"/> | High potential at which sweep reverses |
| Tdep | s | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | | Deposition time |
| Tcond | s | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | | Conditioning time |
| Tequil | s | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | | Equilibrium or rest time |
| Scan rate | V/s | <input type="checkbox"/> | <input type="checkbox"/> | | <input type="checkbox"/> | Rate at which voltage sweep is executed |
| Freq | Hz | | | <input type="checkbox"/> | | Frequency of square wave |

Method sequence

Once the method parameters have been set up the analytical scan can be run. The scans run in the following sequence:

1. Conditioning Step
2. Deposition Step
3. Equilibration Step at Ebegin
4. Scan Step

Once the scan is finished the graph will be drawn of potential vs current.



If the repeats box on the control panel is set to 2 or more the sequence will repeat from step 1 drawing a graph after each 4th step.

User configurable parameters and ranges

Min current/Max current - The current range should be optimised for the height range of peaks that are obtained. Run a preliminary scan with Min current set to 1nA and Max current set to 1mA to judge the expected current range of peak heights, then set both to create a window.

Edep – Deposition potential. -2.00V to +2.00V

Econd – Conditioning potential. -2.00V to +2.00V

Estep – Determines the distance in V between data points. 0.005V to 1V.

Tpulse – Time of pulse. 0.01 to 0.3 seconds.

Eamp – pulse amplitude. 0.001 to 0.25V.

Ebegin – Start potential of stripping step. -2.00V to +2.00V

Eend – End potential of stripping step. -2.00V to +2.00V

Elow - lowest potential of cyclic voltammetry sweep. -2.00V to +2.00V

Ehigh – highest potential of cyclic voltammetry sweep. -2.00V to +2.00V

Tdep – Deposition time. 0 to 4000 seconds

Tcond – Conditioning time. 0 to 4000 seconds

Teq – Time for the HM5000 to equilibrate the potential at Ebegin. 0 to 4000 seconds

ScanRate – How fast the sweep will occur. $>0.2 \cdot Estep$ to 0.5V/s.

Freq – Frequency of square wave. 1 to 100Hz.

Power supply frequency – 50Hz or 60Hz (See appendix 1)

NB: Ebegin must not be the same value as Eend. If the two values are set the same when the 'Start Analysis' button is clicked, the HM5000 will display an error message. For thin film plating of the working electrode, set Eend to 10mV less negative than Ebegin. For thin film plating, Ebegin and Eend should always be set at a potential less positive than the stripping potential of the plate.

12. CARE AND MAINTENANCE

With careful use, the maintenance requirements of the HM5000 instrument should be minimal. However, to ensure the continued operation of the instrument and minimise the potential for sample contamination, the instrument should be carefully cleaned at the end of every session

Instrument cleaning

Trace2o strongly recommends the use of IPA wipes for routine cleaning of the Metalyser HM5000 Benchtop instrument, sensor module, and electrodes. Other polar protic solvents (i.e. methanol, water) are also suitable for cleaning the instrument. Acetone and other polar aprotic solvents (DCM, THF) will damage the surface of the instrument and void the manufacturer's warranty.

Sensor module cleaning

The sensor module should be thoroughly rinsed with deionised water at the start and end of use, and whenever changing between samples. It is recommended to hold a sample cup as close to the electrodes as possible and squirt the electrodes using a wash bottle.

For in-situ deeper cleaning of the sensor module, use dilute hydrochloric or nitric acid (0.1M) in a sample beaker and run the stirrer for several minutes.

Care of your electrodes

Counter electrode – an occasional quick visual inspection is required to ensure that the electrode has no physical damage.



Reference electrode – regularly perform a visual inspection to ensure that the electrode has no physical damage, and that there is sufficient liquid (at least 1/3rd full) in the reference chamber, and no bubbles in the inner tube. The fill solution in the reference chamber should be kept replenished using the Reference Electrode Fill Solution provided in the kit. For best performance, the reference electrode tip should be kept wet. This can be accomplished by adding a few drops of deionised water to the protective cap when storing the electrode.

