CVS – Cyclic Voltammetric Stripping with Metrohm

CVS in the Laboratory
CVS Atline Systems
CVS Online Systems

Metrohm

CVS – Cyclic Voltammetric Stripping

02

Cyclic Voltammetric Stripping (CVS) and Cyclic Pulse Voltammetric Stripping (CPVS) are widely used methods in the electroplating industry for the determination of organic additives in electroplating baths. A simple, robust and affordable rotating disk electrode made of platinum is used in these analyses. For many technical coatings, particularly in the manufacture of PCBs and semiconductor components, this method is an essential part of production control.

The most important fields of application are acidic copper baths and tin-lead baths. Quantitative determination of the additive is carried out via its influence on the deposition of the main component of the electroplating bath. As the measurement is based on an electrode reaction that corresponds to the production process, the activity of the additive and thus its effectiveness in the electroplating process can be measured directly. The concentration of the additive can be determined exactly with CVS or CPVS. The effective concentration of the particular additive in the bath sample is shown and printed out directly in mL additive per L bath liquid. This means that topping up to the preset bath concentration can be carried out with highest accuracy. This guarantees continuous and interference-free operation of the process. The method has become generally accepted in the electroplating industry, in particular thanks to the accuracy of the analytical results it supplies.

The most important fields of application

Suppressors
DT – Dilution Titration

Brighteners
LAT – Linear Approximation Technique
MLAT – Modified Linear Approximation Technique

Leveler
RC – Response Curve

Applications

Suppressor determination

Irrespective of whether hourly, daily or even weekly determination of suppressor components is necessary, dilution titration allows the suppressor concentration to be reliably determined.

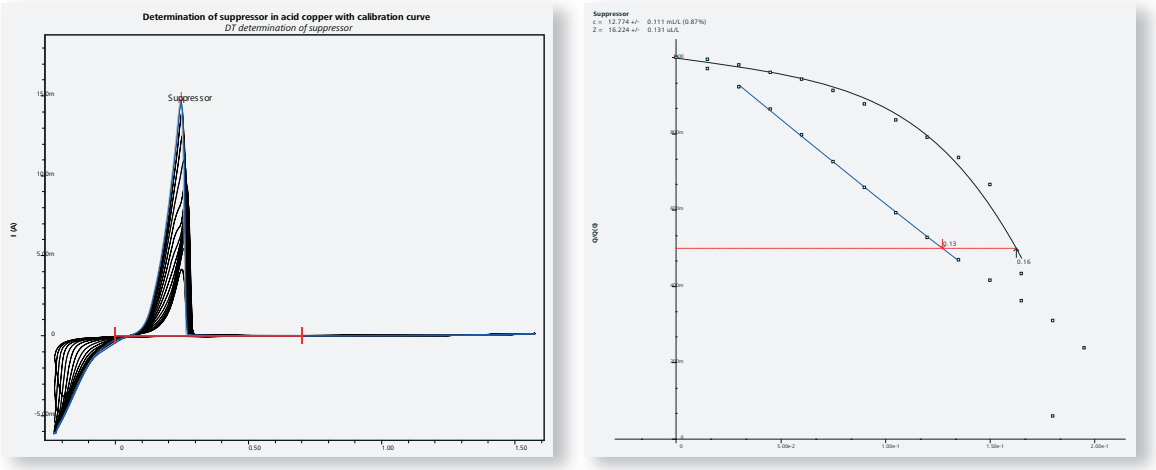
Calibration curve

To calibrate the method, a standard solution is added to a base solution (VMS = Virgin Make-up Solution) in very small steps and a calibration curve is recorded. This calibration curve is valid over a long period, but can be checked or renewed at any time. A new file is created for each measurement to avoid overwriting of previous calibration data.

Each measurement is traceable and can be saved to the hard disk together with the calibration if required.

