

# ESA COULOCHEM III

Clarity Control Module

ENG

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To facilitate the orientation in the **ESA Coulochem III** manual and **Clarity** chromatography station, different fonts are used throughout the manual. Meanings of these fonts are:

Instrument (blue text) marks the name of the window to which the text refers.

Open File (italics) describes the commands and names of fields in **Clarity**, parameters that can be entered into them or a window or dialog name (when you already are in the topic describing the window).

WORK1 (capitals) indicates the name of the file and/or directory.

ACTIVE (capital italics) marks the state of the station or its part.

The bold text is sometimes also used for important parts of the text and the name of the **Clarity** station. Moreover, some sections are written in format other than normal text. These sections are formatted as follows:

 Note:
 Notifies the reader of relevant information.

 Caution:
 Warns the user of possibly dangerous or very important information.

#### Marks the problem statement or trouble question.

Description: Presents more detailed information on the problem, describes its causes, etc.

Solution: Marks the response to the question, presents a procedure how to remove it.

# **1 ESA Coulochem III Control Module**

This manual describes the setting of the **ESA Coulochem III** detector. The control module enables direct control of the instrument over USB.

*Caution:* **ESA Coulochem III** is only supported on 32-bit Windows OS. This is caused by the Motorola drivers for USB port in the detector, which are not available in 64-bit OS version.

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*Caution:* A single **Clarity** station can only control one ESA Coulochem III detector. This is the restriction of communication libraries used to perform the instrument control.



Fig 1: ESA Coulochem III detector

Direct control means that the detector can be completely controlled from the **Clarity** environment. Instrument method controlling the analysis conditions will be saved in the measured chromatograms.

# 2 Requirements

- Clarity Installation CD ROM with LC Control module (p/n A24).
- PC with 32-bit Windows OS (drivers for USB port are not available for 64bit OS).
- Free USB port in the PC.
- USB A-B cable (p/n SK06).

*Note:* Cables are not part of **Clarity** Control Module. It is strongly recommended to order required cables together with the Control Module.

# **3 Installation Procedure**

# 3.1 Hardware - Wiring

The **ESA Coulochem III** detector is controlled by RS232 communication via USB. The detector has also serial port available on the back panel, but this cannot be used for control. The communication is performed via standard USB A-B cable.

# **3.2 Clarity Installation**

The **ESA Coulochem III** detector control module is not a part of the **Clarity** typical installation. During the installation process, the user is eventually asked to choose the Components of the installation - *Typical*, *Full*, *Minimal* or *Custom*. It is either necessary to select the *Full* installation, or a *Custom* one while adding the **ESA Coulochem III** detector from the *Detector* section (check the checkbox before the item) in order to install the **ESA Coulochem III** detector driver.

*Note:* During the installation process, the error message about the hardware drivers not being signed may appear. In such case, choose to install the drivers anyways.

# 3.3 Clarity Configuration

Setup Cont	trol Modules		Number of Ir	nstruments: 2	
AS CLC CC CC CC CC CC CC CC CC C	Used S/N 777111 Instrument 1 Instrument 1		A hatument 1 AA haturent 1 Instrument Type LC Image for Closed Instrum AB C C AB C C C C C C C C C C C C C C C C C C C	ment 2 × Instrument 3 × ment 2 × Instrument 3 × ment Image fr ment Image fr From Coulochem III Coulochem III Coulochem III From Pervice	Instrument 4
Add	About Setup	)	Ext. Start Dig. Input: Ready Dig. Output:	Coulochem III Coulochem III OK Cancel	I I I I I I I I I I I I I I I I I I I
Available Control Modules					
<b>F</b>		Elter	2		
+ -		Filter:	2 Al •	esa	
Name       Image: Solution of the second	Status Installed	Filter: Vendor ESA	Al Comment Bectrochem	esa Module Info	

Fig 2: System Configuration

- Start the **Clarity** station by clicking on the **M** icon on the desktop.
- Invoke the System Configuration dialog accessible from the Clarity window using the System Configuration... command.
- Press the *Add* button ① (see **Fig 2** on pg **4**.) to invoke the Available Control Modules dialog.
- You can specify the searching filter 2 to simplify the finding of the driver.
- Select the **Coulochem III** detector and press the *Add* (3) button.

