Application Bulletin 276/2 e

Validation of Metrohm VA instruments using Standard Operating Procedures

Of interest to:
General analytical laboratories;
B 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16

Summary

GLP (**G**ood **L**aboratory **P**ractice) requirements include the periodic check of analytical instruments for reproducibility and accuracy using **Standard Operating Procedures (SOPs).**

The user is advised to validate the Metrohm VA instruments as a complete, integrated voltammetry system, i.e. to perform a voltammetric determination using standard solutions of known content and critically assess the results using statistical methods.

Checking of the electronic and mechanical components of measuring instruments can and should be undertaken by qualified personnel of the manufacturing company as part of regular servicing. All Metrohm instruments are provided with start-up test routines. Each time the instrument is switched on, these test routines check whether the relevant assemblies are working correctly. If no error message is displayed, it can be assumed that the instrument is functioning faultlessly. Metrohm instruments are also supplied with built-in diagnostic programs that enable the user to check the functioning of certain components in the event of malfunctions or erratic behavior and to localize the fault. These diagnostic programs may also be integrated into a validation procedure.

The procedure described below is meant as a guideline for the preparation of standard operating procedures for checking your voltammetry instrument. The limiting values specified must be considered as recommendations. The limiting values to be applied depend on the requirements concerning the accuracy of the analysis system and must possibly be redefined for the particular standard operating procedure.

Application range

These test specifications are applicable to the following Metrohm VA instruments:

- 693 VA Processor with 694 VA Stand
- 746 VA Trace Analyzer with 747 VA Stand
- 757 VA Computrace
- 797 VA Computrace

Test intervals

Annually repeated tests of VA instruments appear appropriate. Additional validation runs are recommended whenever one or more components of the VA system are replaced.

A special validation is advisable whenever one or more components of the VA system have been replaced.

Internal instrument test routines

Metrohm instruments are equipped with internal instrument start-up test and test routines. In the start-up test of processor-controlled instruments, the display elements are checked and the contents of the program memories are tested by means of a checksum test. Proper functioning of the data memory area is tested with a write/read test.

In the case of the 693 VA Processor or 746 VA Trace Analyzer, the presence and operational readiness of 685 Dosimats, 700 Dosinos or a 695 VA Autosampler is checked. In the case of the 797 VA Computrace, the presence and operational readiness of 800 Dosinos is checked.

If the VA instruments are regularly serviced, it is generally possible to dispense with the specific validation of the instrument electronics.

Maintenance/Service

Careful maintenance and cleaning are indispensable requirements for the GLP-compliant operation of all instruments used in the laboratory. Particular attention should also be paid to the correct handling of such instruments. The Instructions for Use supplied with the instrument should be accessible to all workers in the laboratory. We also recommend regular servicing of all measuring instruments involved once a year. Many Metrohm agencies offer favorably priced service contracts for your instruments.

Method

In many cases the daily work involves only a few specific voltammetric methods. For the validation of the VA instrument, it is advisable to select a voltammetric method as similar as possible to one of the most frequently used methods. In addition, it should be possible to eliminate any method-specific error sources.

Metrohm recommends to validate the instrument in two steps:

1. ***Electronic validation (dummy-cell test)***

   In this step the complete electrical part of the voltammetry system is tested. If the result of the tests fulfills the specifications defined below, perfect functioning is assured.

   The detailed procedure for the dummy-cell test is described in the Instructions for Use of the corresponding instrument.

2. ***Chemical validation with lead-ion standard,*** including the electrodes, applying the method described below using the Pb ion standard solution (6.2301.100) and the KCl electrolyte (6.2308.020). Both solutions are delivered with the instrument.

   This step confirms the correct functioning of the entire voltammetry system including the electrodes.

The software of the 797 VA Computrace offers a GLP Wizard that supports the user during instrument validations.

VA systems with Dosimats, Dosinos, or an Autosampler

If dispensing devices are used for automatic addition of auxiliary solutions or standard solutions, or if an Autosampler is used, it is recommended to validate the VA system together with these peripheral instruments. Instead of introducing the necessary solutions manually into the measuring vessel, the dispensing devices should be used to add the solutions automatically.

By this procedure, the accuracy of the dispensing devices is tested as well.

Equipment required

- Pipets for 20 mL, 0.5 mL and 100 µL.

Chemicals required

- Ultrapure water
- Pb ion standard solution (6.2301.100)
- KCl electrolyte (6.2308.020)

Preparatory work

The pipets should be validated first.

New reference electrodes should be prepared as follows: fill the inner reference system and the electrolyte vessel with KCl electrolyte. Leave the completely prepared electrode standing for 30 minutes without immersion into the solution. This serves to obtain complete soaking of the diaphragm with electrolyte. After this, the electrode is ready for use.

