

---

Voltammetric Analysis System

# VAS User's Guide

Issue 5



Copyright © 2008 Cogent Environmental Ltd. All rights reserved.

Cogent Environmental Ltd.  
184 Cambridge Science Park  
CAMBRIDGE CB4 0GA  
UNITED KINGDOM

Email: [info@cogentenvironmental.co.uk](mailto:info@cogentenvironmental.co.uk)  
Web address: [www.cogentenvironmental.co.uk](http://www.cogentenvironmental.co.uk)  
Telephone: +44 (0) 1223 395450  
Fax: +44 (0) 1223 395 451

P/N. 210-033-8

# Contents

<b>Introduction</b>	<b>1</b>
Scope .....	2
What Is VAS? .....	2
What's New .....	2
Contacting Cogent Environmental .....	2
Typographical Conventions .....	3
<b>Getting Started</b>	<b>9</b>
Installing VAS .....	5
Personal Computer Requirements .....	5
Installing .....	5
Running VAS .....	6
Uninstalling VAS .....	6
<b>Introduction to Voltammetry</b>	<b>7</b>
The Voltammetric Method .....	8
Detection Windows .....	9
Peak Measurement .....	9
Blanks and Artificial Blanks .....	10
The Standard Comparison Method .....	10
The Standard Addition Method .....	11
<b>Supported Analysers</b>	<b>13</b>
PDV 3000 .....	14
PDV 6000, PDV6000 <i>plus</i> .....	14
PTEA 6000 .....	14
TEA 3000V .....	15
Analyser Preparation .....	15
Setting up a PDV 3000 .....	15
Setting up a PDV 6000, PDV6000 <i>plus</i> .....	15
Setting up a PTEA 6000 .....	16
Setting up a TEA 3000V .....	16
Configuring VAS For Analyser Communications .....	16
PDV 3000, PDV 6000, PTEA 6000, or PDV6000 <i>plus</i> .....	16
TEA 3000V .....	17
VAS Analyser Commands .....	17
Initiate Run .....	18
Condition Electrode .....	18
Drain Cell .....	18
Rinse Cell .....	19
Get Data .....	19
Upload Data .....	19
Program Metal Menus .....	19

Program Conditioning Menus.....	19
Program Upgrade.....	20
Program Properties.....	20
<b>Data Management</b>	<b>21</b>
<hr/>	
The VAS Data Structure .....	22
Managing Projects .....	23
Managing Files .....	23
Using Comments .....	24
Exporting and Importing Projects.....	25
Generating Project Reports.....	26
Printing Data.....	26
<b>Collecting Voltammetric Data</b>	<b>27</b>
<hr/>	
Set the Run Configuration .....	28
PDV 3000.....	28
PDV 6000, PTEA 6000 Run Configuration.....	28
PDV6000 <i>plus</i> Run Configuration.....	28
TEA 3000V Run Configuration.....	28
Prepare Working Electrode.....	29
PDV 3000.....	29
Action a Voltammetric Run.....	29
Blank Runs .....	30
Standard Runs.....	31
Sample Runs.....	31
Standard Addition Runs.....	32
<b>Graphing Voltammetric Data</b>	<b>34</b>
<hr/>	
Graphing a Voltammogram .....	35
Zooming a Graph.....	36
Graphing Options .....	37
Graphing Multiple Voltammograms.....	37
<b>Analysing Voltammetric Data</b>	<b>39</b>
<hr/>	
Setting Detection Windows .....	40
Editing Detection Windows.....	40
Detecting Peaks and Editing Baseline .....	42
Standard Comparison Analysis.....	43
Standard Addition Analysis.....	44
Calculating Artificial Blanks .....	44
<b>Glossary of Terms</b>	<b>45</b>
<hr/>	
<b>Index</b>	<b>47</b>

---

# Table of Figures

Figure 1: Data Curve Voltammogram for Cadmium, Lead and Copper.....	8
Figure 2: Peak Size Calculation By Peak Height and Peak Area.....	9
Figure 3: Blank Subtraction.....	10
Figure 4: Standard Comparison.....	11
Figure 5: Standard Addition.....	12
Figure 6: VAS Fruit Juice Project.....	22
Figure 7: Use Comments To Identify VAS Data.....	25
Figure 8: VAS Graph Window.....	35
Figure 9: VAS Graph Window Displaying Multiple Curves.....	38
Figure 10: Highest Point Of Peak For All Curves Must Be Under Detection Window.....	40
Figure 11: Graph Detection Window Areas.....	41
Figure 12: Detection Windows dialog.....	42
Figure 13: Good Peak Sample.....	43

---

## CHAPTER 1

# Introduction

This chapter introduces you to your VAS software.

