

Trace₂

METALYSER[®] SOILS HM4000

Portable Heavy Metals
Analyser for Soils



CONTENTS

1.	Introduction	3
2.	Kit Content	4
3.	Metalyser®	5
3.1	Getting to Know Your Metalyser®	5
3.2	Getting Started	15
3.3	Operating Procedure	18
3.4	Troubleshooting	25
3.5	Interference Effects	27
3.6	Specifications	27
4	Soil Extraction Kit	28
4.1	Getting to Know Your Soils Kit	28
4.2	Safety First	29
4.3	Digestion Procedure for Dry Soils	30
4.4	Digestion Procedure for Wet Soils (Quick)	31
4.5	Digestion Procedure for Wet Soils (Precise)	33

1. INTRODUCTION

The Metalyser® Soils HM4000 kit is ideal for testing of very low heavy metal concentrations in soil samples, for example in industrial sites, farms, mines and river banks etc. The HM4000 is a rugged field kit that tests for a range of 6 heavy metals and can be used both as a collection and measurement device for at-site analysis. The kit contains a digestion kit for preparation of soil extracts. These aqueous extracts can then be analysed using the Metalyser® instrument. It is very portable, using a battery as its power source and can provide results to <10mg/kg rapidly.

The Metalyser® HM4000 utilises a voltammetric technique to detect heavy metal ions in solution. The technique is not new – it was first proposed in the 1920s – but recent advances in electronics and software have provided a means to make the instrument portable. The instrument takes a complex idea but makes it easy to understand and is therefore usable for a competent person not trained in voltammetry. No complex chemical techniques need to be employed during the course of the analysis.

The HM4000 is very robust and has been designed to be virtually maintenance free. The instrument is sealed against water ingress and is housed in a waterproof case, making it suitable for use onsite for carrying out field screening analysis. The kit is equipped with all necessary instrumentation, consumables and safety apparatus for safe and precise analysis of soil samples.

The Metalyser® is ready out-of-the-box and can be used after charging the built-in battery. After a period of non-use, the Metalyser® is ready to operate very quickly after putting the sonde head into the water sample.

The soil extraction procedure is outlined in Section 4 of this manual. The soil extract prepared in this stage can be used for analysis of its heavy metal contents using the Metalyser® HM4000 instrument as outlined in Section 3 of this manual.

It is recommended to familiarize yourself with the instrument first using drinking water samples before advancing to the soil extraction procedures.

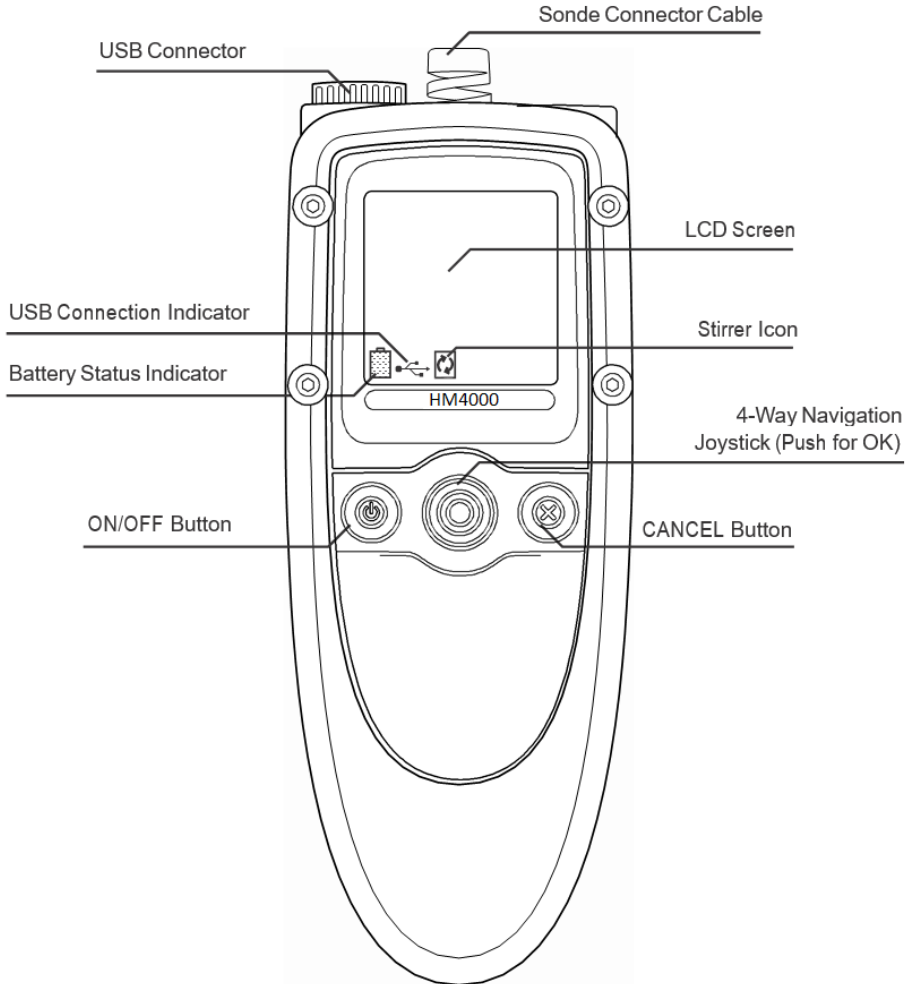
Note The results shown will be in units of mg/kg but for water samples this can be directly correlated to ppb.

2. KIT CONTENT



3. METALYSER®

3.1 GETTING TO KNOW YOUR METALYSER®



USB Connector

The USB connector can be used to charge the battery using the supplied car or mains charger, and also for download/upload and charging via a PC or laptop.