Part 1: 693 VA Processor / 746 VA Trace Analyzer

Electronic validation (dummy-cell test)

Procedure

1. Attach the electrode cables of the VA Stand to the connectors of the dummy cell as follows:

   Cable AE → Connector AE

   Cable RE → Connector RE
2. Load the method «Test694.mth» or «Test747.mth» from the method storage.
3. Start the method and follow the instructions appearing on the screen.
4. Connect

   Cable WE → Connector WE-L.
5. A linearity test is carried out and a diagonal line is registered.
6. Connect

   Cable WE → Connector WE-D.
7. A peak is registered.
8. The registered voltammograms are printed.

The detailed procedure is described in the instrument manual:

693 VA Processor: chapter 7.7.1
(Method «Test694.mth»)

746 VA Trace Analyzer: chapter 7.7.1
(Method »Test747.mth»)

Voltammetric settings

Linearity test (connection WE-L)	
Electrode type	RDE
Measurement mode	DCT
Start potential	–200 mV
End potential	+200 mV
Voltage step	6 mV
Voltage step time	0.1 sec

Peak test (connection WE-D)	
Electrode type	RDE
Measurement mode	AC1
Start potential	–200 mV
End potential	–800 mV
Voltage step	10 mV
Voltage step time	0.2 sec
U.amplitude	25 mV
Phase angle	0°
Modul. frequency	25 Hz
Prep. cycles	0
Meas. cycles	2

Interpretation of results

The two diagrams recorded should be assessed as follows:

Linearity test (diagonal)

The diagonal must be straight and smooth.

Current at –200 mV	–2 ±0.4 µA
Current at +200 mV	+2 ±0.4 µA

Peak test

A symmetrical, bell-shaped peak should be obtained. The evaluation yields the peak potential, which is printed out in the full report. The peak potential and the shape of the peak are the most important characteristics. The curve must be smooth.

Peak potential	–500 ± 50 mV

If one of the test results exceeds the above tolerances, the instrument is probably not functioning properly. Please contact your local Metrohm representative.

Chemical validation using a lead ion standard

Preparing the test solution

1. Pipet 20 mL ultrapure water into the measuring vessel.
2. Pipet 0.5 mL KCl electrolyte (c(KCl)=3 mol/L) into the measuring vessel.
3. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel.

Performing the measurement

1. Load the method «TestPb.mth» from the method storage.
2. Start the method.
3. The solution is degassed and the polarogram registered three times.
4. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel and press <Enter>.
5. The polarogram of the first standard addition is registered three times.
6. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel and press <Enter>.
7. The polarogram of the second standard addition is registered three times.
8. The full report and the voltammograms are printed.

Voltammetric settings

Electrode type	DME
Measurement mode	DP
Start potential	–200 mV
End potential	–550 mV
Voltage step	6 mV
Voltage step time	0.4 sec

The detailed procedure is described in the instrument manual:

Chapters 7.7.2 to 7.7.5 (Method «TestPb.mth»).

Interpretation of results

The decisive parameters for the validation of measuring instruments are the accuracy and the scatter of the result.

Both values are calculated automatically by the microprocessor-controlled Metrohm 693 VA Processor and 746 VA Trace Analyzer.

To assess the recorded lead determination, the results printed in the full report for the concentration of lead and its total scatter are used. Both results depend greatly on the care taken in the preparation of the analysis solution and in the dispensing of the standard addition solutions.

If the analysis has been carried out with the required care, the following results should be obtained:

Accuracy	95%...105%	Final result	1 ±0.05 g/L
Scatter	≤ ±3%	Res. dev.	≤ ±0.03 g/L (±3%)

Part 2: 757 VA Computrace

Electronic validation (dummy-cell test)

Procedure

1. Attach the electrode cables of the VA Computrace to the connectors of the dummy cell as follows:

   Cable AE → Connector AE

   Cable RE → Connector RE

   Cable WE → Connector WE-L.

2. Load the method «Test757_L» from the «methods» directory.
3. Start the method.
4. A linearity test is carried out and a diagonal line is registered and printed.
5. Connect

   Cable WE → Connector WE-D.

6. Load the method «Test757_D» from the «methods» directory.
7. A peak is registered and printed.

The detailed procedure is described in chapter 7.9 of the software manual:

«Linearity test» (Method «Test757_L»)

«Peak test» (Method «Test757_D»)

Voltammetric settings

Linearity test (connection WE-L)	
Electrode type	RDE
Measurement mode	DCT
Start potential	–200 mV
End potential	+200 mV
Voltage step	6 mV
Voltage step time	0.1 sec

Peak test (connection WE-D)	
Electrode type	RDE
Measurement mode	DP
Start potential	–200 mV
End potential	–800 mV
Voltage step	10 mV
Voltage step time	0.4 sec
Pulse amplitude	50 mV
Pulse time	40 ms

Interpretation of results

The two recorded diagrams should be assessed as follows:

Linearity test (diagonal)

The diagonal must be straight and smooth.

Current at –200 mV	–2 ±0.4 µA
Current at +200 mV	+2 ±0.4 µA

Peak test

A symmetrical, bell-shaped peak should be obtained. The evaluation yields the peak potential, which is printed out in the full report.