It tells you what VAS is about, what's new in the current release together with contact details should you require any assistance with your VAS software.

### In This Chapter

Scope .....	7
What Is VAS?.....	7
What's New.....	7
Cogent Environmental.....	7
Typographical Conventions.....	8

---

## Scope

This manual applies to VAS software V4 or later.

---

## What Is VAS?

VAS is a 32-bit Windows application designed for use with Cogent Environmental's family of voltammetric analysers. This tool, running on a personal computer (PC), brings data management, visual display and manipulation of information and advanced analysis techniques to your voltammetric system.

---

## What's New

For existing users of VAS 3, notable changes in VAS 4.0 version are:

- Support is provided for Cogent Environmental's newest handheld voltammetric analyser the PDV6000 *plus*.
  - Linear, Differential Pulse, and Square Wave type voltametric sweeps are supported for the PDV6000 *plus* and TEA 3000V instruments.
  - Cathodic voltametric sweeping is provided for the PDV6000 *plus* and TEA 3000V instruments.
  - Graphing and calculation actions support cathodic sweeps data.
  - VAS is no longer distributed on floppy disk. Install from the resource CD.
  - The project wizard is no longer provided. Standard applications are now loaded directly as other projects. Standard application projects are available from MTI and are provided on the VAS resource CD with the associated application notes.
  - Provisionally, more than one instrument can be controlled at one time.
  - Many subtle changes have been made to the dialogs and controls to simplify workflow. The intention is to extend the flexibility and options in setup without presenting unnecessary details to casual users. Options and settings where only occasionally important have been moved to the bottom of a dialog box, or hidden behind an 'advanced' option.
- 

## Contacting Cogent Environmental

Your local agent will be familiar with setting up and using this software. If you require assistance please first contact your local agent.

Cogent Environmental also provides customer support and service via the internet. For the latest details of all support and upgrades, FAQs, etc. please visit our website at [www.cogentenvironmental.co.uk](http://www.cogentenvironmental.co.uk).

We hope you enjoy using VAS and find it useful in your voltammetry work.

Cogent Environmental Ltd.  
184 Cambridge Science Park  
CAMBRIDGE CB4 0GA  
UNITED KINGDOM

Email: [mti@cogentenvironmental.co.uk](mailto:mti@cogentenvironmental.co.uk)  
Web address: [www.cogentenvironmental.co.uk](http://www.cogentenvironmental.co.uk)  
Telephone: +44 (0) 1223 395450  
Fax: +44 (0) 1223 395 451

---

## Typographical Conventions

The following kinds of formatting in the text identify special information.

<u>Formatting convention</u>	<u>Type of Information</u>
<i>Emphasis</i>	Used to emphasise the importance of a point.
<b>Special Bold</b>	Items you must select, such as menu options, command buttons, or items in a list.
Triangular Bullet(➤)	Step-by-step procedures. You can follow these instructions to complete a specific task.
<u>Note:</u>	A tip or an item of note.

For information on specialised terms used in the documentation, see the Glossary at the end of this document.

## CHAPTER 2

# Getting Started

This chapter gets you up and running.

It tells you how to install and run the VAS software on your PC.

### In This Chapter

Installing VAS .....	10
Running VAS .....	11
Uninstalling VAS .....	11

---

# Installing VAS

VAS can be installed on a PC running Windows 98, ME, NT, 2000 or XP.

Please follow the installation instructions carefully when installing VAS. This will ensure a trouble-free installation enabling you to begin using VAS as quickly as possible.

## Personal Computer Requirements

### Minimum System Requirements

- Windows 98SE, ME, NT, 2000 or XP with Microsoft's minimum recommended processor speed and memory for the operating system installed. (VAS is expected and is reported to run on the Vista operating system also).
- Visual display of at least 256 colours and 800 x 600 resolution.
- 10 megabytes of spare hard drive space available.
- CD-ROM drive
- Standard 9 pin RS232C serial port or USB to Serial adaptor and cable.