LCD Screen

The LCD screen tells you what is happening as you go along. It will continually display the current date and time as well as other useful indicators such as battery and connection status. The screen is also backlit enabling use of the instrument under poor lighting conditions.

ON/OFF Button

The ON/OFF is used to turn the instrument on and off, there is also an optional ten minute auto shutdown feature should you leave the instrument unattended.

Navigation Joystick

The joystick allows you to quickly and easily navigate your way through the menus and features of the Metalyser®. The joystick provides five controls; Up, Down, Left, Right and OK. To navigate up, down, left and right simply push the joystick in the required direction. To select an option or 'OK' a choice press the joystick in the centre.

Cancel Button

Pressing the cancel button will return you to the previous menu or screen.

On Screen Indicators

At the bottom of the screen these symbols will appear periodically to provide information about the instrument's status.



Battery fast charge indicator



Battery trickle charge indicator



Battery fault/overheat indicator



Battery Full



Battery $\frac{3}{4}$ Full



Battery $\frac{1}{2}$ Full



Battery $\frac{1}{4}$ Full



Battery empty

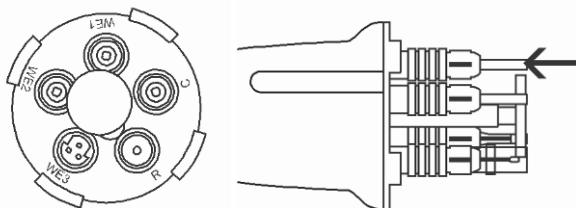


Stirrer on indicator

Components and care of components

The Sonde

The sonde head contains the electrodes, stirrer and temperature probe to carry out analysis. It comes pre-assembled with the 4 electrodes and a Sample Analysis Beaker (SAB). The electrodes are the WE1 (Working Electrode 1), WE2 (Working Electrode 2), R (Reference Electrode) and C (Counter Electrode). The sonde has the electrode letter references embossed onto it to ensure they are connected in the correct positions.



To fit an electrode, simply align the arrow on the electrode with the arrow on the sonde head and push the electrode into its socket. 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 the connector shroud with the arrow on and the connector will pull off.

When attaching or removing the electrodes, be careful to avoid touching the stirrer.

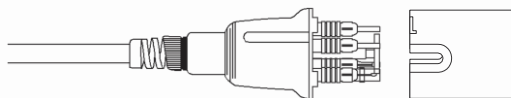
The different Working Electrodes can be identified by the number of connecting pins and are not interchangeable.

Electrode WE1 (6 pins) is to be fitted in position WE1.

Electrode WE2 (3 pins) is to be fitted in position WE2.

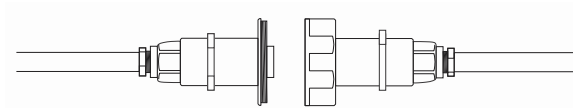
Do not mix up the electrodes as this can cause damage to the instrument.

The sonde can be assembled in one of two ways. Firstly, the short cable attached to the Sonde can be connected directly to the instrument. It can then be attached to the clamp and stand included in the kit and fitted inside the box. The second option is to fit the extension cable between the sonde and the instrument. This allows the instrument to be used to collect samples as well as analyse them.



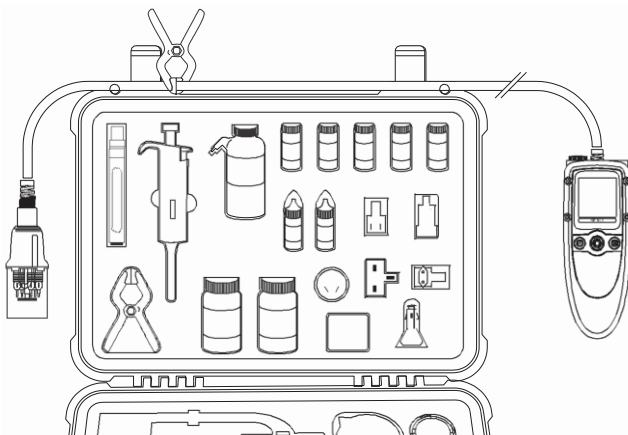
The sonde (electrodes x 4)

SAB (Sample Analysis Beaker)



In-line waterproof cable connector

Warning: Sonde head is designed for submersion up to 1 metre in water. Lowering the head deeper than this may result in water ingress which will require sonde head replacement.



Note: The sonde cable may be fixed to the lid of the Metalysen[®] box, using the box closure tabs and the supplied cable clamp. Alternatively, the user may attach the cable to a suitable fixture at the sample site, e.g. branch, rope, fence, barrier.

Alternatively, a clamp and stand are provided. The stand slots into the hole in the right hand rear corner of the box. This can be used to clamp the sonde as show above, which improves stability during the analysis.

Electrodes



Counter electrode – this electrode needs the least maintenance of all. A quick visual inspection to ensure the electrode has no physical damage.



Reference Electrode – this electrode contains a liquid filled tube, the liquid can be replenished by using the **Reference Electrode Fill Solution** provided in the kit. Perform a visual inspection to ensure it has no physical damage and ensure there is enough liquid in the outer tube and that there are no bubbles in the inner tube. **For best performance, the Reference Electrode tip should be kept wet. Please remove cap before use, and replace after use.** Always place a few drops of deionised water in the cap before replacing it on the electrode.