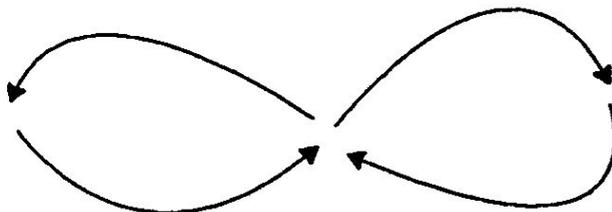


Working electrode – the electrode surface should be examined for cracks and imperfections that will affect analysis. The glassy carbon should be regularly polished to remove contaminants and ensure a smooth mirror-like surface using the supplied glass platen, polishing cloth and polishing solution. Ensure that the protective rubber cap is replaced during storage of the electrode to prevent damage to the electrode surface.



Polishing technique

To polish the electrode firstly make sure the glass platen and holder are clean and free of dust or dirt which may cause scratching. Place a clean cloth on the platen and dampen the cloth with the polishing solution provided. Hold the electrode perpendicular to the platen and use a smooth figure of eight motion as indicated.



Polish until the surface has a mirror finish and no scratches or imperfections are seen.

Maintenance and warranty

With normal usage, the HM5000 should only require minimal maintenance – however if your instrument develops a fault, contact Trace2o Technical Support or your distributor in the first instance. Trace2o products are provided with a full one year warranty. It is recommended that your HM5000 is returned to your local dealer for an annual service to keep the instrument in good condition. For more information, please contact Trace2o.

Firmware and software updates for the HM5000 are available, free of charge, via the Trace2o website or through your local distributor.

NB: The HM5000 is shipped with tamper-proof labels. Any attempt to disassemble the instrument without authorisation from Trace2o will damage the tamper-proof labels and void the manufacturer's warranty. Trace2o will not accept any returns of items disassembled without authorisation, and Trace2o warranty department will not repair items without an intact label.

13. SPECIFICATIONS

Metalyser HM5000 Benchtop Unit

Power supply: 12V DC, 1.5A, 2.1mm DC Jack

Nitrogen inlet: 4mm push fit

Nitrogen outlet: 4mm PTFE rigid tube

Nitrogen regulator: Max pressure 10Bar

Materials: ABS-ESD, PC, PEEK, PTFE

Communications:

USB: Virtual Comm port 57600 Baud

Bluetooth: V2.0 2.4Ghz

Potentiostat:

DC Potential range: $\pm 2V$

DC Potential resolution: 0.001V

Output current range: $\pm 10mA$

Input current range: 1nA – 1mA (7 ranges)

Resolution: 0.1% of current range

AC Potential Range: 0 – 0.25V

AC Potential Resolution: 0.001V

Electrodes:

Working electrodes: 3mm Glassy Carbon disc. Impedance < 1.5 ohm

Counter (Auxiliary) Electrode: Platinum foil. Impedance < 0.2 ohm

Reference Electrode: Silver/Silver Chloride double junction. 3M KCL filled

APPENDIX 1: POWER SUPPLY FREQUENCY

Regardless of the measurement technique used, the power supply frequency appropriate to the country of use must be set, to either 50 Hz or 60 Hz. This eliminates interference from fluctuations in mains AC power supply. Set the power supply frequency to that of your country. A table is provided below listing the power supply frequencies in most countries. The list is not intended to be exhaustive and may change over time. Some geographical and other exceptions are listed in footnotes.