## Installing

- To install the VAS software from the CD-ROM.
  1. For Windows NT, 2000 and XP ensure you have logged on using Administrator privileges.
  2. Insert the Voltammetry Resource CD containing the VAS installation software into your PC. In most cases the CD menu program will start automatically after a brief pause, if auto start is not disabled. If it does not, select **Run** from the **Start** menu. Then in the Run dialog box, enter the following:  
D: \menu where D: is your CD ROM, then press the **Enter** key.  
The Resource CD menu will appear with various options shown.
  3. Click on the **Read me** button to open the readme.txt file to obtain important corrections and updates. Return to the CD menu.
  4. Click on the **Install VAS Software** button. The install process will start. Follow the installation instructions to install VAS. The default installation directory is: C: \VAS, but this can be changed during the setup if so desired.  
VAS installation complete.
  5. Return to the CD menu. Click the **Exit** button to close the CD menu.

Note the Resource CD contains much more than the VAS software. Please be sure to browse this CD for:

- Manuals in PDF format for printing or viewing on your computer.
- The latest software, standalone metals and conditioning menus that can be programmed into your voltammetric analyser.
- The latest application notes detailing the run parameters to be used to detect a particular trace element. Each application note includes a VAS project that you import into VAS to get you up and running with the minimum of effort.

---

## Running VAS

- To run the VAS software.
  1. Simply select VAS from the Start menu. For the default installation click **Start**, point to **Programs**, point to **VAS**, then click **VAS**.

---

## Uninstalling VAS

This uninstall will not delete your data files. VAS application components are removed only.

### Uninstalling VAS (Windows 98, ME or NT)

- To uninstall the VAS software.
  1. Click **Start**, point to **Settings**, and then click **Control Panel**.
  2. Double-click **Add/Remove Programs**.
  3. On the **Install/Uninstall** tab click **VAS** in the list of installed programs, then click the **Add/Remove** button.
  4. Click the **Yes** button in the confirmation dialog to remove VAS from your system.

### Uninstalling VAS (Windows 2000 or XP)

- To uninstall the VAS software.
  1. Click **Start**, point to **Settings**, and then click **Control Panel**.
  2. Double-click **Add/Remove Programs**.
  3. Click **VAS** in the list of currently installed programs and then click the **Change/Remove** button.
  4. Click the **Yes** button in the confirmation dialog to remove VAS from your system.

## CHAPTER 3

# Introduction to Voltammetry

This chapter explains what voltammetric analysis is and how it can be used to determine the composition of a sample.

The following sections provide detailed information on voltammetric analysis.

## In This Chapter

The Voltammetric Method .....	13
Detection Windows .....	14
Peak Measurement.....	14
Blanks and Artificial Blanks .....	15
The Standard Comparison Method.....	15
The Standard Addition Method.....	16

## The Voltammetric Method

Anodic stripping voltammetry is an electrochemical technique that can be used to determine the concentrations of certain metal ions in liquids. The method involves placing the liquid into a cell with three electrodes immersed into it. Firstly a negative reducing potential is applied to accumulate, or plate, metal ions on the working electrode. The potential is then ramped in a positive direction to a final potential, causing the metal to be stripped off back into solution. During this last phase the current in the cell is measured and analysed to determine the metal ion concentration.

Graphing the cell current versus cell voltage during the sweep portion of the voltammetric cycle gives a curve like that below. This is known as a voltammogram.