Determination

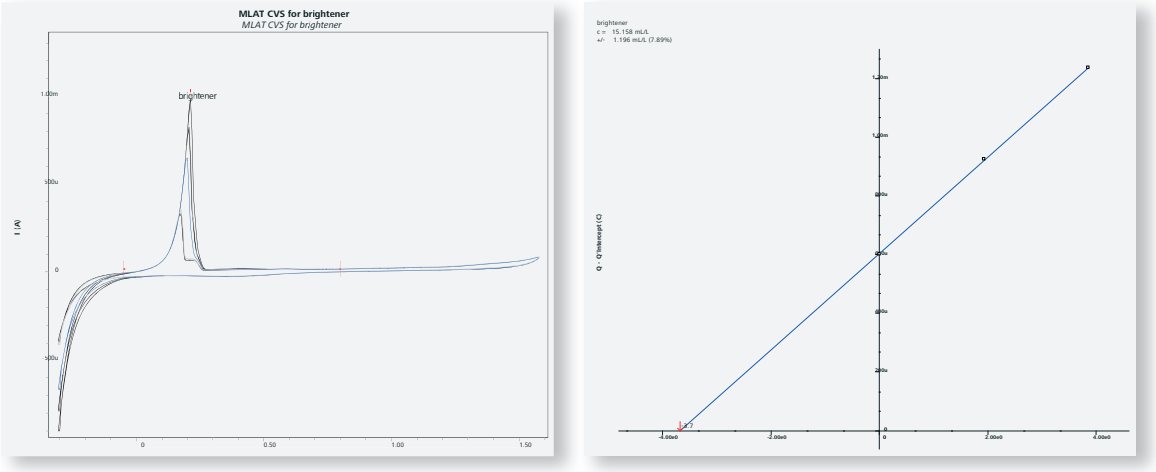
The software automatically takes over the measurement parameters and the calibration data from the previously recorded calibration, inserts these into the measurement method and is therefore ready for measurement in just a few steps. Simple matching of the addition volume to the sample, even saving VMS is possible.



Measuring curve for a suppressor determination – dilution titration

Brightener determination

MLAT (Modified Linear Approximation Technique) or LAT (Linear Approximation Technique) allows fast, simple and precise quantification of the amount of added brightener. Within just a few minutes time the result can be read off the screen, exported to a LIMS or used for the automatic generation of a report. The quantitative determination of the additive is carried out via its influence on the deposition of the main component of the electroplating bath. As the measurement is based on an electrode reaction corresponding to the production process, the activity of the additive and thus its effectiveness in the electroplating process can be measured directly.



Measuring curve for a brightener determination – MLAT

Leveler determination

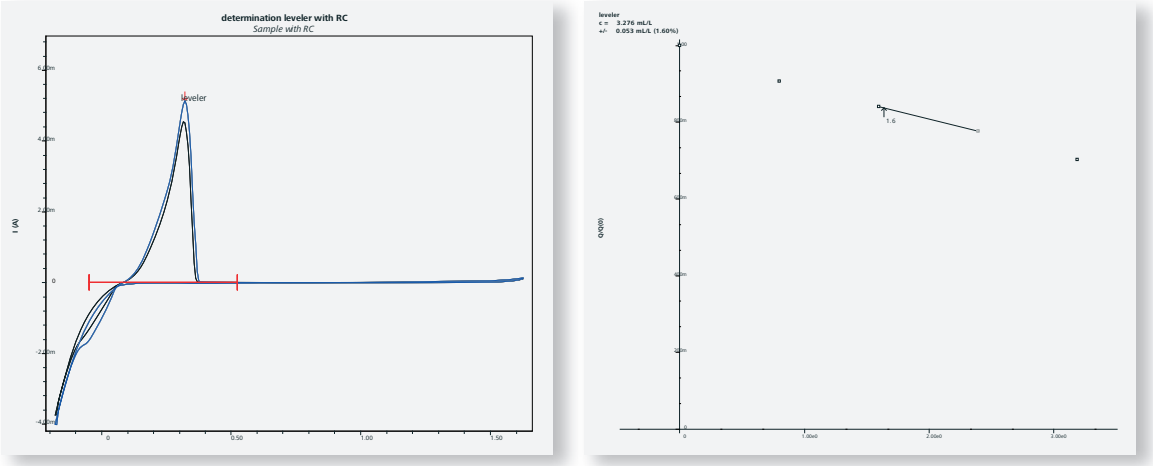
Besides the additives previously mentioned (brightener and suppressor), modern electroplating baths have a third substance added (leveler). This bath additive can also easily be analyzed with the 797 VA Computrace. The method for determining levelers, which is also performed in two steps (calibration and determination), completes the comprehensive monitoring of the components used.