The ESA Coulochem III Setup dialog will appear.

ESA Coulochem III	Setup				X
Serial Number	777111		. (	Autodetect	
	DC Module I	nstalled e Installed eater Installed	1	Acquisition Mode OC Pulse	
Signal 1					
Name	Detector 1				
Potential Limit	2000	mV			
Quantity:	Current		Units:	μA	
Signal 2					
Name	Detector 2				
Potential Limit	2000	mV			
Quantity:	Current		Units:	μА	
٥	igital Input Nam igital Output Nar	es [	Change Change	]	
	ОК	Cancel	He	p	

Fig 3: ESA Coulochem III Setup

- Press the Autodetect button to find the ESA Coulochem III detector, the installed options will be autodetected and pre-filled. Exit the dialog by pressing the OK button.
- *Note:* The ESA Coulochem III Setup dialog is more closely described in the chapter **"ESA Coulochem III Setup"** on pg **17**.

The **Coulochem III** detector item will appear in the *Setup Control Modules* list of the System Configuration dialog.

- Drag and drop the Coulochem III icon from the Setup Control Modules (4) list on the left side of the System Configuration dialog to the desired Instrument (5) tab on the right side (6) (or use the -> button (7) to do so).
- Set the *Ext. Start Dig. Input* and *Ready Dig. Output* numbers (8) for your system according to the wires being used for synchronization with other devices.

# 4 Using the control module

New Acquisition tabs (one for each detector signal assigned to the Instrument) are created in the Method Setup dialog. They can be accessed by switching to the desired signal in the *Select Detector* section on the top of the dialog.

# 4.1 Method Setup - Acquisition

The Method Setup - Acquisition tab is present in one copy for each signal of the **ESA Coulochem III** detector. The number of signals and the appearance of the tabs is dependent on the mode the detector is switched in - in the DC mode, two DC Mode Method tabs are present, while in the Pulse mode one Pulse Mode Method tab is present. In the right section on all Method Setup - Acquisition sub-tabs for **ESA Coulochem III** detectors two buttons are placed:

#### From Det

Pressing the button loads all detector parameters as they are set in the **ESA Coulochem III** detector. These parameters are set in appropriate fields of the Method Setup - Acquisition dialog.

## Det Status

Hardware Configuration	n 🛛 🛛
USB ID	0
Serial Number	060989129
Instrument Revision	1241526329
Firmware Version	3.12
Installed Modules	- DC module
Acquisition Mode	DC
ОК	Cancel

Fig 4: Hardware Configuration

After pressing the *Det Status* button the Hardware Configuration dialog opens. When the detector is not in demo mode, it displays the internal detector *USB ID*, its *Serial Number*, *Instrument Revision* and *Firmware Version*. Moreover, it shows the installed parts and selected operation mode in the *Installed Modules* and *Acquisition Mode* fields, as set in the ESA Coulochem III Setup dialog. In the demo mode, only the *Installed Modules* and *Acquisition Mode* values are shown, other fields are displayed with the *Demo* inscription.

		tequient		
Method Se	etup Test			
Select Det	ector Detector 1	<b>_</b>	✓ Enabled	
DCI	Node Method	Couloche	em III Detector Method	Det Status
	Cell Potential	50 mV	Common for All Detector	5

•

-

-

Cancel

Event Table Measurement Acquisition Integration Calculation Advanced OK

# 4.1.1 Method Setup - Acquisition - DC Mode Method

Full Scale Gain Range 1 mA

Filter Time Constant 5 s

Analog % Offset 0

Ready

Analog Range 1.0 V

Fig 5: Method Setup - Acquisition - DC Mode Method

Send method

The DC Mode Method sub-tab of the Method Setup - Acquisition dialog is used to specify the detector program when the detector is switched to the DC mode (uses the DC Module card).

Guard Cell Potential

Thermal Organizer

Temperature

Perform Autozero before Run

Report

From Det

Help

Audit Trail

#### Cell Potential

Status

Sets the potential of the measurement cell. The allowed range for the Cell Potential is defined by the value set in the Potential Limit field in the ESA Coulochem III Setup dialog, which delimits the absolute value of the possible Cell Potential.