Peak potential	–500 ±50 mV

If one of the test results exceeds the above tolerances, the instrument is probably not functioning properly. Please contact your local Metrohm representative.

Chemical validation using a lead ion standard

Preparing the test solution

1. Pipet 20 mL ultrapure water into the measuring vessel.
2. Pipet 0.5 mL KCl electrolyte (c(KCl)=3 mol/L) into the measuring vessel.
3. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel.

Performing the measurement

1. Load the method «TestPb in ion standard solution.mth» from the «methods» directory.
2. Start the method.
3. The solution is degassed and the polarogram registered three times.
4. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel and press <Start>.
5. The polarogram of the first standard addition is registered three times.
6. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel and press <Start>.
7. The polarogram of the second standard addition is registered three times.
8. The full report and the voltammograms are printed.

Voltammetric settings

Electrode type	DME
Measurement mode	DP
Start potential	–200 mV
End potential	–550 mV
Voltage step	6 mV
Voltage step time	0.4 sec

The detailed procedure is described in the instrument manual:

Chapter 7.9 «GLP test»
(Method «Test Pb in ion standard solution.mth»)

Interpretation of results

The decisive parameters for the validation of a measuring instrument are the accuracy and the scatter of the result.

Both values are calculated automatically by the PC program of the 757 VA Computrace.

To assess the recorded lead determination, the results printed in the full report for the concentration of lead and its total scatter are used. Both results depend greatly on the care taken in the preparation of the analysis solution and in the dispensing of the standard addition solutions.

If the analysis has been carried out with the required care, the following results should be obtained:

Accuracy	95%...105%	Final result	1 ±0.05 g/L
Scatter	≤ ±3%	Res. dev.	≤ ±0.03 g/L (±3%)

Part 3: 797 VA Computrace

Validation using the GLP Wizard

Diagnostics

Procedure

1. Activate the GLP Wizard start button by checking the «GLP Control» box.
2. Entries:

Procedure	Entry	Effect
Validation interval time	Number of days	Defines the interval between validations.
Next validation	–	Time remaining until next validation.
Display a message XX days before the certification expires	Number of days	Advance warning time span.
Action if validation expires	Show warning only	Issues warning after expiration of the validation interval.
	Stop measurements	Blocks any measurement after expiration of the validation interval.

Please note: *«Blank users», i.e. users without user names, are* ***not allowed.***

3. Start the GLP Wizard by clicking on <GLP Wizard>.

Diagnostics	Carries out several instrument hardware tests. Select the tests to be performed in the Diagnostics window.
Dummy cell tests	Carries out an electronic validation of the 797 VA Computrace.
Electrode test (only MME)	Carries out a validation of all three electrodes of the 797 VA Computrace.
Validation of a chosen method	Carries out, by means of Standard Operating Procedures (SOPs), a validation of accuracy and precision.

Please note: *Metrohm recommends to select all four procedures in the «GLP Wizard 797 VA Computrace» window. The tests are carried out in the sequence in which they appear on the list.*

4. Click on <continue>.
5. Connect the electrode cables of the VA Stand to the dummy cell as follows:

   Cable AE → Connector AE

   Cable RE → Connector RE

   Cable WE → Connector WE-L.
6. Click on <continue>.
7. In the «Diagnostics» window that has just appeared, click on the <Start> button to start the diagnostics program manually.
8. All the tests selected in the list are carried out.
9. Before starting the «Manual Stirrer Test», the sequence stops and waits for a decision by the user. If the stirrer performs correctly, confirm this by clicking on <Succeeded>.
10. The «Manual Valve Test» requires also a decision by the user.
11. Functions that are OK are marked by a green tick. Should any function fail to pass the diagnostic test, it will be marked by a red cross.
12. In the menu, under «File» → «Save Report as», a report can be stored on a data storage medium as a text file.
13. In the menu, under «File» → «Print Report as», a report can be issued on a connected printer.
14. Click on «File» → «Exit» to finish the diagnostic program. The GLP Wizard continues automatically with the dummy-cell test.

Dummy-cell test

Procedure

1. Attach the electrode cables of the VA Stand to the connectors of the dummy cell as shown on the screen:

   Cable AE → Connector AE

   Cable RE → Connector RE

   Cable WE → Connector WE-L.

2. Click on <continue> to start the method.
3. A linearity test is carried out and a diagonal line is registered and printed.
4. The screen displays a summary of the registered data and the corresponding comments.
5. Click on <continue>; the screen displays the prompt:
6. Connect

   Cable WE → Connector WE-D.

7. Click on <continue> to start the method.
8. A peak is registered and printed.
9. The screen displays a summary of the registered data and the corresponding comments.
10. Click on <continue>; this brings you to the electrode test.

Remark

The dummy-cell tests can also be carried out independently of the GLP Wizard. The detailed procedure is described in chapter 8.10 of the 797 software manual.