Working Electrode – the electrode surface should be examined for cracks and imperfections that will affect analysis. The electrode should be regularly polished to remove contaminants and ensure a smooth mirror-like surface using the supplied glass platen, polishing cloth and polishing slurry (see below). Ensure that the protective rubber cap is replaced during storage of the electrode to prevent damage to the electrode surface. After polishing, conditioning steps needs to be undertaken before analysis. The different working electrodes can be identified by the number of connecting pins and are not interchangeable. Electrode WE1 (6 pins) is to be fitted in position WE1. Electrode WE2 (3 pins) is to be fitted in position WE2. **Do not mix up the electrodes.**

Refilling the Reference Electrode

The Reference Electrode cap needs to be removed prior to analysis and replaced when the electrode is going to be stored. It is best practise not to let the tip of the Reference Electrode dry out. To ensure this, always add a few drops of deionised water into the cap before replacing on the electrode.

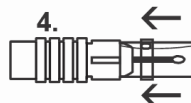
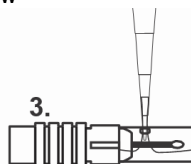
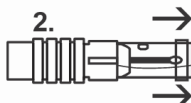
The Reference Electrode must contain **Reference Electrode Fill Solution** to operate. Ensure the Reference Electrode is held vertically with the connector upwards and determine that there is enough solution in the Reference Electrode.



Fill Solution at max



Fill Solution level too low



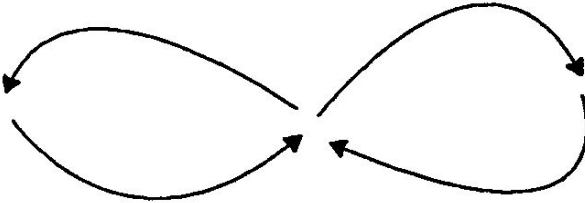
The outer tube needs to be at least 1/3 full. To top the **Reference Fill Solution**:

1. Place the reference electrode horizontal with the hole facing up.
2. Slide the band down from the hole.
3. Use the pipette to drop enough solution into the reference electrode to fill it up.
4. Once topped up, replace the band to prevent the electrode fill solution escaping.

Note: Only use supplied Reference Electrode Fill Solution.

Polishing technique for Working Electrode

To carry out analysis, the Working Electrode must first be polished to give a smooth surface to allow for plating. 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.

The instrument also consists of:

Stirrer – the stirrer will not operate all the time during the different stages of a run. The stirrer is crucial to the operation of the instrument and needs to be checked to ensure it is working properly. During a run, look through the translucent beaker or lightly touch the bottom of the SAB to determine that the stirrer is rotating when indicated on the handset. The stirrer has been designed to minimise turbulence thus ensuring increased repeatability. If the stirrer fails to rotate, turn the sonde head upside down, add a few drops of the **Stirrer Oil (do not use any other oil)** down the stirrer shaft at the base near the sonde head (near where it enters the sonde) and rotate gently to ensure the lubricant coats the bottom of the shaft.

Sample Analysis Beaker (SAB) – the SAB has a fixed volume so that when full and removed from the water course, excess sample will empty out of the holes levelling off at a constant volume.

Instrument – the handset is waterproof and robust. A periodic visual inspection to ensure it is not damaged and that the screen is readable will suffice.

Buffers – the buffers are powders, which should be stored in a dark, cool, dry environment. If stored correctly, these have a shelf life of 3 years. Refer to individual sachets for expiry dates.

Standards – the standards should be stored in a dark, cool place and have a shelf life of 12 months. Refer to standards for expiry dates. Should any contaminant come into contact with the standards, they should be discarded and replaced.

Plating Solutions – the plating solutions should be stored in a cool, dark place and have a shelf life of one year. Refer to solutions for expiry dates. Should the efficacy of the plating solution deteriorate before this during use, they should be discarded and replaced.

Menu System

The Metalyser® is controlled via a system of on-screen menus. The structure of these menus is illustrated below to assist you in navigating them.

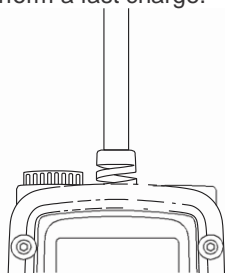
Main Menu

Test Methods	→	Condition/Mix	Used to condition the working electrode ready for use or mix samples when using digester.
	→	Analyse Sample	Used with Calibrate function. Calibration must be undertaken before analyse sample can be used
	→	Standard Addition	Used to analyse samples and calculate the unknown concentration by the addition of a known standard
	→	Calibrate	Used with analyse sample. Calibration must be performed before analyse sample is used
	→	Blank subtraction	Used to set or replace a baseline if contaminants are suspected in the buffer.
Data Log	→	View Log	Data Log viewing options
	→	Last Result	Displays latest result and graph
System	→	Backlight	Backlight control options
	→	Set date/time	Used to set the instrument date and time
	→	Language	Used to select the display language
	→	Charge	Battery charging options
	→	Auto switch off	Auto off select
	→	Program P/STAT	Program internal hardware (Not used in normal operation)

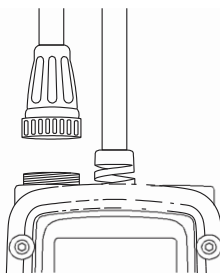
3.2. GETTING STARTED

Charging the battery

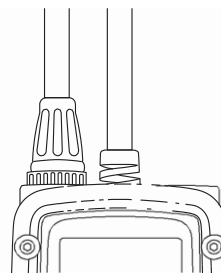
When you first receive your Metalysers® it would be advisable to fully charge the battery. This can be done several ways using the USB lead and chargers provided with the kit. Connect the device as shown below, then connect to the chosen charging system (mains/12V). If a computer is used, the software drivers will need to be installed to perform a fast charge.