Response Curve

In this measurement mode, which is similar to suppressor determination with DT, a calibration or response curve is recorded in the first step. An electrolyte made up of VMS and additives is used as the base solution here. This calibration curve is also valid over a long period, but can be checked or renewed at any time. Each individual calibration is traceable and is saved to the hard disk. Calibration can be carried out at any time during, between or after a sample series.

Determination

The software automatically takes over the measurement parameters and the calibration data from the previously recorded response curve, inserts this data into the method and is therefore ready for measurement in just a few steps. The bath sample can be added directly to the prepared electrolyte solution or solutions can be exchanged.



Measuring curve for leveler determination – Response Curve

797 VA Computrace – the laboratory solution

The lab measuring system allows fast, flexible and, of course, also precise performance of additive determination. CVS measurements can be realized in every laboratory, even in a very limited space.

Manual or automated operation

Even without additional accessories, the 797 VA Computrace is a fully functional analysis system meeting highest standards of accuracy and sensitivity. We offer you enhanced convenience with various upgrade options.

MVA systems for the CVS technique

Our recommendation for additive determination with CVS

The semi-automated 797 VA Computrace system for easy determination of additives with CVS.

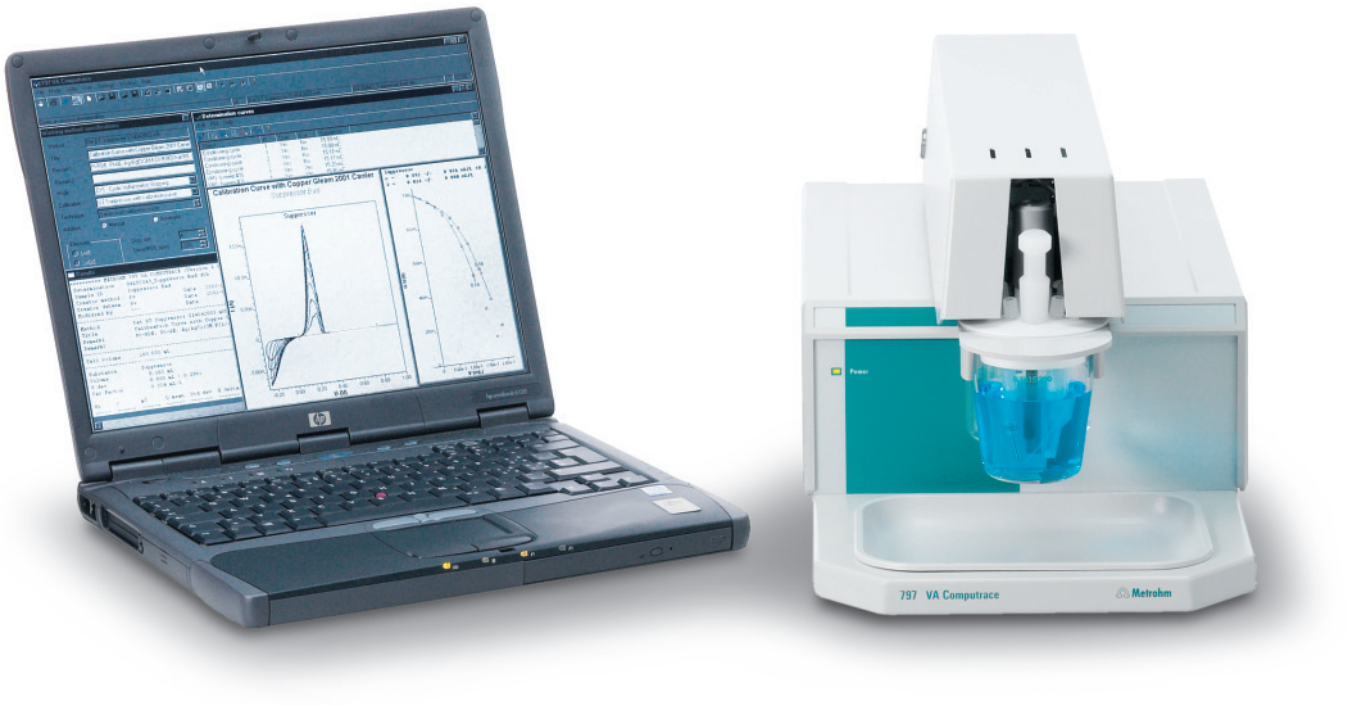
MVA-12 is the standard system for routine determination of organic additives in individual samples. Without a sample changer, it allows convenient determinations with a minimum of operator interventions. Automatic addition of auxiliary solutions such as VMS, intercept or standard solutions is carried out with model 800 Dosino dosing systems. For brightener determination the sample has to be added manually. For suppressor determination, sample addition is also automatic. With the aid of an optional pump, the measuring vessel can be automatically rinsed after each analysis.

