HM3000

APPLICATION NOTE T2O-AN-303

ARSENIC (III + V) METHOD

The following application note explains the procedure for the detection of Arsenic (III) and Total Arsenic using the HM3000 Metalyser®. Total Arsenic can be measured by the addition of hydrochloric acid which is not supplied as standard.
PLEASE READ THIS APPLICATION NOTE CAREFULLY. TRACE2O® HAS ALTERED THE NAMES OF SOME REAGENTS FOR SIMPLICITY AND SO THE PROCEDURE MAY BE DIFFERENT FROM THAT FOLLOWED PREVIOUSLY.

Equipment:

- **HM3000 Kit**
- **HM3 Buffer** (Previously M3 Buffer sachets)
- **AS50 As standard** (Previously M3 Standard (5ppm))
- **AU500 Au Plating Solution** (Previously M2&3 Conditioning Solution)
- **AS1 buffer** (Only required for total As – Supplied separately)
- **AS2 preparation solution** (Only required for total As – Supplied separately)

Electrode conditioning:

- Polish WE2 Electrode to a mirror finish and check using a magnifier that no scratches or scuffs are present.
- Half-fill the sample analysis beaker (SAB) with **AU500 Au Plating Solution** and fit to the Sonde.
- Select 'M2&3 Conditioning' from the 'Measurements available menu' then click 'Condition Electrode' on the Analysis panel.
- The M2&3 conditioning step will take approximately 5 minutes.
- Once completed, return the **AU500 Au Plating Solution** to the **AU500 Au Plating Solution** bottle and rinse the Sonde and SAB with the Electrode Rinse Solution and/or deionised water.

Analysis procedure for Arsenic (III)
Sample preparation:

- Add **one sachet of HM3 Buffer** to the SAB.
- Add 70ml of sample water to the SAB, either by adding a measured volume or by fitting the SAB to the Sonde and submerging in the water source until the bubbles stop.

There are three methods for carrying out the analysis. The single-point standard addition is the fastest option, using two data points to calculate concentration. This is also less accurate. The multi-point standard addition uses four data points to calculate concentration, and allows repeat scans for stabilisation purposes. This method takes longer and uses more chemicals. The calibration option is

designed for rapid analysis of several samples with a similar matrix (i.e. several samples from different points along the same river bank).

Analysis (single-point standard addition method)

- Fit the SAB to the Sonde (If not using submersion method).
- Select 'As(III)' from the 'Measurements Available' drop down menu, then click 'Condition Electrode' from the analysis panel and wait approximately two minutes for it to complete.
- Click 'Standard Addition' in the analysis panel. Wait approximately two minutes until prompted to add 20ppb of the standard. When prompted use the pipette to add 280µL of the **AS50 As standard** into the SAB and click 'OK'. The analysis will continue to run for approximately two further minutes after which the results will be displayed.

Result (single-point standard addition method)

- The result(s) are shown in the Analysis window until 'Ok' is clicked. Following this the results are displayed as a graph on the graph tab and the ppb results will be automatically entered into the results log. There will two graphs displayed, the first is the scan of the original sample concentration and the second is after the standard addition. If As (III) has been detected the peak will be identified as As (III) and will be marked up as such. The original sample concentration will be reported in ppb.

Analysis (multi-point standard addition method)

- Fit the SAB to the Sonde (If not using submersion method).
- Select 'As (III)' from the 'Measurements Available' drop down menu, then click 'Condition Electrode' from the analysis panel and wait approximately two minutes for it to complete.
- On the Control panel, ensure that Deposition Time is set to 60 seconds. Ensure that 'Background subtraction' is checked.
- Run the stirrer as required to dissolve the buffer into the sample water.
- Click 'Start Analysis' and wait for approximately 2 minutes for the graph to be displayed. Repeat until stable. Note that the reading displayed in ppb should be ignored for this method.
- Using the micropipettes, perform a standard addition from the **AS50 As standard** bottle. Use an appropriate addition volume for the sample volume and desired concentration. For better accuracy, a standard addition should be as close as possible to the expected sample concentration. For example, if 10ppb As is expected in the sample, the standard addition should add a further 10ppb.
- Reminder – to calculate addition volume, use the following formula:

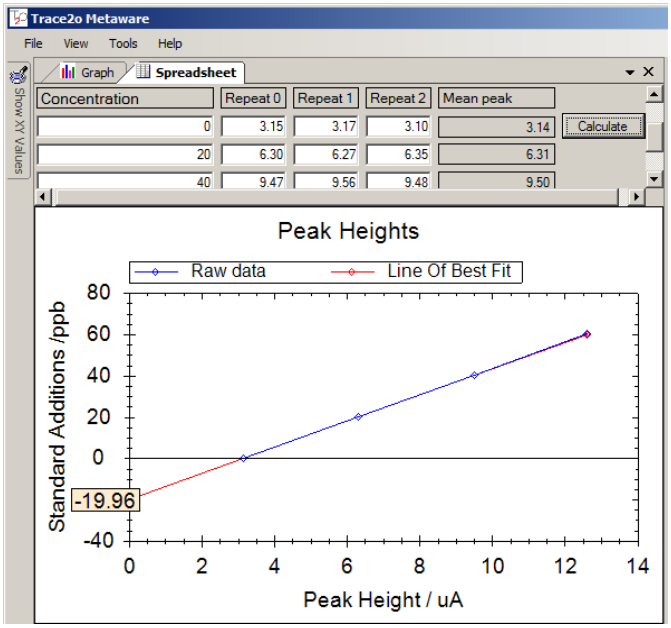
  $$\text{Standard addition vol. (mL)} = \frac{\text{Sample vol. (mL)}}{\text{Standard conc. (ppb)}} \times \text{Addition conc. (ppb)}$$

  For example, with a sample volume of 70mL, standard concentration of 5ppm (or 5000ppb) and a desired addition concentration of 10ppb, the standard addition volume would be:

  $$\frac{70}{5000} \times 10 = 0.14\text{mL} = 140\mu\text{L}$$
- Click 'Start Analysis' and wait for approximately 2 minutes for the graph to be displayed. Repeat if required.
- Repeat the standard addition/Start Analysis procedure a further two times.