Unscrew the blue cap



Insert mini USB lead



Tighten the screw cap

The Metalysers® is capable of charging at a fast rate taking approximately 7 hours to fully charge the battery, or a trickle charge rate which should be used for overnight top up charge if the instrument is only used once or twice a day.

From the system menu select Charge and you will be given three options:

100mA	Trickle charge rate
500mA	Fast charge rate
AUTO	The instrument will automatically select the best charge rate.

To select the charge rate follow these steps:

- From the main menu select **System**
- From the system menu select **Charge**
- Highlight required charge rate and press **<OK>**
- Press **<cancel>** to return to main menu

Note: The fast charge option is only available when the instrument is switched on. The instrument will also charge at a trickle rate if the instrument is connected for charging but not switched on

Driver Installation

Your Metalyser® 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 Software USB stick provided for all versions of Windows post Windows 95.

- Insert the USB memory stick into an available USB port on your PC
- Connect the USB cable supplied to the Metalyser® instrument as described for battery charging. Connect the other end to an available USB port on your PC
- 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', navigate to USB memory 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 and your Metalyser® should be installed
- To verify correct installation select fast charge on the instrument and check that the fast charge icon (see page 7) is displayed on the Metalyser®'s screen

Setting the Date and Time

The Metalyser® contains a calendar and clock which is used to date stamp the results log. The clock may be set to UTC when you receive the instrument so may need re-setting. It will also need re-setting if the battery is disconnected. To set the clock follow these steps.

- From the main menu select **System**
- From the system menu select **Set date/time** and press <OK>
- Use the navigation key to alter the date and time
- Press <OK> when finished to save settings

Backlight

Your Metalyser® is equipped with a backlight to aid viewing in poor lighting conditions. Selecting backlight from the system menu will present the following options:

ON	The backlight will be on at all times ¹
OFF	The backlight will be off at all times
AUTO	The backlight will switch on when a key is pressed or the screen refreshes. It will turn off after five seconds of inactivity.

¹ The battery life will be shortened in this mode.

To change the backlight settings follow the steps below.

- From the main menu select **System**
- From the system menu select **Backlight**
- Highlight required setting and press **<OK>**
- Press **<cancel>** to return to main menu

Language Selection

The Metalyser® comes with English, French and Spanish languages pre-installed.

To Change the language follow the steps below:

- From the main menu select **System**
- From the system menu select **Language**
- Highlight required language; **English, French, Spanish** and press **<OK>**
- Press **<cancel>** to return to main menu

Auto switch off

The Metalyser® can be set to automatically switch off after ten minutes of inactivity.

This is recommended to extend battery life. To change this setting select 'Auto Switch Off' from the System menu and then select On or Off.

- From the main menu select **System**
- From the system menu select **Auto switch off**
- Highlight required setting and press **<OK>**
- Press **<cancel>** to return to main menu

3.3. OPERATING PROCEDURE

3.3.1. METHODS

The Metalysers® operating procedure consists of two main steps – plating (conditioning) and analysis. The plating step forms a plate on the surface of the working electrode which can be seen as either a grey or yellow/gold layer on the tip of the Working Electrode. The quality of this plate is essential to achieving reliable results.

The following table illustrates which electrode to use for each element.

Elements analysed	Working Electrode used	Colour of Working Electrode after Plating
Cd, Pb	WE1	Grey
Hg	WE2	Yellow/Gold
As	WE2	Yellow/Gold
Cu	WE1	Grey
Zn	WE1	Grey

If WE1 is to be used in the analysis, then WE2 is not part of the circuit and has no role in the analysis. Likewise for WE1 during analyses using WE2.

When switching between different analyses, the unused electrode needs to be removed, otherwise results may be affected.

Procedures for each parameter are detailed in the relevant application notes

3.3.2. PLATING

This is a process used to form a very thin plate on the surface of the Working Electrode and only takes a few minutes to complete. The plating step is necessary prior to the analysis of each element, however it is possible to analyse elements consecutively if they share a common plating solution (e.g. Cd, Pb and Zn all use HG500 Hg Plating Solution). Before carrying out the plating step, the Working Electrode surface must be polished to remove any scratches and imperfections (see page 12).

There are two preliminary steps required for any analysis with the Metalyser®. The plating step uses the HG500 Hg Plating Solution, HG1000 Thick Hg Plating Solution or AU500 Au Plating Solution, added to the SAB, to form a plate on the Working Electrode. After plating, the mirror-finish black tip is covered with a plate that is either grey (Hg Plating Solutions) or yellow/gold (Au Plating Solutions).

The element being tested determines the plating solution required:

HG500 Hg Plating Solution – Cd, Pb, Cu, Zn

AU500 Au Plating Solution – Hg and As

WE1 is used for Cd, Pb, Cu and Zn analysis

We2 is used for Hg and As analysis

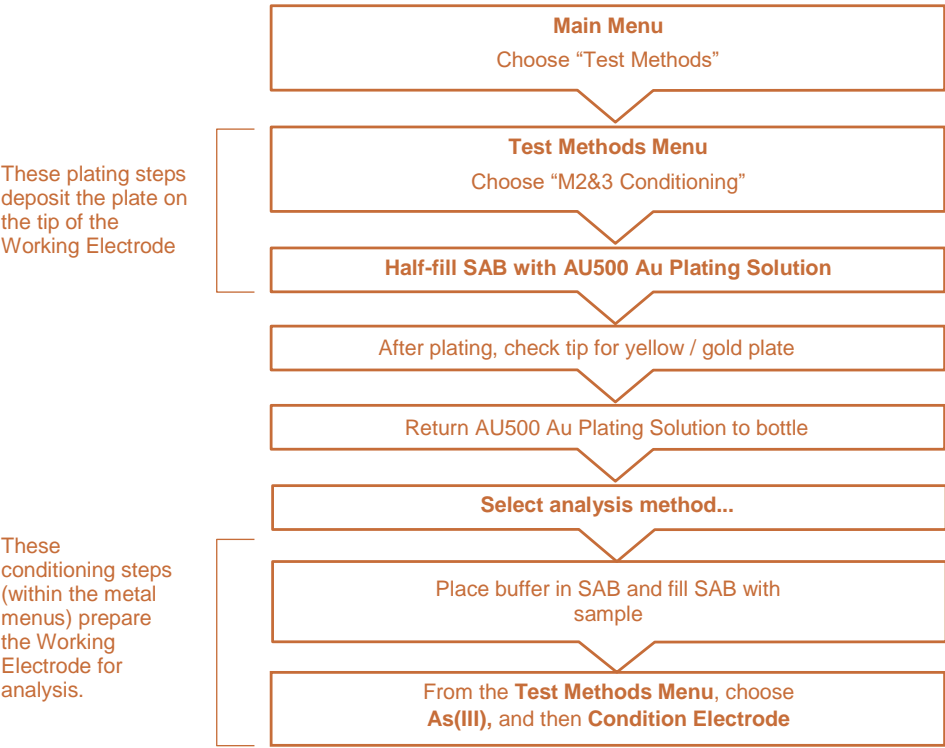
It is necessary to perform the plating and conditioning before an analysis session or if the sensitivity of the analysis decrease. This is because, over time, the plate will reduce in thickness and eventually come off. Typically this will be after 20+ consecutive samples or a period of 2-3 hours. It is therefore suggested to regularly re-plate the electrode; if analysing a lot of samples, it is recommended to plate in the morning and again at the beginning of the afternoon session.

If in doubt, remove the old plate by wiping the surface, polishing the tip of the Working electrode to a mirror-finish (with the polishing kit provided), then rinse with the wash bottle and perform a new plating.

When analysing for elements requiring a plate different to the one currently being used, the electrode not in use will need to be removed from the sonde to prevent interference. It is advised to group together analyses using the same plate to save switching between them.

The conditioning of the electrode occurs when the element of interest is selected in the **Test Methods** menu and then choosing condition electrode, which is at the top of the list for the selected electrode. The conditioning step should be carried out in sample water, and undertakes a run which sensitises the Working Electrode.

Example: Overview of As (III) procedure to first plate then sensitise the electrode:



Refer to Application notes for Operating Procedures particular to each parameter.

3.3.3. ANALYSIS

Before performing a test using the Metalyser®, ensure that the working electrode has been successfully plated and conditioned first for the metal of interest.

(See Section 3.3.2 Plating and relevant application notes).

There are now two ways to analyse a sample, either via the Standard Addition method or by an in-field calibration of the instrument.

1. The preferred method of analysis for the Metalyser® is for the user to add a known amount of standard to the solution giving a known peak height, the **Standard Addition** method. The instrument then determines the value for the unknown sample previously analysed.
2. The in-field calibration method establishes a calibration curve first before analysing a number of samples rapidly. The Metalyser® firstly needs to establish the calibration curve using the Calibrate option in the menu of each metal of interest. Once this has been successfully completed an **Analyse Sample** can be undertaken

(Note: using **Analyse Sample** without establishing a new calibration curve, the previous calibration will be used which may not be correct for the current analysis).

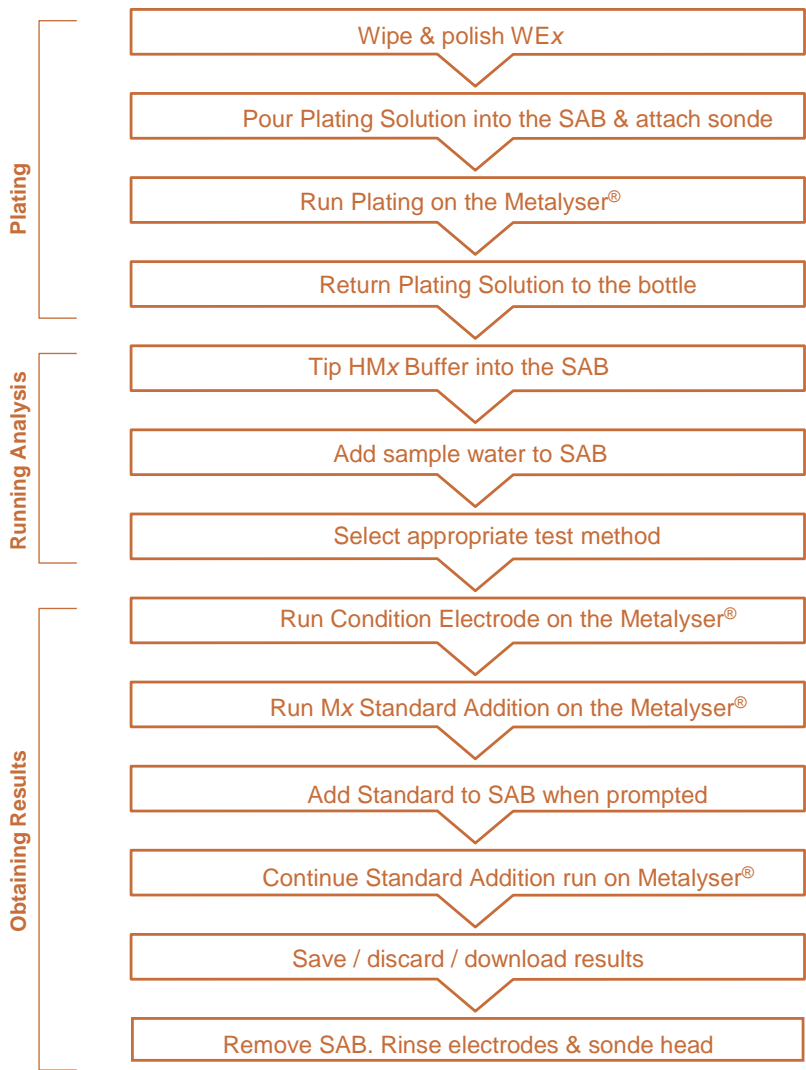
The calibration needs to be established just prior to analysis and also after a new plate has been applied to the tip of the working electrode. Many factors affect the calibration curve and the greater the time the analysis occurs after the calibration curve was established the greater the chance that there will be a decrease in accuracy. This is due to a number of factors including variability with the plate over time and even the components of the solution changing. It is important when calibrating the instrument that it is calibrated on a similar type of water to that being analysed, i.e. if a river sample from a particular river is being analysed then the Metalyser® needs to be calibrated using that river water, if a new river is to be analysed then recalibrate using a new river sample from the new location.