Fully automatic determination of organic additives in sample series.

Fully automatic system with sample changer for CVS analysis in the routine laboratory.

MVA-13 is our high-end system for the determination of additives in electroplating baths with the samples being added automatically by the 838 Advanced VA Sample Processor. The suppressor content of up to 56 samples can be determined.

When determining brighteners, up to 28 samples can be analyzed. The possibility of recalibrating determinations during a series of samples guarantees outstanding accuracy. It is also possible to combine different methods in a single measuring procedure. The 843 Pump Station is used to automatically empty and rinse the measuring vessel after each sample.



The 10 most important benefits

The 797 VA Computrace opens up new possibilities:

- Determination of additives in electroplating and voltammetric trace analysis with a single instrument
- Outstanding precision thanks to the combination of proven Metrohm solid and reference electrodes with a highly sensitive potentiostat
- Perfect Liquid Handling with proven Dosino technology
- Automation with the 838 Advanced VA Sample Processor, 875 ProcessLab or an Applikon ADI 2045 VA online system
- Important analysis methods from leading bath manufacturers are supplied
- Results output in many different formats
- Built-in quality assurance with GLP mode, individual rights of access for each user and automatic electrode test
- Simple operation thanks to the user interface oriented on Windows operating procedures
- Connection to the USB port of the PC
- Thermostated operation (optional)

Quality assurance made easy with the 797 VA Computrace

Acces rights
Acces rights to every program part can be freely defined for each user. This allows convenient user management.

Validation with the GLP Wizard
The software automatically checks the validation intervals of the analysis system and informs the user when the next validation is due. Each report shows whether the validation is still valid. The GLP Wizard guides the user step by step through the various validation tests and automatically determines whether the test has been passed or not.