«Linearity test» (Method «Test797_L.mth»)

«Peak test» (Method «Test797_D.mth»)

Voltammetric settings

Linearity test (connection WE-L)	
Electrode type	RDE
Measurement mode	DCT
Start potential	–200 mV
End potential	+200 mV
Voltage step	6 mV
Voltage step time	0.1 sec

Peak test (connection WE-D)	
Electrode type	RDE
Measurement mode	DP
Start potential	–200 mV
End potential	–800 mV
Voltage step	10 mV
Voltage step time	0.4 sec
Pulse amplitude	50 mV
Pulse time	40 ms

Interpretation of results

The GLP Wizard evaluates the measurements automatically and assesses the results independently. If the dummy-cell test is carried out manually, the two recorded diagrams should be assessed as follows:

Linearity test (diagonal)

The diagonal must be straight and smooth.

Current at –200 mV	–2 ±0.4 µA
Current at +200 mV	+2 ±0.4 µA

Peak test

A symmetrical, bell-shaped peak should be obtained. The evaluation yields the peak potential, which is printed out in the full report.

Peak potential	–500 ±50 mV

If one of the test results exceeds the above tolerances, the instrument is probably not functioning properly. Please contact your local Metrohm representative.

Electrode test using the Multi-Mode Electrode (MME)

Preparing the test solution

1. Pipet 20 mL ultrapure water into the measuring vessel.
2. Pipet 0.5 mL KCl electrolyte (c(KCl) = 3 mol/L) into the measuring vessel.

Carrying out the electrode test

1. After the dummy-cell test you are prompted by the GLP Wizard to connect the cables to the electrodes.

   Cable AE → Auxiliary Electrode (AE)

   Cable RE → Reference electrode (RE)

   Cable WE → MME Working Electrode (WE)

Please note: *The electrode test can only be carried out if a Multi-Mode Electrode (MME) is connected to the 797 VA Computrace.*

2. Click on <continue> to start the electrode test.
3. If the electrode test has been successful, the screen displays the message «passed».
4. If one or several electrodes are not functioning properly, you get a message informing you which electrode showed a malfunction.
5. Click on <continue> to pass on to chemical validation.

Chemical validation using a lead ion standard

Preparing the test solution

1. Pipet 20 mL ultrapure water into the measuring vessel.
2. Pipet 0.5 mL KCl electrolyte (c(KCl)=3 mol/L) into the measuring vessel.
3. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel.

Performing the measurement

1. After the electrode test the GLP Wizard asks you to select the method to be validated.

Please note: *Metrohm recommends to carry out the chemical validation with the pre-installed method «Test Pb in standard solution.mth»*

2. Click on the < … > button to display the selection window.
3. Select the method «Test Pb in standard solution.mth» from the «methods» directory.
4. Click on <continue>.
5. Enter the following settings:

	Entry	
Number of measurements	1	
		☑
Substance *(is automatically taken from the method)*	Pb	
Analyte content	1	g/L
Tolerance final result [%]	5	
Maximum Res. dev. [%] (maximum scatter)	3	

6. Check the entries and correct them if required.
7. Click on <continue> to start the measurement.
8. When the sample ID is queried you can enter a sample identification. The volumes must not be altered.
9. To continue, confirm your entries with <OK>.
10. The solution is degassed and the polarogram registered three times.
11. You are prompted to add standard solution.
12. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel and press <OK>.
13. The polarogram of the first standard addition is registered three times.
14. You are again prompted to add standard solution.
15. Pipet 100 µL Pb ion standard solution (c(Pb) = 1 g/L) into the measuring vessel and press <OK>.
16. The polarogram of the second standard addition is registered three times.
17. The full report and the voltammograms are printed.
18. The screen displays a summary of all the tests performed. This summary can be issued on a printer via <print>.

Please note

Chemical validation using the lead ion standard can also be carried out without applying the GLP Wizard. The corresponding procedure is described in detail in the software manual of the instrument.

Chapter 2.7 «GLP»
(Method «Test Pb in ion standard solution.mth»)

Voltammetric settings

Electrode type	DME
Measurement mode	DP
Start potential	–200 mV
End potential	–550 mV
Voltage step	6 mV
Voltage step time	0.4 sec

Interpretation of results

The GLP Wizard evaluates the measurements automatically and assesses the results independently.

If the test is carried out manually, the results have to be assessed by the user. The decisive parameters for the validation of the measuring instrument are the accuracy and the scatter of the result.

Both values are calculated automatically by the PC program of the 797 VA Computrace.

To assess the recorded lead determination, the results printed in the full report for the concentration of lead and its total scatter are used. Both results depend greatly on the care taken in the preparation of the analysis solution and in the dispensing of the standard addition solutions.