The **Blank Subtraction** method is used for creating a new baseline for the instrument for the analysis. It is to be used only when a suspected contaminant is present in the buffers being used. Performing this will replace the previous blank.

After each analysis, the sonde head and the electrodes are to be washed thoroughly. This is in order to minimise carry-over and to clean off all traces of the previous analysis. This can be achieved by using the deionised water or by washing the sonde head (without the SAB) in the sample water. This is particularly important following an arsenic analysis.

3.3.4. STANDARD ADDITION ANALYSES - OVERVIEW

Performing a Standard Addition method analysis consists of Plating, running Analysis and obtaining a result. The following main steps will assist you in understanding how to perform a Standard Addition based test. More extensive information on performing analyses can be found in the application notes.



Where 'WEx', use WE1 or WE2 for your required analysis
Where 'Mx' use M1, 2, 3, 4 or 5 for your required analysis

3.3.5. ADDING A STANDARD ADDITION

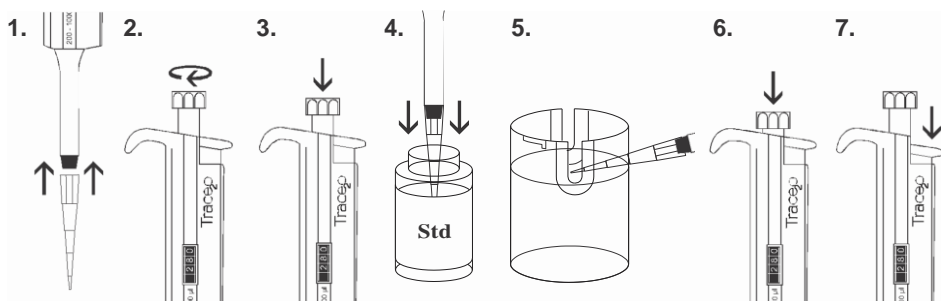
When prompted, a 20mg/kg Standard Addition needs to be added to the SAB. This is achieved by using the pipette (which is set to 280µl). The 20mg/kg default setting can be changed by pushing the joystick left and right. Each 140µl increase on the pipette is equivalent to 10mg/kg.

On the handheld, when prompted to add 20mg/kg of a particular element(s) then the highlighted value of 20mg/kg can be altered by pushing the joystick to the left and to the right. If the user wants to measure higher levels then a higher value closer to the concentration level will be required. The accuracy will increase the closer the Standard Addition is to the actual concentration. For example, an unknown sample is expected to be around 15mg/kg then a 20mg/kg standard in this case is sufficient. If the user expects the analysis to be 100mg/kg then, for example, a 120mg/kg standard would be suitable.

How to use a pipette

1. Place a clean pipette tip on the pipette
2. Select the volume required by twisting the dial at the top of the pipette
3. Hold the pipette with the thumb uppermost and press the button down gently to the first stop on the top plunger
4. Place the pipette tip in the standard bottle just underneath the top of the liquid and slowly release the plunger
5. Move the pipette to the SAB and locate the pipette tip through the hole in the SAB
6. Push the plunger all the way down to the second stop and hold then slowly withdraw the pipette tip from the SAB
7. To release the pipette tip after use, push the white button at the side of the pipette

Before carrying out testing, become familiar using the pipette and the two different stops on the plunger to ensure the correct volume of standard is added.



Altering deposition times

The standard default deposition time of 60 seconds is suitable for all analysis down to 5mg/kg for most elements, 10mg/kg for arsenic. To increase accuracy for the lower levels an increased deposition time is recommended. If the user expects the analysis to be higher, then a shorter deposition time should be used. This can be adjusted when prompted to choose a deposition time at the beginning of the test methods. 30, 60 or 120 seconds can be selected by moving the joystick to the right and to the left on this screen and then pushing the centre of the joystick to accept it.

Deposition time	Concentration range
30 seconds	50mg/kg to 500mg/kg
60 seconds	10mg/kg to 50mg/kg
120 seconds	<10mg/kg

Suggested ranges for choosing deposition times

The instrument has been designed to have a maximum reading of 500mg/kg. Should the user wish to measure higher than this they will need to dilute the sample. For example, if the user wishes to measure 600mg/kg dilute the sample by half and then multiply the result by 2. Always dilute with water that will not contribute to the heavy metals being tested. Deionised water is ideal.

Blank subtraction

Blank subtraction is recommended to be used for all of the analyses and is the default setting. It is particularly important for the analysis of Hg and As.