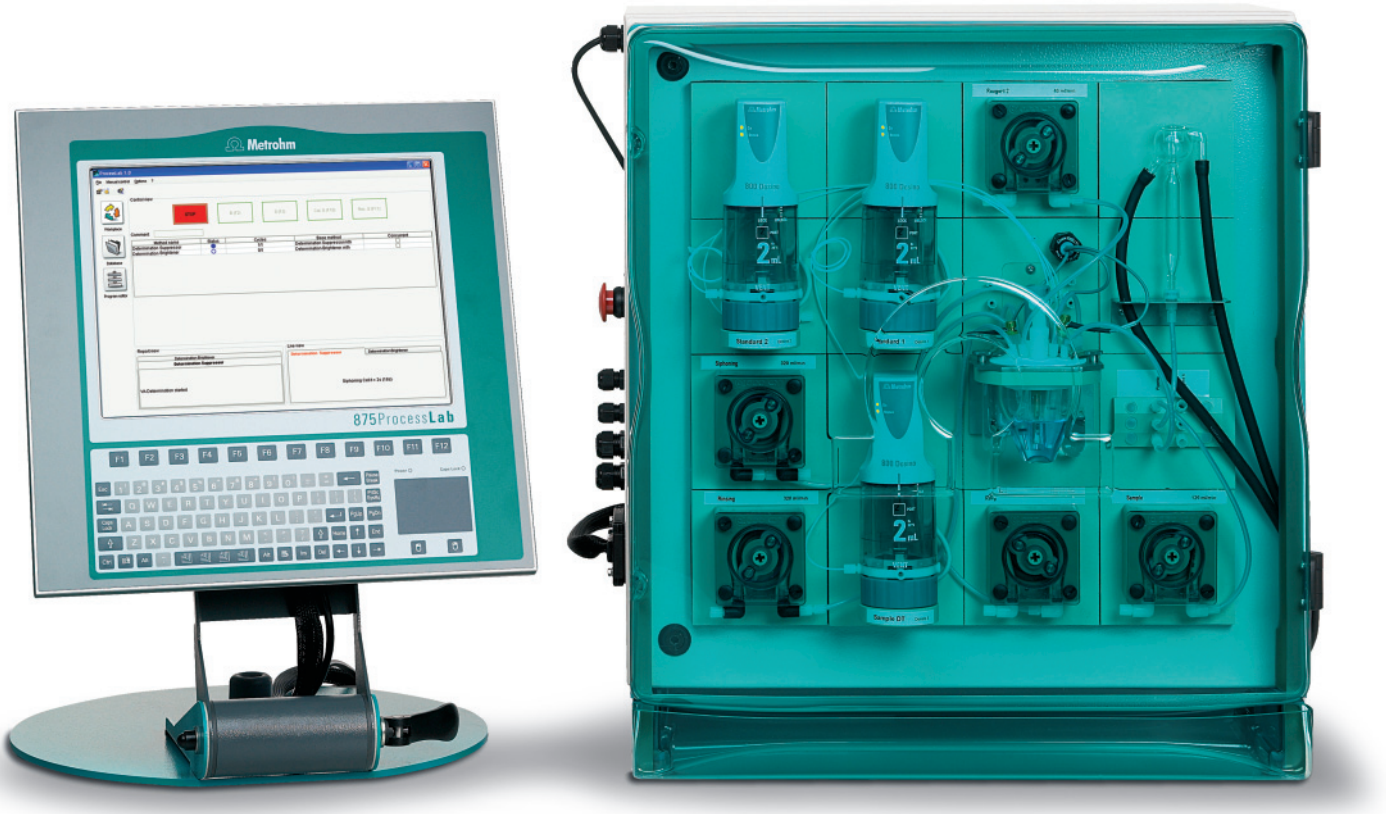
Diagnosis
The built-in diagnosis program allows the individual components of the instrument to be checked. This diagnosis is a part of the GLP Wizard, but it can also be carried out independently.

Electrode test
The electrodes used are automatically checked before each determination. If problems occur then the faulty electrode is identified and the fault shown on the screen. This test can, of course, also be triggered manually to check the system.

Curve evaluation
The software of the 797 VA Computrace automatically evaluates the measured curves and calculates the final result.

Certified quality
The development and manufacture of the instrument and the software have been carried out in accordance with the most stringent quality assurance criteria, which we guarantee by issuing a quality certificate. The reference electrode and the electrolyte and standard solutions contained in the accessories can be identified by their serial number and are supplied together with individual certificates.

ProcessLab Atline Systems – process-oriented, fully automatic determination of organic additives



Atline versus online analysis systems

In an atline system the sample is taken manually and transferred to the system. In this way various samples from different process stages or units can be easily analyzed. In contrast to this, in an online system the sample is automatically transferred to the analyzer via a bypass.

Fully or semi-automated system with or without sample changer for atline CVS process analysis

Voltammetry (VA), which is already well established in the laboratory, and Cyclic Voltammetric Stripping (CVS) are also available for on-site operational routine analysis. ProcessLab is optimally designed for additive determination in electroplating baths, whereby the samples are transferred to the system either one by one or automatically by a sample changer.

All components are accommodated in a rugged splash-protected housing (IP 54) for use in rough production environments. An operating unit with touch-sensitive surface allows easy and convenient operation.

The system is controlled by ProcessLab software with a simplified user interface. Defined analysis sequences can be started at the press of a button thus ensuring error-free operation. The system is configured according to the process requirements and is therefore optimally matched to the process.