If the analysis has been carried out with the required care, the following results should be obtained:

Accuracy	95%...105%	Final result	1 ±0.05 g/L
Scatter	≤ ±3%	Res. dev.	≤ ±0.03 g/L (±3%)

Part 4: General information

Recommendations for troubleshooting

For troubleshooting hints see also the manuals of the instruments concerned. Additional information is also available in the Metrohm Monograph «First aid for polarography and voltammetry» (8.693.1073). The following lists are not complete and only show examples for possible sources of errors.

Possible sources of errors

Measuring vessel	contaminated
Pipets	contaminated, inaccurate
Water, electrolyte	contaminated
Working electrode	capillary blocked or defective
	needle soiled or deformed
	mercury oxidized
	loose electrical contact
Reference electrode	electrode empty
	blocked diaphragm
	loose electrical contact
Auxiliary electrode	loose electrical contact

If the systematic error is too high, i.e. if the accuracy is insufficient

- Check ultrapure water for impurities.
- Clean pipets.
- Check pipets for accuracy and reproducibility.
- Clean measuring vessel.
- Check electrodes and voltammetric parameters.
- Check electrolyte for impurities.
- Check nitrogen purity.
- Increase degassing time.

If the relative standard deviation is too high, i.e. if the reproducibility is bad

- Clean pipets.
- Check pipets for accuracy and reproducibility.
- Check pressure reducing valve for accuracy.
- Check electrodes and voltammetric parameters.
- Check nitrogen gas cylinder for minimum pressure.

Procedure in case of non-conforming values

All non-conforming values must be commented on in the validation record and the subsequent procedure noted.

If excessive deviations are found, the different points under «Recommendations for troubleshooting» must be carefully checked and the interferences eliminated. It is essential to repeat the validation. If unsatisfactory results are still obtained when the test series is repeated, the validation must be performed again by a different person.

If doubt exists regarding the precision of the dispensing systems, these latter can be checked separately (see Metrohm Application Bulletin no. 283).

Literature

Further information on voltammetry can be found in the following publications:

- Metrohm Application Bulletin no. 283, Validation of Metrohm burets
- Metrohm Monograph «First Aid in Polarography and Voltammetry»

Please note

On the following pages you will find an example of a validation. The last page can be used as a master for copies of the validation record.

Validation Record **VA Instrument**		Company : Division :	*Metrohm Ltd* *CC VA*
Date:	*14.06.2006*	User:	*Metrohm*
Time:	*17:00*		
Instrument :	*797 VA Computrace*	Serial number:	*02173*

Electronic Validation (Dummy Cell Test)

Linearity Test:	Measured values	Tolerances	Test passed
Current at –200 mV	*-2 µA*	-1.6 µA ... –2.4 µA	yes ☒ no ☐
Current at +200 mV	*+2 µA*	+1.6 µA ... +2.4 µA	yes ☒ no ☐

Peak Test:	Measured values	Tolerances	Test passed
Peak voltage	*-497 mV*	-450 mV ... –550 mV	yes ☒ no ☐

Chemical Validation

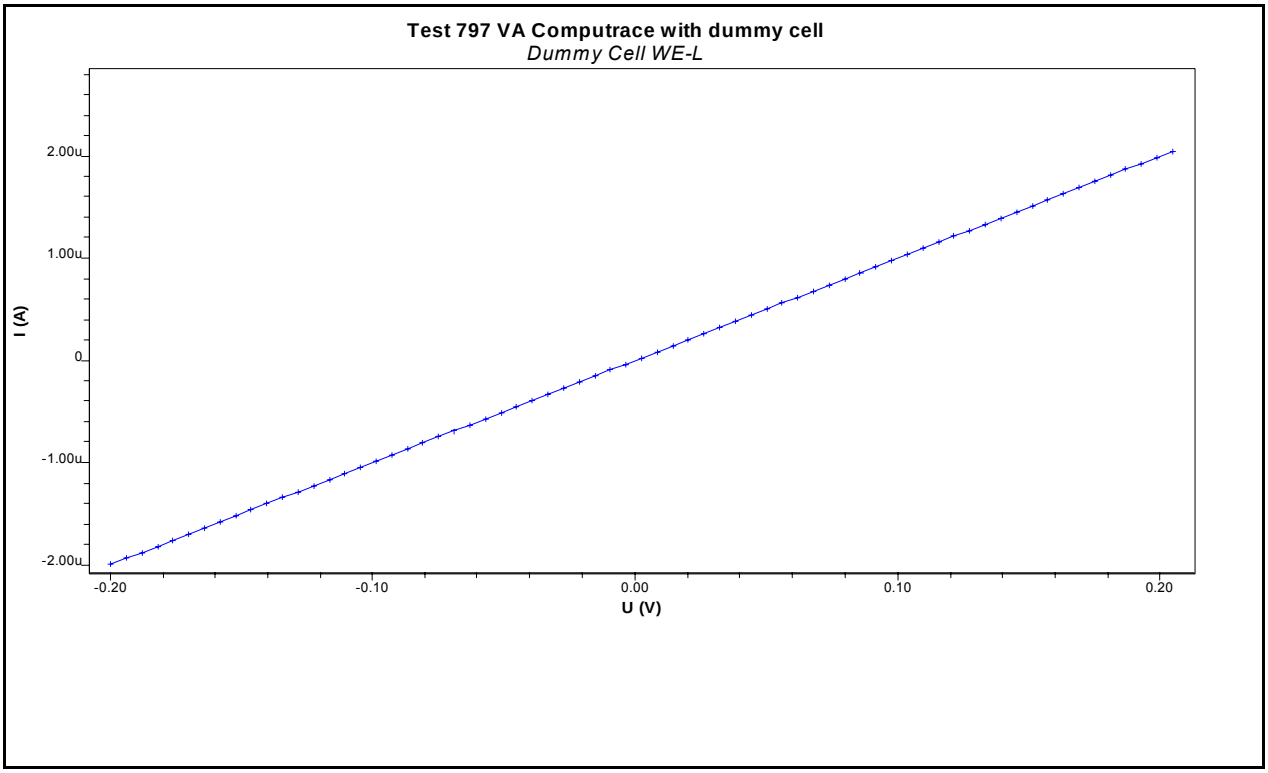
Measuring solution:	*20 mL water + 0.5 mL KCl + 100 µL Pb standard*
Electrolyte:	*c(KCl) = 3 mol/L*
Standard Solution:	*β(Pb) = 1 g/L*