#### **Full Scale Gain Range**

Sets the signal range of the ESA Coulochem III detector, which should be set above the highest current expected for the analyte of interest over the course of the experiment. It ranges from 10 pA to 1 mA in steps of 1, 2, 5, 10. 20. 50. 100. 200 and 500.

#### Filter Time Constant

Sets the filter on the data gained from the ESA Coulochem III detector. Possible values are none, 0.2 s, 0.5 s, 1.0 s, 2.0 s, 5.0 s and 10.0 s. The higher the filter will be, the smoother the data will be, but at the cost of the signal precision. For very sensitive analyses, the value should be set as low as possible.

Another filter is afterwards applied in Clarity as a part of the Global Peak Note: Width integration function.

## Analog % Offset

Defines the offset (shift of the signal on the y-axis) of the analog signal. Possible values range between -50 and 50 per cent, applied on the *Analog Range* set. Thus, when 1.0 V *Analog Range* is selected, the signal can be shifted by  $\pm 0.5$  V via the *Analog % Offset* function.

## Analog Range

Defines the voltage range that will be set on the analog output on the back of the **ESA Coulochem III** detector. Possible values are -1.0 V, -0.1 V, 0.1 V and 1.0 V.

## **Common for All Detectors**

Fields and items in the following section are common for both signals of the same detector - when both cells from the DC mode are configured in the **Clarity** Instrument, the parameters in question will influence both cells in the same way.

## Sample Rate

Sets the sample rate of the ESA Coulochem III detector.

## **Guard Cell Potential**

Sets the potential on the **Guard Cell**, if it is installed. The field is enabled and editable regardless or the guard cell's actual presence, the possible Potentials range from -2000 to 2000 mV.

## **Thermal Organizer**

Sets the Thermal Organizer Module power on or off, if it is installed. Furthermore, allows to set the temperature that should be maintained in the organizer.

## Perform Autozero Before Run

When checked, performs the autozero operation on the beginning of the analysis run. Both detectors are zeroed this way.

*Note:* Autozero operation takes a lot of time to perform - around half a minute or so. It is not advisable to use the *Perform Autozero Before Run* checkbox in shorter analyses, and the integration interval should be adjusted accordingly.

# 4.1.2 Method Setup - Acquisition - Pulse Mode Method

Me	thod Setup Test					3
9	ielect Detector De	tector 1	•	Enabled		
			Couloche	m III Detector Method	Det Status	
	Pulse Mode Method	3				
	E1 Potential	200	m∀	Full Scale Gain Range	1 mC 💌	
	E1 Pulse Width	500	ms	Filter Time Constant	none 💌	
	Acquisition Delay	300	ms	Sample Rate	10 Hz 💌	
	E2 Potential	-1000	m∀	Analog % Offset	0 %	
	E2 Pulse Width	10	ms	Analog Range	1.0 V 💌	
	E3 Potential	600	m∀	Thermal Organizer	Power On	
	E3 Pulse Width	1	ms	Temperature	•c	
	E4 Potential	-100	mV		,	
	E4 Pulse Width	10	ms	🗌 Perform Autozero	before Run	
	Status F	?eady			From Det	
	Event Table Measure	ment Acqui	isition Integration	Calculation Advanced		
		OK	K Cano	el Send method Ro	eport Audit Trail Help	

Fig 6: Method Setup - Acquisition - Pulse Mode Method

## E1 Potential

Defines the measurement potential.

## E1 Pulse Width

Sets the time for which the measurement potential will be maintained.

## **Acquisition Delay**

Sets the time that will be used to establish the correct value of *E1 Potential*, before the measurement itself starts. The time difference between *E1 Pulse Width* and *Acquisition Delay* values is the measurement time.

## E2 (..E4) Potential

Sets the potential value for the *E2 Pulse Width* (*E3 Pulse Width*, *E4 Pulse Width*) time interval. The field is only informative as the value can not be sent to the detector (limitation of the detector firmware).