3.4. TROUBLESHOOTING

Whilst your Metyliser® is designed to be very reliable, problems may occur throughout its working life. The following tables are intended to help you diagnose and resolve these problems simply and quickly. Should you not be able to resolve the problem please contact your supplier and they will be able to assist you.

When troubleshooting your Metyliser® the following checks should be undertaken in this order:

1. Reference Electrode – does the Reference Electrode contain the right level of electrode fill solution? Is it damaged at all? Are there air bubbles in the inner tube?
2. Stirrer – does the stirrer rotate? Does it mix the powder reagents when operating?
 - a. To check this run the stirrer according to the instructions on page 13
2. Working Electrode – is the Working Electrode plated correctly?
 - a. Is WE1 grey in colour or is WE2 yellow in colour? Has the grey WE1 Hg Plate been wiped off before the WE2 plating?
3. Solutions – could the solutions have been contaminated? Have the correct buffers been added in the correct order? Has the right amount of standard been added?

The Metyliser® will try and help you with determining the problems. See below.

Displayed Error	Probable Cause
> L.O.D	The metal concentration is above the maximum limit of 500mg/kg. Dilution should be used.
< L.O.D	The metal concentration is below the minimum limit of detection for the method being used.
Addition error	The increase in metal response was not sufficient enough to determine the addition.
Calibration error	The addition was not added or the metals have not been detected.

Problem	Possible Cause	Solution
Peak not visible	Conditioning step not completed	Wipe end of Working Electrode, polish and re-plate as per procedure
	Plate Damaged	Re-plating required
	No heavy metals in solution	Test with known amount of standard solution
Results lower than expected	Stirrer not rotating	Ensure the stirrer is rotating by looking into the SAB or lightly touching the bottom to determine it is working; if the stirrer is stuck, use stirrer oil as described on page 13
	Physical mask over the electrode	Ensure no debris – for example, leaves – are physically masking the electrode surface, preventing the analysis from occurring
Results increasing with each run	Organics present	Instrument is not designed to deal with strong organics; system is for natural rivers and water courses
Sample > LOD	Overrange on instrument	The Metalyser has been designed for low level mg/kg analysis in natural water and soils. The instrument will measure high limits by: a) choosing a shorter deposition time (i.e: change default from 60 to 30 seconds when prompted in the Set Deposition Time menu during the course of the run) b) diluting the sample by a known amount and multiplying the result back to the original solution
Metalyser not responding to key press	Internal fault	First turn off the Metalyser, wait for a few seconds and then turn it back on. If this does not fix the problem, disconnect the battery and reconnect. If the fault persists, contact the supplier to organise a return.
	Connected to Metaware	Disconnect Metalyser from Metaware, turn off the Metalyser and then turn it back on after a few seconds. This should give control back to the Metalyser.

Note: Periodically, it is good practise to check the response of the instrument and hence the quality of the plate during the course of the day by looking at the graph of the last result and looking for peaks. The information on this graph is very informative and tells the operator what is happening with the results.

To see the graph, navigate to the main menu, choose **Data Log**, then **Last Result**. The result will appear first; pressing **<OK>** will display the graph.

Press **<OK>** again to exit the graph.

3.5. INTERFERENCE EFFECTS

The Metalys[®] has been designed to test very low levels of metals in soils and as such is very sensitive. Due to the interaction of other metals and organics in the sample, interferences can occur as with any system of this type.

3.6. SPECIFICATIONS

Metalys[®] HM4000 Handheld Unit

Input Power: 4.5–5.5 V DC, 250mA.

Power Connector Type: mini USB Type B (Bulgin micro series).

Li-Ion Internal Battery

Output voltage: 3.3V

Output current: 2200mAh

	Parameter	Lower Limit*	Upper Limit*
Arsenic (III)	As (III)	5	500
Copper	Cu	5	500
Mercury	Hg	5	500
Cadmium	Cd	3	500
Lead	Pb	5	500
Zinc	Zn	5	500

*Upper limit fixed. If the user requires values higher than this then a dilution will be needed.

*Lower limits achievable using the Handset at 60 seconds deposition.

Increased accuracy can be achieved by setting the deposition time to 120 seconds

Variability at 20mg/kg \pm 5% with Metaware software, \pm 7.5% with handheld

Accuracy dependent on element measured, sample matrix and type

4. SOIL EXTRACTION KIT

4.1 GETTING TO KNOW YOUR SOILS KIT

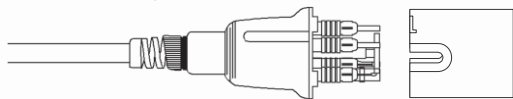
The Soil extraction kit contains all the equipment and accessories required to carry out a digestion/extraction procedure for 5 metals, Cd, Pb, Cu, As(Total) and Hg. These require three digestants, labelled as D1, D2 and D3.

The digestants are supplied in recyclable 4ml bottles and contain the correct amount so no measurement is required.

Each set of digestant is sufficient to provide enough extract to perform at least 3 tests. One extract can be used for the analysis of Cd, Pb, Cu, As & Hg.

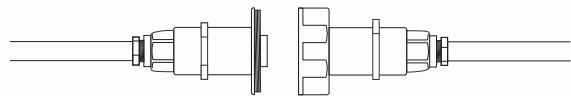
To carry out an extraction procedure you will require the mixer. The mixer is very similar to the sonde and is connected to the hand held instrument in the same way as follows:

The Mixer Assembly



The Mixer

SAB (Sample Analysis Beaker)



In-line waterproof cable connector

Also contained within the box is a portable balance which is used to weigh the soil samples. The balance can be used at angles up to 15° allowing it to be used in the Peli case on uneven ground but the box should be stable and the balance zeroed once in position and kept in that position during use.