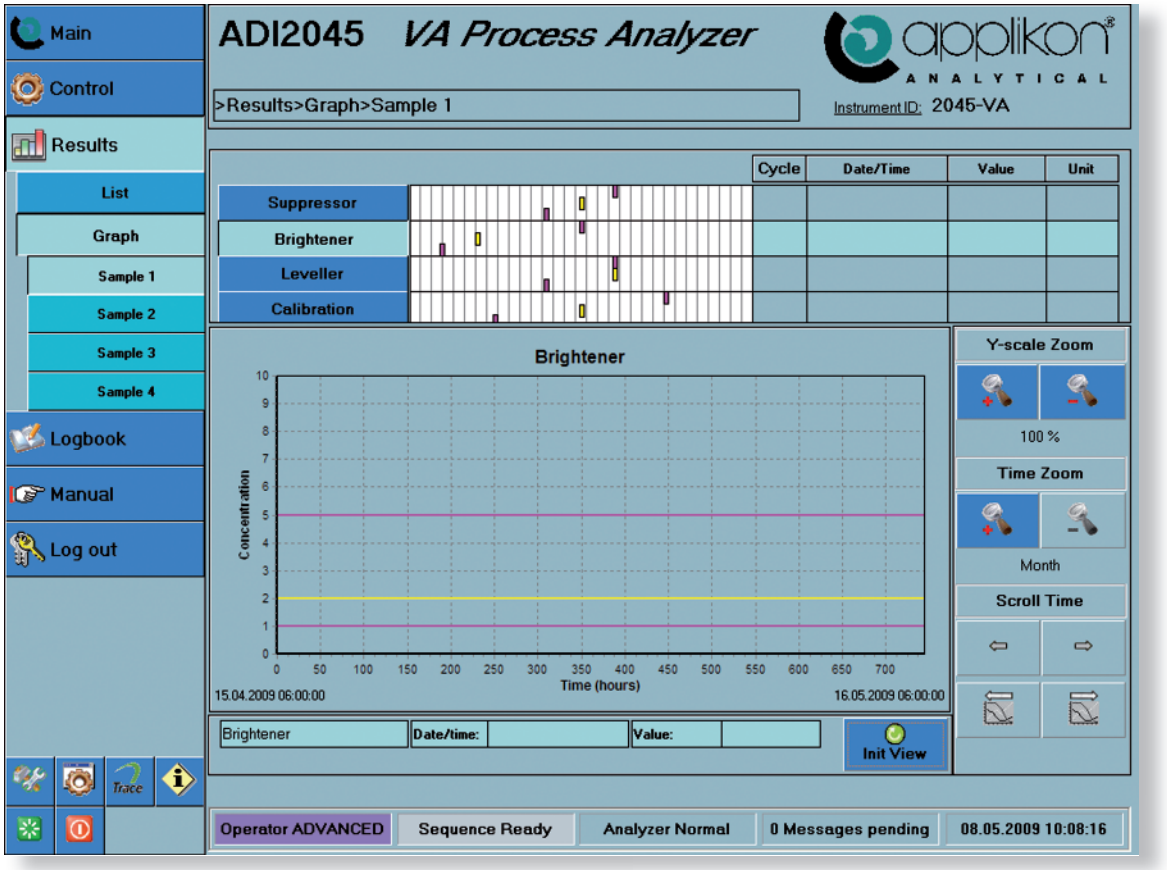
A database with the option of graphical presentation of the results in an SPC chart (Statistical Process Control chart) provides information on the status of the production line under control at any time.