	Measured values	Tolerances	Test passed
Final Result:	*1.034 g/L*	0.95 ... 1.05 g/L	yes ☒ no ☐
Res. Dev.:	*1.199 %*	≤ ± 0.03 g/L (± 3%)	yes ☒ no ☐

Validation passed: yes ☒ no ☐

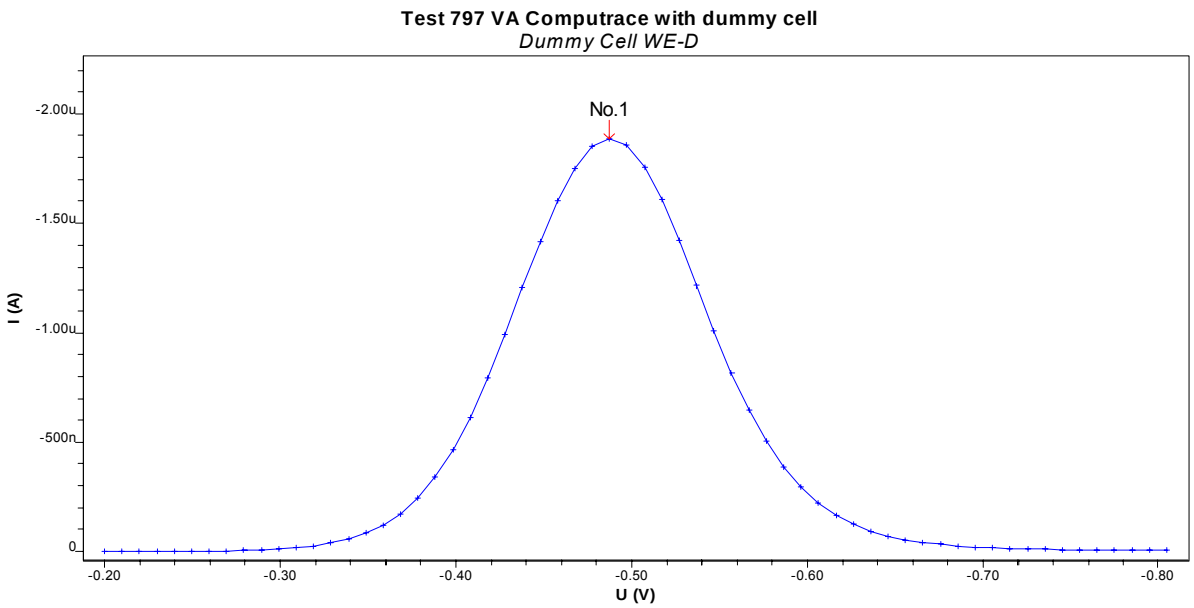
Signature:		Visa:	