| 50 Hz | | | | |
|------------------|-----------------------------|------------------|---------------|---------------|
| Afghanistan | Burma (Myanmar) | Eritrea | India | Macedonia |
| Albania | Burundi | Estonia | Indonesia | Madagascar |
| Algeria | Cambodia | Ethiopia | Iran | Madeira |
| Andorra | Cameroon | Faroe Islands | Iraq | Malawi |
| Angola | Canary Islands | Falkland Islands | Ireland | Malaysia |
| Argentina | Cape Verde | Fiji | Isle of Man | Maldives |
| Armenia | Central African Republic | Finland | Israel | Mali |
| Australia | Chad | France | Italy | Malta |
| Austria | Channel Islands | French Guiana | Jamaica | Martinique |
| Azerbaijan | Chile | Gaza Strip | Jordan | Mauritania |
| Azores | China | Gabon | Kazakhstan | Mauritius |
| Bahrain | Comoros | Gambia | Kenya | Moldova |
| Balearic Islands | Congo, Republic | Georgia | Kiribati | Monaco |
| Bangladesh | Congo, Democratic Republic | Germany | Kuwait | Mongolia |
| Barbados | Cook Islands | Ghana | Kyrgyzstan | Montenegro |
| Belarus | Côte d'Ivoire (Ivory Coast) | Gibraltar | Laos | Morocco |
| Belgium | Croatia | Greece | Latvia | Mozambique |
| Benin | Curaçao | Greenland | Lebanon | Namibia |
| Bhutan | Cyprus | Grenada | Lesotho | Nauru |
| Bolivia | Czech Republic | Guadeloupe | Liberia (1) | Nepal |
| Bosnia | Denmark | Guinea | Libya | Netherlands |
| Botswana | Djibouti | Guinea-Bissau | Lithuania | New Caledonia |
| Brunei | Dominica | Hong Kong | Liechtenstein | New Zealand |
| Bulgaria | Egypt | Hungary | Luxembourg | Niger |
| Burkina Faso | Equatorial Guinea | Iceland | Macau | Nigeria |

| 50 Hz | | | | |
|------------------|---------------------------|--------------|--------------|----------------|
| Norway | Rwanda | Slovakia | Tajikistan | Ukraine |
| Oman | St. Lucia | Slovenia | Tanzania | UAE |
| Pakistan | Saint Pierre and Miquelon | Somalia | Thailand | United Kingdom |
| Papua New Guinea | St. Vincent & Grenadines | South Africa | Timor-Leste | Uruguay |
| Paraguay | Samoa | Spain | Togo | Uzbekistan |
| Poland | São Tomé and Príncipe | Sri Lanka | Tonga | Vanuatu |
| Portugal | Senegal | Sudan | Tunisia | Vietnam |
| Qatar | Serbia | Swaziland | Turkey | Yemen |
| Réunion | Seychelles | Sweden | Turkmenistan | Zambia |
| Romania | Sierra Leone | Switzerland | Uganda | Zimbabwe |
| Russia | Singapore | Syria | | |

| 60 Hz | | | | |
|----------------|--------------------|------------|---------------------|--------------------------|
| American Samoa | Cayman Islands | Guyana (3) | North Korea | Saudi Arabia |
| Anguilla | Colombia | Guatemala | Okinawa | South Korea |
| Antigua | Costa Rica | Haiti | Panama | Suriname |
| Aruba | Cuba | Honduras | Peru (4) | Taiwan |
| Bahamas (2) | Dominican Republic | Mexico | Philippines | Trinidad & Tobago |
| Belize | Ecuador | Micronesia | Puerto Rico | United States of America |
| Bermuda | El Salvador | Montserrat | St. Kitts and Nevis | Venezuela |
| Brazil | Guam | Nicaragua | St. Martin | Virgin Islands |
| Canada | | | | |

| Mixed | | |
|---------------------------|-----------|------------|
| Caribbean Netherlands (5) | Japan (6) | Tahiti (7) |

Footnotes/Exceptions

- 1) Previously 60 Hz, now officially 50 Hz. Many private power plants are still 60 Hz.
- 2) 50 Hz in some outlying areas.
- 3) Mixture of 50 Hz and 60 Hz distribution. Conversion of 50 Hz to 60 Hz ongoing.
- 4) Some areas 50 Hz.
- 5) Bonaire 50Hz, Saba and St. Eustatius 60Hz.
- 6) East Japan 50Hz (Tokyo, Kawasaki, Sapporo, Yokohama, and Sendai); West Japan 60 Hz (Okinawa, Osaka, Kyoto, Kobe, Nagoya, Hiroshima)
- 7) Marquesas Islands 50 Hz