Result (multi-point standard addition method)

- To calculate the peak height, first position the stylus over the data-point you wish to use as the right hand side of the peak, then tap-and-hold the stylus and click ‘calculate peak’. If a valid point is not found this function will be greyed out.
- Move the stylus to the left hand side of the peak and tap once. The baseline will be drawn in and the peak height shown, reported as a current (µA).
- To calculate the concentration, input the peak heights into the spreadsheet tab.



The table has four possible entries for the sample concentration. The first entry would normally be zero, 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 standard addition. In the above example three standard additions are performed each of 20ppb. The peak heights are calculated for each of the additions and entered in the table. To achieve greater accuracy, repeat analysis can be run after each addition to give an average over three readings although it is not necessary to fill the table. When all the data is entered, click **Calculate**. The mean peak heights will be calculated and a graph of concentration vs peak height plotted. 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. The value is shown as a negative because this indicates the amount which needs to be added to each of the data points to make the line of best fit go through the origin which in this case is 19.96ppb.

Analysis (Calibration Method)

- Fit the SAB to the Sonde.
- Select ‘As (III)’ from the ‘Measurements Available’ drop down menu, then click ‘Condition Electrode’ from the analysis panel and wait approximately two minutes for it to complete.
- Click ‘Calibration’ in the analysis panel. Wait approximately two minutes until prompted to add 20ppb of the standard. When prompted use the pipette to add 280µL of the standard into the SAB and click ‘OK’. The analysis will continue to run for approximately two further

minutes until prompted to add another 20ppb of standard. Repeat the addition process and click ‘ok’. The analysis will run for another two minutes before completing the calibration.

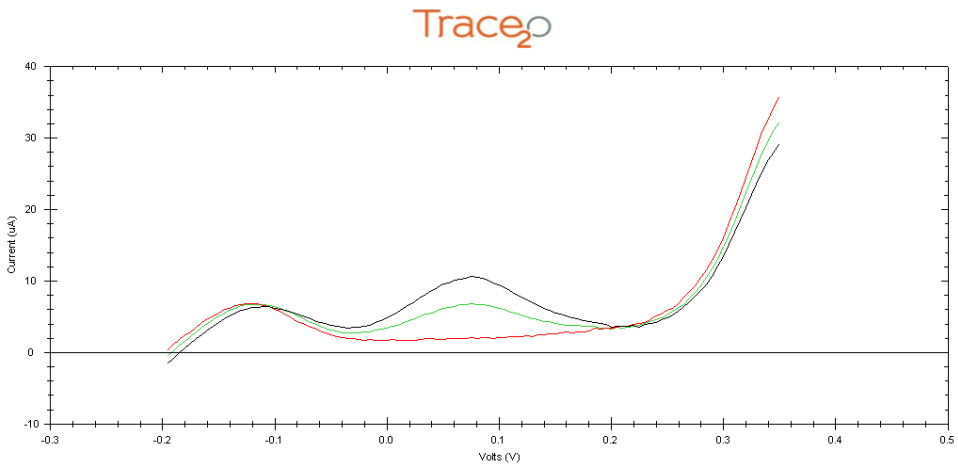
- Once the calibration has been completed several consecutive analyses can be carried out.
- Click ‘Analyse Sample’ on the analysis panel and wait approximately two minutes for the analysis to complete.

Result (calibration method)

- The result(s) are shown in the Analysis window until ‘Ok’ is clicked. Following this the result(s) are displayed as a graph on the graph tab. If As (III) is present a peak will be seen in the graph which will be marked up as As and the calculated sample concentration displayed.

Graph

- Arsenic appears as a broad peak centred around 0.05 and 0.1V. Peak will appear to the left and right of the arsenic, as shown – this is as a consequence of unavoidable contaminants in the buffer. These contaminants do not affect the result in any way.



LOD

- The Lower LOD is 10ppb, upper LOD is 500ppb.
- To increase the range the sample can be diluted using Ultra-pure de-ionised water. Other water types could introduce contamination.

Analysis method for Total Arsenic

Sample preparation:

- Add 70ml of sample water to the SAB. Either by adding a measured volume or by fitting the SAB to the Sonde and submersing in the water source until the bubbles stop.
- Add 2.15g of AS1 buffer and 280uL of AS2 preparation solution to the SAB. The AS1 functions as the buffer, and AS2 as an oxidising agent in coordination with the Metalyser®. There is no need for an HM3 Buffer sachet.

Analysis Method:

- Follow the procedure for Arsenic (III), but select As (III+V) from the ‘measurements’ drop down menu in place of Arsenic(III).

Results :

- If a result for Arsenic (V) is required, Arsenic (III) analysis can be carried out, followed by Arsenic (III+V) analysis. The result for Arsenic (V) can be found by subtracting the Arsenic(III) result from the Arsenic (III+V) result.