4.2 SAFETY FIRST

The extraction sachets (D1, D2 and D3) contain concentrated strong acids and oxidising agents, which are highly corrosive and harmful to eyes and skin. Therefore, extra care should be taken whilst handling these chemicals.

Safety glasses and gloves are provided as part of the soils extraction kit. These should be worn at all times whilst carrying out the digestion procedure. Protective clothing should also be worn (Not provided).



All containers and equipment should be disposed of in accordance with local legislation.

4.3 DIGESTION PROCEDURE FOR DRY SOILS

1. Take an excess amount of dry soil sample using the scoop provided.
2. Use the provided pestle and mortar to crush and soften the soil clumps and mix it up until a uniform powdery soil sample is obtained.
3. Place the empty graduated measuring beaker on the balance provided and press Tare or Zero on the balance, and wait until it displays “0.00 g”.
4. Use the spatula to gently add the crushed soil into the digestion beaker, until it displays “1.00 g”
5. Add approx. 30mL of de-ionised water to the graduated beaker.
6. Add D1, D2 and D3 to the graduated beaker and then fill up with de-ionised water to the 50ml mark.
7. Pour the full contents of the graduated beaker into the digestion beaker and swirl to mix.
8. Decant from beaker to beaker to ensure full transfer of soil
9. Connect the digestion beaker to the mixer.
10. Choose the “Digest” method from the list of methods in the Metalyser®. Select condition and allow the stirrer to run for 10 minutes.
11. After 10 mins, remove the beaker from the stirrer head and let it settle for 5 minutes.
12. Connect a syringe filter to a 5mL syringe. Attach a short length of tubing to the filter and use the syringe for taking 3.5ml of the digested sample.
13. Add the 3.5 mL sample to the sample analysis beaker and fill to the mark just below the overflow with de-ionised water.
14. The soil extract sample is now ready to be analysed using the Metalyser®.

4.4 DIGESTION PROCEDURE FOR WET SOILS (QUICK)

1. Take an excess amount of wet soil sample using the scoop provided.
2. Remove any big pieces of stone or wood out of your soil sample.
3. Add de-ionised water to the wet/moist soil sample dropwise, until it is saturated with water.
4. Use the pestle and mortar to mix up the soil until a uniform wet soil sample is obtained.
5. Place the empty graduated beaker on the balance provided and press Tare or Zero on the balance, and wait until it displays “0.00 g”.
6. Use the spatula to gently add the appropriate weight of wet soil into the graduated beaker (according to the table below), until it shows the correct weight of the soil sample.

Description	Amount of we soil to give 1g dry soil (g)
Soil with no wood and organic content	1.9
Soil mixed with wood and organic content	1.6
Sandy and stony soil	1.2
Light soil and compost	7.5

7. Add approx. 30mL of de-ionised water to the graduated beaker.
8. Add D1, D2 and D3 to the graduated beaker and then fill up with de-ionised water to the 50ml mark.
9. Pour the full contents of the graduated beaker into the digestion beaker and swirl to mix.
10. Decant from beaker to beaker to ensure full transfer of soil
11. Connect the digestion beaker to the mixer.

12. Choose the “Digest” method from the list of methods in the Metalyser®. Select condition and allow the stirrer to run for 10 minutes.
13. After 10 mins, remove the beaker from the stirrer head and let it settle for 5 minutes.
14. Connect a syringe filter to a 5mL syringe. Attach a short length of tubing to the filter and use the syringe for taking 3.5ml of the digested sample.
15. Add the 3.5 mL sample to the sample analysis beaker and fill to the mark just below the overflow with de-ionised water.
16. The soil extract sample is now ready to be analysed using the Metalyser®.

4.5 DIGESTION PROCEDURE FOR WET SOILS (PRECISE)

1. Take an excess amount of wet/moist soil sample using the scoop provided.
2. Remove any big pieces of stone or wood out of your soil sample.
3. Use the pestle and mortar to mix up the soil until a uniform wet soil sample is obtained.
4. Transfer an excess amount the wet or moist (ca. 10-20g) soil in a small oven dish and leave it in an oven at temperature of 100°C for 1 hour.
5. After 1 hour, take the dry soil sample out and let it cool down for 10 - 15 minutes.
6. Place the empty digestion beaker on the balance provided and press Tare or Zero on the balance, and wait until it displays "0.00 g".
7. Use the spatula to gently add the dried soil into the digestion beaker, until it shows the weight of "1.00 g" of the soil sample.
8. Add approx. 30mL of de-ionised water to the graduated beaker.
9. Add D1, D2 and D3 to the graduated beaker and then fill up with de-ionised water to the 50ml mark.
10. Pour the full contents of the graduated beaker into the digestion beaker and swirl to mix.
11. Decant from beaker to beaker to ensure full transfer of soil
12. Connect the digestion beaker to the mixer.
13. Choose the "Digest" method from the list of methods in the Metalys[®]er, select 'Condition' and let it stir for 10 minutes.
14. After 10 mins, remove the beaker from the stirrer head and let it settle for 5 minutes.

15. Connect a syringe filter to a 5mL syringe. Attach a short length of tubing to the filter and use the syringe for taking 3.5ml of the digested sample.
16. Add the 3.5 mL sample to the sample analysis beaker and fill to the mark just below the overflow with de-ionised water.
17. The soil extract sample is now ready to be analysed using the Metalysers®.

NOTES



The Technology Centre
Wagtech Court
Station Road
Thatcham
Berkshire RG19 4HZ
United Kingdom

T : +44(0) 1635 866772

Fax : +44(0) 1635 873509

E : sales@trace2o.com

W: www.trace2o.com