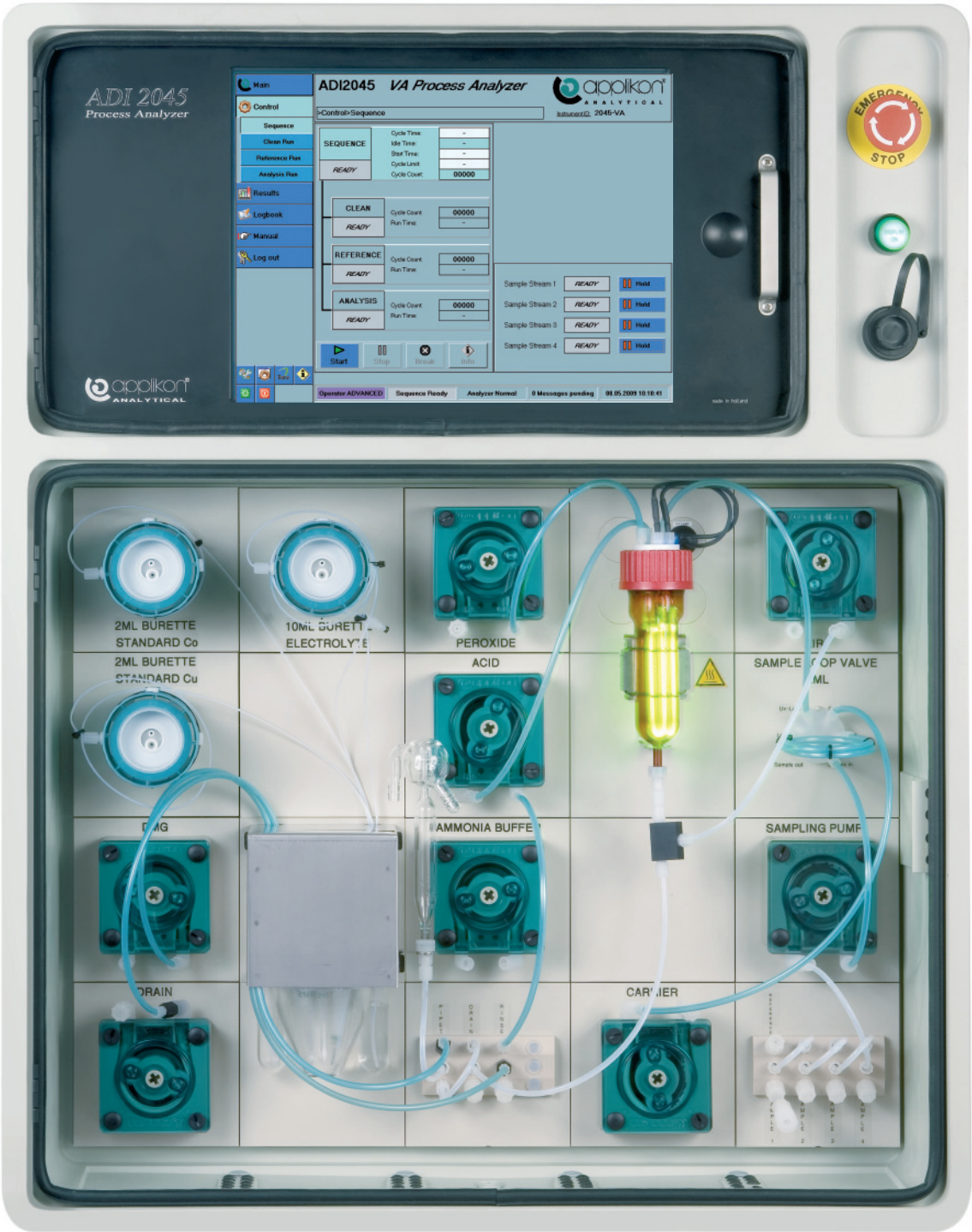
CVS online process analysis

Applikon ADI 2045 – Online – fully automated determination of organic additives with CVS

The Applikon 2045VA is an online instrument for performing voltammetric analyses. This measuring technique has been used for decades in routine laboratory operation; Applikon has now also successfully implemented it for online analysis. Quality control of electroplating baths with CVS can now take place directly in an operational environment.

The system is controlled by a combination of industrial PC and programmable logic controller (PLC). Whereas the PLC controls all the external units and the additional pumps, the PC controls the VA Computrace and the Dosino dosing systems connected. The touch screen user interface allows easy and intuitive operation. An additional USB keyboard or mouse can be connected to the system, as required. The operating section of the software contains a large number of options, whereas in the status section the current system status can be checked at any time.