## E2 (..E4) Pulse Width

Defines the duration of the interval for which the *E2 Potential* (*E3 Potential*, *E4 Potential*) will be set. The field is only informative as the value can not be sent to the detector (limitation of the detector firmware). Whole measurement procedure performed during the Pulse mode is described by the **Fig 7** on pg **11**.

*Note:* The potentials set during the T2, T3 and T4 time intervals are used to renew the surface of the electrodes. The *E4 Potential* and *E4 Pulse Width* stages are not described on the **Fig 7** on pg **11**.



Fig 7: Pulse mode

#### Full Scale Gain Range

Sets the signal range of the **ESA Coulochem III** detector, which should be set above the highest charge expected for the analyte of interest over the course of the experiment. It ranges from  $10 \ pC$  to  $10 \ mC$  in steps of 1, 2, 5, 10, 20, 50, 100, 200 and 500.

#### **Filter Time Constant**

Sets the filter on the data gained from the **ESA Coulochem III** detector. Possible values are *none*, *low*, *medium* and *high*. The higher the filter will be, the smoother the data will be, but at the cost of the signal precision. For very sensitive analyses, the value should be set as low as possible.

Note:

Another filter is afterwards applied in **Clarity** as a part of the *Global Peak Width* integration function.

#### Sample Rate

Sets the sample rate of the **ESA Coulochem III** detector. When the sample rate is greater than the rate of changing pulses, the data will still be given, although the **ESA Coulochem III** detector will interpolate them from the real pulse data.

## Analog % Offset

Defines the offset (shift of the signal on the y-axis) of the analog signal. Possible values range between -50 and 50 per cent, applied on the *Analog Range* set. Thus, when 1.0 V *Analog Range* is selected, the signal can be shifted by  $\pm 0.5$  V via the *Analog % Offset* function.

#### Analog Range

Defines the voltage range that will be set on the analog output on the back of the **ESA Coulochem III** detector. Possible values are -1.0 V, -0.1 V, 0.1 V and 1.0 V.

## **Thermal Organizer**

Sets the Thermal Organizer Module power on or off, if it is installed. Furthermore, allows to set the temperature that should be maintained in the organizer.

#### Perform Autozero Before Run

When checked, performs the autozero operation on the beginning of the analysis run.

*Note:* Autozero operation takes a lot of time to perform - around half a minute or so. It is not advisable to use the *Perform Autozero Before Run* checkbox in shorter analyses, and the integration interval should be adjusted accordingly.

# 4.2 Method Setup - Event Table

Name Input Output									
	Name	Туре	Source	Input	Value	Units	Output Type	Output	Parame
1	Peak Start	Input Run >	Detector 1		100,000	μA	Coulochem III	Digital Output 1	High
2	Peak End	Input Run <	Detector 1		70,000	μA	Coulochem III	Digital Output 1	Low
3	Pump Error	Dig. Input Run	Coulochem III	CELL ON/OFF	Up		Coulochem III	Digital Output 2	Pulse
4	1	B							

Fig 8: Method Setup - Event Table

Both the inputs and outputs of the **ESA Coulochem III** detector can be used in the **Event Table**, either as the input conditions or for the output reaction.

# 4.3 Method Setup - Advanced

Method Setup Test
Common for all detectors
Subtraction Chromatogram [None] Matching No Change Set None
Column Calculations Unretained Time 0 [min.] Column Length 50 [mm] C Statistical Moments C From Width at 50%
Auxoliary Signal Store 1 Temperature Coulochem III 🔽
Event Table         Measurement         Acquisition         Integration         Calculation         Advanced           OK         Cancel         Send method         Report         Audit Trail         Help

Fig 9: Method Setup - Advanced

When the Organizer Heater compartment of the **ESA Coulochem III** detector is installed, it is possible to use the *Temperature* auxiliary signal. The signal can be stored into the measured chromatograms using the checkbox on the Method Setup - Advanced tab.

# 4.4 Device Monitor

The window with the detector status can be invoked by the *Monitor* - *Device Monitor* command from the Instrument window or using the OLC *Monitor* icon. It displays the actual signal belonging to all signals configured on the Instrument, as well as other information such as the detector temperatures. Each detector has its own section in the Device Monitor window.