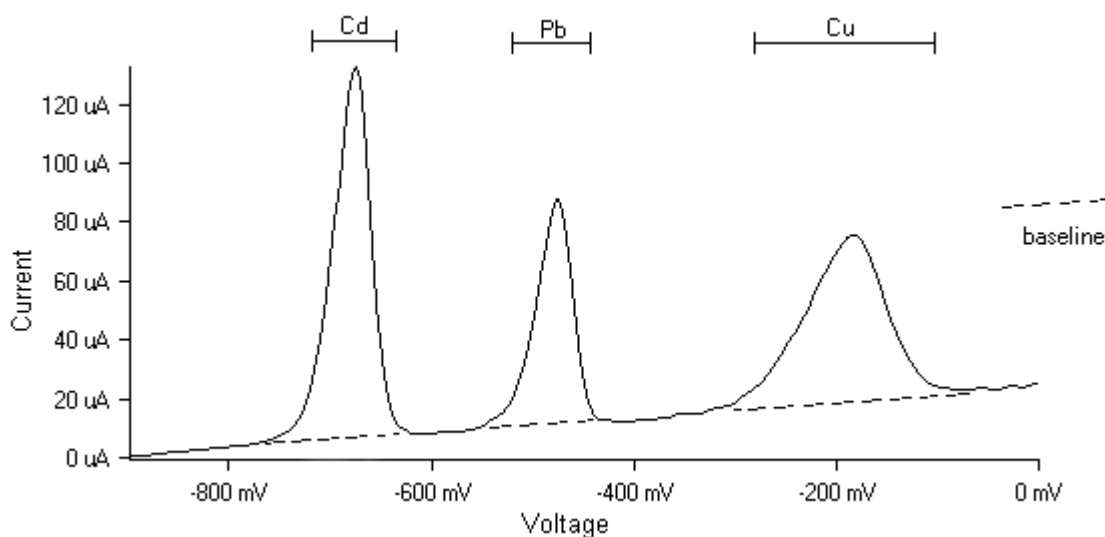


Figure 1: Data Curve Voltammogram for Cadmium, Lead and Copper

As different metals give a peak at their own characteristic potential, each distinct peak corresponds to a particular element in the solution. In the above example they are cadmium, lead and copper in that order. The size of the peak height ( $\mu\text{A}$ ) is proportional to the concentration of that metal in the cell.

There are two methods of determining a samples metal concentration supported by VAS: standard comparison and standard addition. Both of these methods rely upon using a standard solution of known concentration as a reference. For more information see Standard Comparison Analysis (on page **Error! Bookmark not defined.**) and Standard Addition Analysis (on page **Error! Bookmark not defined.**).

## Detection Windows

To differentiate and identify peaks on the voltammetric curve VAS uses detection windows. A detection window is a voltage range in which VAS will search for a peak.

Before finding peaks with VAS it is necessary to set a detection window for each element you wish to measure in the sample. If you know in advance where to place the detection windows you can set them from the **Initiate Run** dialog or they can be placed manually on the voltammogram.

---

## Peak Measurement

To measure the concentration of an element using voltammetry the size of the peak in the voltammetric curve is measured. The size of the peak is proportional to the concentration of the metal producing it, so that doubling the concentration doubles the size of the peak.

When a voltammetric curve is generated, there is an underlying background current due to the electrolyte and the voltammetric cell itself. To compensate for this, a peak *baseline* is drawn. This should simulate the background as closely as possible.

The two main methods of measuring peak size are peak height and peak area. Both of these measurements are taken from the baseline.

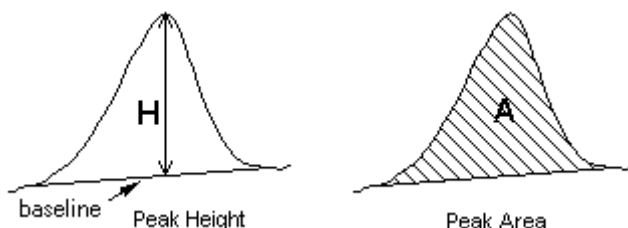


Figure 2: Peak Size Calculation By Peak Height and Peak Area

It is important to note that the absolute peak height is not taken into consideration when either the height or area is measured. For this reason a vertical shift of the entire voltammetric curve will not effect the results.

## Blanks and Artificial Blanks

As mentioned above, when a voltammetric curve is generated, peaks of interest are superimposed on a background current. Generally the baseline described above is sufficient compensation for this. However, to counteract extreme cases of this background current a blank curve (from a cell containing only electrolyte) can be sampled and subtracted from the standard, sample and standard addition curves.

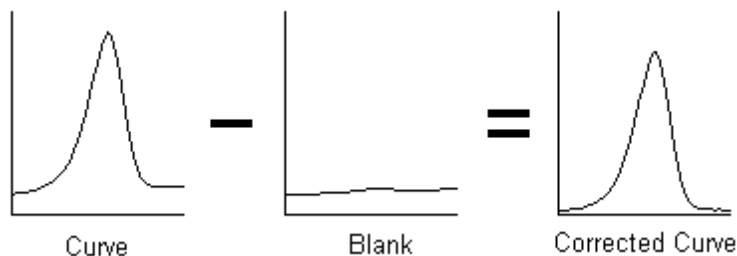


Figure 3: Blank Subtraction

Blank subtraction also serves to remove any bias introduced by impurities already present in the cell. For these reasons it is recommended that a blank curve be taken before a sample is analysed. The cell should not be rinsed after the blank is taken but the other solution should be injected immediately.

If no blank is available, either because the data has already been taken or it would be too slow to take a blank, it is possible to have VAS calculate an artificial blank. This is used in exactly the same way as a blank except that it clearly cannot counteract any impurities in the cell. See Calculating Artificial Blanks (on page **Error! Bookmark not defined.**).