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Technical specifications

Laboratory system

797 VA Computrace potentiostat / galvanostat	Voltammetric measuring stand with built-in potentiostat and galvanostat	
	Voltage range	±5 V
	Output voltage	±12 V
	Current range	±80 mA
	Current measurement	7 ranges (10 nA to 10 mA)
	Sweep rate (CV)	<1 mV/s...3 V/s (at 1 mV resolution)
		<1 mV/s...35 V/s (at 10 mV resolution)
Power supply	Voltage	100...240 V; frequency 50...60 Hz
	Power consumption	120 VA
Temperatures	Nominal working range	0...45 °C at 20…80% relative humidity

ProcessLab – atline-process analysis

Sample	Sampling frequency	1...20 / day
	Analysis time	typically <15 min
	Sample types	>10
	Sample volumes	0.2 mL...250 mL
	Sample temperature	5 °C...45 °C
Connections	Digital inputs	24 V DC, e.g. emergency off, liquid level sensors, ...
	Digital outputs	24 V DC, stirrer, peristaltic pumps, ...
	Analog inputs	4...20 mA, number variable
	Analog outputs	4...20 mA, number variable
	Relay outputs	1 A at 120/230 V
Operation	TFT screen with fully functional membrane keyboard and touch pad	
	Touch screen optional	
Temperature	Nominal working range	0..+40 °C at 20…80% relative humidity

ADI 2045VA – online process analysis

Samples and sample flow	Sampling frequency	programmable
	Analysis time	typically approx. 30 min
	Sample currents	maximum 4
	Sample volume	0.2 mL...50 mL
	Sample temperature	5 °C...45 °C
	Sample pressure	0...0.5 bar, i.e. 0...7.2 psi (without preparation)
Connections	Results output (analog)	4...20 mA, max 16 channels
	Digital remote control	24 VDC, Start, Stop, Continue, Samples 1,2,3,4
	Digital input detector	24 V DC, max 12 channels (leak sensor, reagent monitoring, etc.)
	Digital output relay	2 A, Power On, Sequence Running, Warning, Error, Result alarm, 1,2,3,4, Service
General	Touch screen operation	

Ordering Information

797 VA Computrace – for laboratory use

2.797.0030 797 VA Computrace for CVS (Cyclic Voltammetric Stripping) for analyzing organic additives in electroplating baths. Measuring stand with integrated potentiostat and galvanostat. Three-electrode system with rotating platinum disc electrode (Pt RDE), Ag/AgCI reference electrode and platinum auxiliary electrode. Can be equipped with Metrohm Multi-Mode Electrode (MME). Includes PC program 6.6053.010 and comprehensive accessories for setting up the measuring station.

MVA-12

Semi-automatic 797 VA Computrace system for convenient determination of organic additives.

1 x 2.797.0030	797 VA Computrace
3 x 2.800.0010	800 Dosino
1 x 6.3032.250	807 Dosing Unit 50 mL (glass)
2 x 6.3032.120	807 Dosing Unit 2 mL (glass)
1 x 2.843.0040	843 Pump Station (optional)
1 x 2.846.0010	Dosing Interface (optional to connect 4 additional 800 Dosinos)

MVA-13

Fully automatic 797 VA Computrace system for unattended determination of organic additives.

1 x 2.797.0030	797 VA Computrace
1 x 2.838.0310	838 Advanced VA Sample Processor
1 x 2.843.0040	843 Pump Station
3 x 2.800.0010	800 Dosino
1 x 6.3032.250	807 Dosing Unit 50 mL (glass)
3 x 6.3032.120	807 Dosing Unit 2 mL (glass)
1 x 2.846.0010	Dosing Interface (optional to connect 4 additional 800 Dosinos)

ProcessLab – atline process analysis

A wide range of additional modules and components are available. Here is just a small selection:

- ProcessLab measuring amplifier for connecting various sensors
- ProcessLab digital input and output 24 V DC
- ProcessLab analog input and output 4...20 mA
- ProcessLab relay output
- ProcessLab sensor connection
- ProcessLab measuring vessel with stirrer and various measuring vessels
- ProcessLab peristaltic pump with 40 mL/min, 120 mL/min or 320 mL/min
- ProcessLab sampling system with associated sample loops
- ProcessLab overflow pipettes in various sizes
- ProcessLab solenoid valve module to control liquid currents
- Containers in various sizes, with liquid level sensor

Please contact your local representatives for precise dimensions and design of your ProcessLab system. Thanks to its modularity, the instrument can be optimally adapted on-site to meet specific requirements.

ProcessLab contact address
www.metrohm-processlab.com

Online process analysis contact address
www.applikon-analyzers.com

www.metrohm.com

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