ᅇ Instrument 1	- Device Monitor		
Eile Control View	v <u>W</u> indow <u>H</u> elp	<b>6 6 T B</b> 9	3 0 7 7 6 0
🔽 Coulochem III (0	60989129)		Ready 🔘
Zero Detector Set Cells Off	Thermal Organizer Not installed		Det Status
Input Current no. State:	Descriptions:	Output Current no. State:	Descriptions:
1 🔿	CELL ON/OFF	1 🔾 🖊	Digital Output 1
2 🔿	AUTOZERO	2 💽 🖊 L	Digital Output 2
з 📀	START	3 💽 🖊 L	Digital Output 3
4		4 💽 🖊 L	Digital Output 4
5		5 💽 🖊 L	Digital Output 5
For Help, press F1			

Fig 10: Device Monitor

The detector can be partially controlled from the **Device Monitor** window. Available control commands and fields are:

#### **Zero Detector**

This button performs the autozero operation - the output signal of the detector is set to zero. If the detector provides more than one signal, all of them will be zeroed. The button is not available during the analysis run.

#### Set Cells Off/On

Invoking this button shuts the detection cells off or on. The button name changes according to the actual state of the detector cells. It is only possible to turn the cells off during the analysis run, then the button gets disabled until the analysis is finished. If the detector provides more than one signal, all of them will be turned off/on.

Note:

It is not possible to start the analysis when the detection cells are off, unless the start is triggered by external signal.

## **Thermal Organizer**

Shows the status of the Thermal Organizer, e.g. if it is installed, powered on or off and what is the temperature in it.

#### Det Status

Using the button opens the Hardware Configuration dialog for ESA Coulochem III detector more closely described the chapter "Method Setup - Acquisition" on pg 7.

## Digital inputs and Digital Outputs section

This section allows monitoring the state of the **ESA Coulochem III** detector inputs and monitoring and control of the **ESA Coulochem III** detector digital outputs.

# 4.5 ESA Coulochem III Setup

ESA Coulochem III	Setup		×
Serial Number	777111	Auto	odetect
	DC Module Installed     Pulse Module Installed     Organizer Heater Installed	Acqu	DC     Pulse
Signal 1 Name	Detector 1		
Potential Limit	2000 mV		
Quantity:	Current	Units:	μA
Signal 2 Name	Detector 2		
Potential Limit	2000 mV		
Quantity:	Current	Units:	μA
	Digital Input Names	Change Change	
(	OK Cancel	Help	

Fig 11: ESA Coulochem III Setup

## Serial Number

Shows the autodetected serial number of the **ESA Coulochem III** detector. In case more than one detector is connected to the same computer, the field allows to select which one will be used in configuration.

#### Autodetect

This button is used to test the connection to the **ESA Coulochem III** detector. When the detector is connected, the *Serial Number* field is filled with the autodetected serial number of the detector.

#### DC Module Installed

Enables the use of the installed DC card. When this card is present, the *DC Acquisition Mode* can be selected.

#### **Pulse Module Installed**

Enables the use of the installed Pulse Mode card. When this card is present, the *Pulse Acquisition Mode* can be selected.

#### Organizer Heater Installed

Enables the use of the organizer heater. Checking the option adds a temperature auxiliary signal into the Method Setup - Advanced tab and activates the *Thermal Organizer* section on the Method Setup - Acquisition tab for the **ESA Coulochem III** detector.

#### **Acquisition Mode**

Allows to select the operation mode in case both the *DC Module* and *Pulse Module* are installed.

#### DC

Standard type of operation for the *DC Module* - detector gives two signals of actual Current measured in  $\mu$ A.

#### Pulse

Standard type of operation for the *Pulse Module* - detector gives only one signal of the Charge measured in  $\mu$ C.

*Note:* When both communication cards are present in the **ESA Coulochem III** detector and the *Pulse* mode is selected, it is not possible to read the method from the detector without sending it to the detector first after the start of the detector.