Linearity test



Peak test

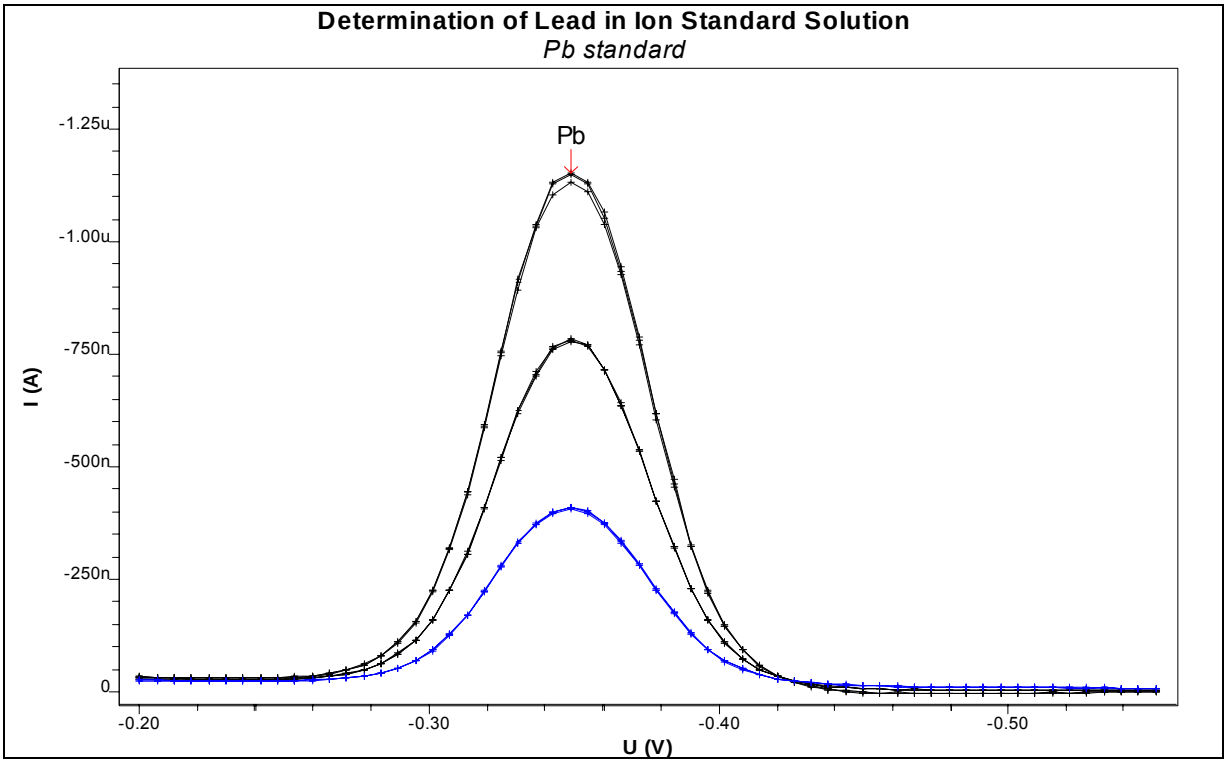
```
========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 2173) ===========
Determination  : 06010805_Dummy Cell WE-D.dth
Sample ID      : Dummy Cell WE-D
Creator method : Metrohm          Date : 2005-07-05          Time:  19:02:03
Creator determ.: Metrohm          Date : 2006-06-01          Time:  08:05:49
Modified by    : ---              Date :                     Time:
--------------------------------------------------------------------------------
Method         : Test797_D.mth
Title          : Test 797 VA Computrace with dummy cell
Remark1        : connect to WE-D
Remark2        : connect AE and RE
--------------------------------------------------------------------------------
Sample amount  :  10.000 mL
Cell volume    :  10.000 mL
--------------------------------------------------------------------------------
Substance      : No.1
Conc.          : ---
Conc.dev.      : ---

VR     V       uA      I.mean  Std.Dev.  I.delta   Comments
-----  ------  ------  ------  --------  --------  ----------------------
1 - 1  -0.488  -1.884  -1.884       ---     0.000


Substance    Calibr.      Y.reg/offset        Slope Mean deviat.
------------ ------------ ------------ ------------ ------------

Solutions
--------------------------------------------------------------------------------

 No. Content                                          Predose (mL)
 --- ------------------------------------------------ ------------


Final results                          +/- Res. dev.   %     Comments
----------------------------------    ------------------    -------------------

No.1:
Peak No.1        =      ---   g/L                          No result found
```