---

Note: Wherever a blank or artificial blank can be used, if both are selected then VAS will use only the blank if one is available, or only the artificial blank if no true blank is present. Under no circumstances will both be used together.

---

## The Standard Comparison Method

Standard comparison analysis compares the signal from the sample with the signal from a standard. The Anodic Stripping Voltammetry (ASV) technique produces a linear response for most metals across a concentration range of one to two orders of magnitude, which allows a simple comparison to be made. It is assumed that peak size is linear with concentration and the standard is used to find the slope of the relationship. This is then used to calculate the concentration of the sample. With this technique it is best to use a standard cell concentration that is within the same range as the sample cell concentration.

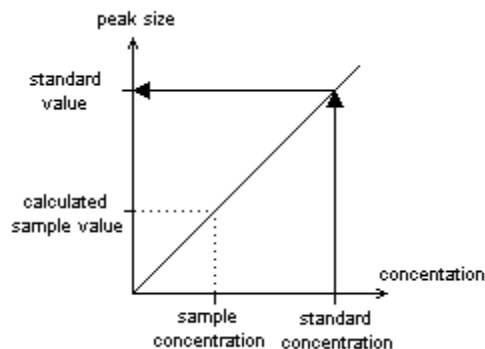


Figure 4: Standard Comparison

- To do a standard comparison analysis of a sample, you should take in the following order:
1. A blank curve (not essential, but recommended).
  2. A standard curve.
  3. A sample curve.

For more information on taking sample and standard curves see Standard Runs (on page **Error! Bookmark not defined.**) and Sample Runs (on page **Error! Bookmark not defined.**).

Also see Standard Comparison Analysis (on page **Error! Bookmark not defined.**) for information on analysing standard comparison data.

---

## The Standard Addition Method

Standard addition analysis initially analyses a sample and then one or more standard addition curves. A standard addition curve is taken by adding a small amount of standard solution into the sample already in the cell. This is repeated up to five times, usually adding the same amount of standard each time so that the concentration of standard steadily increases.

The sample and standard additions data is then plotted on a graph and a line of regression is fitted. This is then used to calculate the concentration present in the sample.

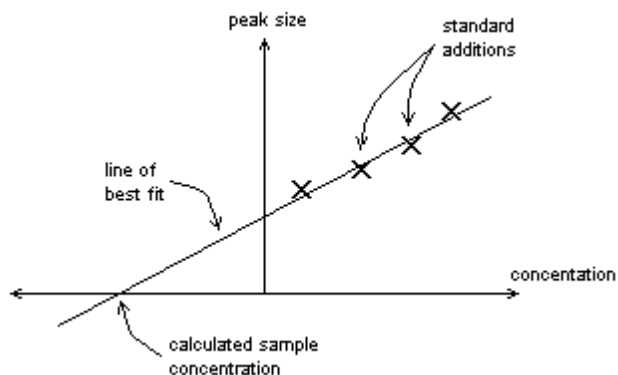


Figure 5: Standard Addition

This technique can compensate for matrix effects that enhance or depress the sample result. Because the matrix is accounted for the errors should be less for the standard addition technique than for standard comparison. For this reason it is normally a more accurate technique, but is more time consuming. For simple screening of samples where samples need to be classified as above or below a certain concentration, the standard comparison technique is perfectly acceptable.

VAS will display the regression graph in a Results dialog when the standard addition technique is used. A correlation value will also be shown, where 1 = sample and standards are all exactly on the regression line and the result will be reliable. Normally expect a correlation value  $> 0.998$ . A correlation value any lower means the sample and standards are scattered about the regression line and the result will be less reliable.

➤ To do a standard addition analysis of a sample, you should take in the following order:

1. A blank curve (not essential, but recommended).
2. A sample curve.
3. One or more standard addition curves.

For more information on taking sample and standard addition curves see Sample Runs (on page **Error! Bookmark not defined.**) and Standard Addition Runs (on page **Error! Bookmark not defined.**).

Also see Standard Addition Analysis (on page **Error! Bookmark not defined.**) for information on analysing standard addition data.

## CHAPTER 4

# Supported Analysers

VAS can be used with the following MTI voltammetric analysers:

- PDV 3000
- PDV 6000
- PTEA 6000
- PDV 6000*plus*
- TEA 3000

TO READ MORE OF THIS MANUAL, YOU WILL NEED TO  
REGISTER IN THE TECHNICAL SUPPORT AREA!