#### Signal 1 (2)

Fields belonging to particular detectors enable to set the relevant detector names and potential limit for each signal. In case the *Pulse* option is selected in the *Acquisition Mode* section, only the Signal 1 is available. Each signal also has the *Quantity* and *Units* values, which are not modifiable. These show the quantity measured by the detector and units in which the signal is gained.

#### Name

Allows to change the name for the particular signal. The name is then displayed in the System Configuration dialog and in the Select Detector section of the Method Setup - Acquisition dialog.

#### **Potential Limit**

Sets the potential limit for the particular signal. When changing the potential used later in the Method Setup - Acquisition dialog, the user is limited by the  $\pm$  *Potential Limit* value. Possible values for the *Potential Limit* range between 0 and 2000 mV.

#### **Digital Input Names**

Using the *Change* button displays the Digital Input Names dialog which allows to set the names for events mapped on the physical inputs of the detector. The meaning of the inputs, however, cannot be changed.

Digital Input Na	mes	X
Input no.	Descriptions:	
1	CELL ON/OFF	]
2	AUTOZERO	]
3	START	
ок	Cancel Help	]

Fig 12: Digital Input Names

## **Digital Output Names**

Using the *Change* button displays the Digital Output Names dialog which allows to set the names for events mapped on the physical outputs of the detector.

Digital Output N	lames	×
Output no.	Descriptions:	
1	Digital Output 1	
2	Digital Output 2	
3	Digital Output 3	
4	Digital Output 4	
5	Digital Output 5	
ОК	Cancel Help	

Fig 13: Digital Output Names

# 4.6 Digital Outputs Control

The **ESA Coulochem III** detector contains five digital TTL outputs which are also designed as relay contacts.

Digital Outputs of Coulochem III (SN:060989129)				
Output no.	Initial State:	Current State:	Descriptions:	
1	0/L	0/L	Digital Output 1	
2	0/L	0/L	Digital Output 2	1
3	0/L	<b>0</b> /L	Digital Output 3	
4	0/L	0/L	Digital Output 4	
5	0/L	0/L	Digital Output 5	
			Close Help	

Fig 14: Digital Output Control

This dialog allows to change the *Initial* and *Actual* state of the digital outputs of the **ESA Coulochem III** detector. The digital outputs may be also controlled from the Method Setup - Event Table tab.

# **5 Report Setup**

All of the detector settings accessible on the Method Setup - Acquisition tab for the given signal are reported. To do so, the *Instrument Control* parameter on the Method tab of the Report Setup dialog must be checked.

Print Pre	eview								
Print	Next Page	Pre <u>v</u> Page	<u>T</u> wo Page	Zoom In	Zoom Out	⊆lose	Print to PDE	Send PDF	
Mode Therma	l Organizer	: DC : Notinstalled		<i>Coul</i> Guard Potenti	oche <i>m III M</i> etho al : 0 mi	d Common part V			
Cell Pot Analog '	ential % Offset	:50 mV :0%		Co Filter Time Cor Analog Output	whochew///Anleth istant :5s Range :1.0	od Detector 1 V			
Cell Pot Analog	ential % Offset	:50 mV :0%		Co Filter Time Cor Analog Output	<i>ukochem///Meth</i> istant :5s Range :1.0	od Detector 2 V			
Page 1									

Fig 15: Report Setup

# **6** Troubleshooting

Clarity method cannot be sent to the ESA Coulochem III detector, the analysis can be run after opening the Clarity Instrument.						
Description:	The detector equilibrates itself after the start, most probably. The equilibration takes approximately three minutes and during that time the method cannot be sent to the detector nor the acquisition cannot be started from <b>Clarity</b> .					
Solution:	Wait until the countdown on the detector display reaches 0 before opening <b>Clarity</b> .					
The signal analysis, an	n the Data Acquisition window cannot be seen at the beginning of the alysis time is not running.					
Description:	Most probably the Perform Autozero Before Run function was used. The use of					

Description: Most probably, the Perform Autozero Before Run function was used. The use of the function stops receiving data for approximately 30s.

Solution: If the data from the start of the chromatogram are needed, switch the autozero function off. As a workaround, you can create an autozero method that will only perform autozero in the dummy run, preceding the real one.