APPENDIX 2: ACCESSORIES & CONSUMABLES

| Replacement parts | |
|---|-----------|
| Replaceable sensor module (electrodes to be ordered separately) | HMA110051 |
| Working electrode* | HMA110002 |
| Reference electrode* | HMA110004 |
| Counter electrode* | HMA110005 |
| Rechargeable battery kit (battery, leads and charger) | HMA120011 |
| Mains adapter* | HMA120021 |
| USB cable* | HMA120002 |
| Beaker, 250mL | HMA120003 |
| Electrode wash bottle | HMA120004 |
| Graduated sample analysis beaker* | HMA120015 |
| Micropipettor, 200-1000 μ L | HMA120009 |
| Micropipettor, 10-100 μ L | HMA120010 |
| USB Bluetooth dongle | HMA120013 |

| Non-chemical consumables | |
|---|-----------|
| Polishing dish with glass platen* | HMC900007 |
| Polishing cloth (Pk 5)* | HMC900008 |
| Polishing solution 30mL* | HMC900009 |
| Polishing solution 250mL | HMC900019 |
| Micro pipette tips 200-1000 μ L | HMC900003 |
| Micro pipette tips 10-100 μ L | HMC900006 |
| Reference electrode fill solution, 8mL* | HMC900004 |

| Buffers and Standards Packs (containing Buffer Sachets & Standards) | |
|---|-----------|
| M1 Buffer & standards pack for 50 tests (Cd, Pb, Cu, Zn) | HMC301050 |
| M1 Buffer & standards pack for 200 tests (Cd, Pb, Cu, Zn) | HMC301200 |
| M2 Buffer & standards pack for 50 tests (Hg) | HMC302050 |
| M2 Buffer & standards pack for 200 tests (Hg) | HMC302200 |
| M3 Buffer & standards pack for 50 tests (As) | HMC303050 |
| M3 Buffer & standards pack for 200 tests (As) | HMC303200 |
| M6 Buffer & standards pack for 50 tests (Mn) | HMC306050 |
| M6 Buffer & standards pack for 200 tests (Mn) | HMC306200 |

| Buffer Sachets | |
|---|-----------|
| M1 Buffer sachets for 50 tests (Cd, Pb, Cu and Zn) | HMC101050 |
| M1 Buffer sachets for 200 tests (Cd, Pb, Cu and Zn) | HMC101200 |
| M2 Buffer sachets for 50 tests (Hg) | HMC102050 |
| M2 Buffer sachets for 200 tests (Hg) | HMC102200 |
| M3 Buffer sachets for 50 tests (As) | HMC103050 |
| M3 Buffer sachets for 200 tests (As) | HMC103200 |
| M6 Buffer pack for 50 tests (Mn) | HMC106050 |
| M6 Buffer pack for 200 tests (Mn) | HMC106200 |

| Standards | |
|---|-----------|
| M1 Standard for Cd and Pb (50 tests at 20ppb per test) | HMC201050 |
| M1 Standard for Cd and Pb (200 tests at 20ppb per test) | HMC201200 |
| M2 Standard for Hg (50 tests at 20ppb per test) | HMC202050 |
| M2 Standard for Hg (200 tests at 20ppb per test) | HMC202200 |
| M3 Standard for As (50 tests at 20ppb per test) | HMC203050 |
| M3 Standard for As (200 tests at 20ppb per test) | HMC203200 |
| M4 Standard for Cu (50 tests at 20ppb per test) | HMC204050 |
| M4 Standard for Cu (50 tests at 20ppb per test) | HMC204200 |
| M5 Standard for Zn (50 tests at 20ppb per test) | HMC205050 |
| M5 Standard for Zn (200 tests at 20ppb per test) | HMC205200 |
| M6 Standard for Mn (50 tests at 20ppb per test) | HMC206050 |
| M6 Standard for Mn (200 tests at 20ppb per test) | HMC206200 |

| Conditioning Solutions | |
|--|-----------|
| M1, M4, M5 Conditioning Solution 100mL | HMC401000 |
| M2 & M3 Conditioning Solution 100mL | HMC402000 |
| M6 Conditioning Solution 100mL | HMC406000 |

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