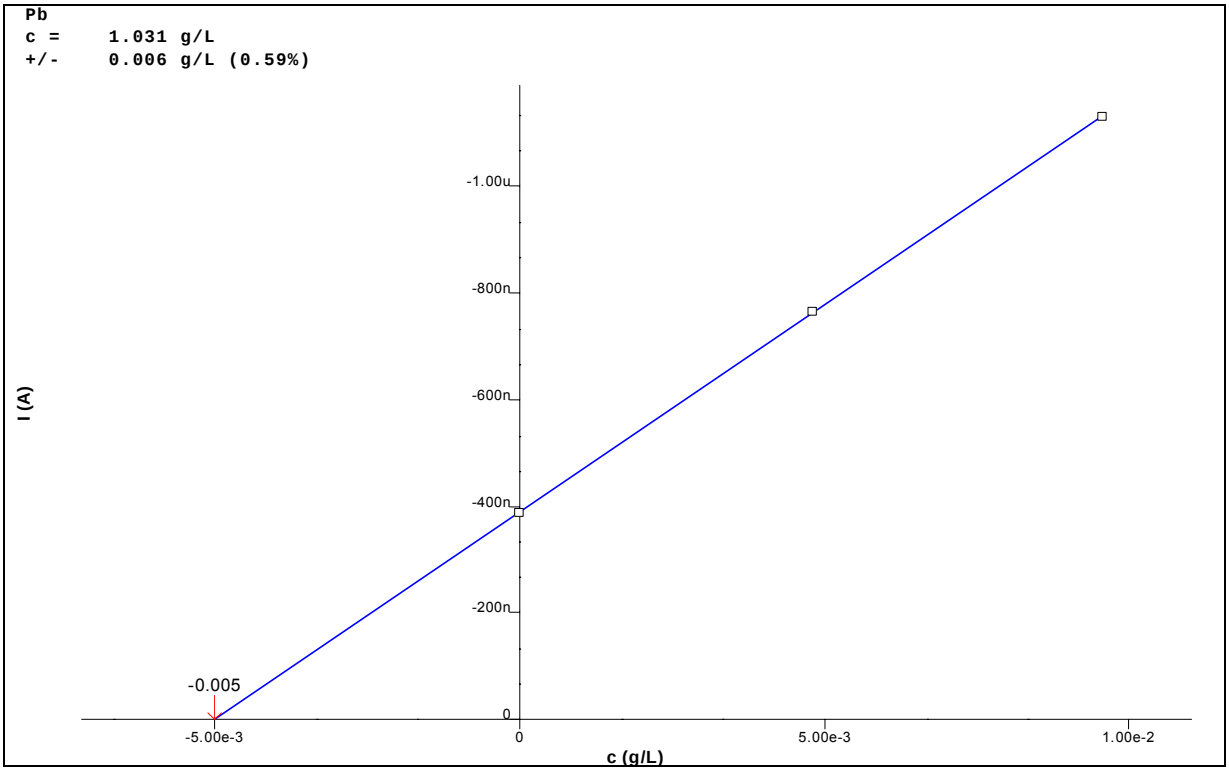


Report of chemical validation

```
============ METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 2173) =========
 Determination  : Test Pb in standard solution.dth
 Sample ID      : Pb standard
 Creator method :                  Date :                    Time:
 Creator determ.: Metrohm          Date : 2005-06-27         Time:  14:45:30
 Modified by    : ---              Date :                    Time:
--------------------------------------------------------------------------------
 Method         : Test Pb in standard solution.mth
 Title          : Determination of Lead in Ion Standard Solution
 Remark1        : 20 ml water + 0.5 ml KCl (3 mol/l) + 100 µl Pb ion standard so
lution
 Remark2        : c(Pb ion standard solution) = 1 g/l
--------------------------------------------------------------------------------
 Sample amount  :   0.100 mL
 Cell volume    :  20.600 mL
--------------------------------------------------------------------------------
 Substance      : Pb
 Conc.          :   5.004 mg/L
 Conc.dev.      :   0.030 mg/L     ( 0.59%)
 Amount         : 103.073 ug
 Add.amount     : 100.000 ug

 VR     V       uA      I.mean  Std.Dev.  I.delta   Comments
 -----  ------  ------  ------  --------  --------  ----------------------
 1 - 1  -0.349  -0.388  -0.389     0.001     0.000
 1 - 2  -0.349  -0.389
 1 - 3  -0.349  -0.390
 2 - 1  -0.349  -0.764  -0.765     0.003    -0.376
 2 - 2  -0.349  -0.768
 2 - 3  -0.349  -0.762
 3 - 1  -0.349  -1.119  -1.132     0.011    -0.367
 3 - 2  -0.349  -1.139
 3 - 3  -0.349  -1.137


 Substance    Calibr.      Y.reg/offset        Slope Mean deviat.
 ------------ ------------ ------------ ------------ ------------
 Pb           std.add.      -3.893e-007  -7.781e-005   4.617e-009

 Solutions
--------------------------------------------------------------------------------

  No. Content                                           Predose (mL)
  --- ------------------------------------------------- ------------


 Final results                         +/- Res. dev.   %     Comments
----------------------------------    ------------------    --------------------

 Pb:
 Lead             =    1.031   g/L    0.006        0.593
```

Voltammogram and calibration curve of chemical validation





Validation Record		Company :	
VA Instrument		Division :	
Date:		User:	
Time:			
Instrument :		Serial number:	

Electronic Validation (Dummy Cell Test)

Linearity Test:	Measured values	Tolerances	Test passed
Current at –200 mV		-1.6 µA ... –2.4 µA	yes ☐ no ☐
Current at +200 mV		+1.6 µA ... +2.4 µA	yes ☐ no ☐

Peak Test:	Measured values	Tolerances	Test passed
Peak voltage		-450 mV ... –550 mV	yes ☐ no ☐

Chemical Validation

Measuring solution:	
Electrolyte:	
Standard Solution:	

	Measured values	Tolerances	Test passed
Final Result:		0.95 ... 1.05 g/L	yes ☐ no ☐
Res. Dev.:		≤ ± 0.03 g/L (± 3%)	yes ☐ no ☐

Validation passed: yes ☐ no ☐

Signature: ╳		Visa: ╳	