

## CTC PAL

Clarity Control Module

ENG

Code/Rev.: M063/80C Date: 2024-02-14

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

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.

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

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

Chromatogram (blue underlined) marks clickable links referring to related chapters.

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 CTC PAL autosamplers**

This manual describes the setting of the CTC LC PAL, CTC GC PAL, CTC Combi PAL and the CTC PAL-xt family (LC-xt, HTC-xt, HTS-xt, HTX-xt, GC-xt, Combixt, Combi-xt extended) autosamplers. The control module enables direct control of the instrument over serial line.



Fig. 1: CTC PAL autosampler

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

*Note:* Sample preparation conditions are all the options from the **Method Setup - Basic/Advanced** dialogs.

# 2 Requirements

## 2.1 Software requirements

• Clarity (p/n C50) Installation CD ROM with AS control module (p/n A26).

**CTC PAL** requires **Microsoft .NET version 4** for operation. This version (or later) is already installed on majority of PCs. Nonetheless you will be notified during installation if your PC is missing **Microsoft .NET**.

Microsoft .NET requires following attributes of your operating system:

- Windows XP SP3 or later (preferably)
- Windows Installer 3.1 and later installed in your OS.

For complete list of .NET requirements, see the .NET Framework System Requirements on Microsoft web page.

*Caution:* Before installing **Clarity**, make sure that your **Windows** are updated to the latest version.

### 2.2 Hardware requirements

- Serial cross RS232-compliant DB9F-DB9F cable (p/n SK01) or LAN cable (UTP with RJ45 connectors).
- *Note:* Cables are not part of **Clarity** Control Module. It is strongly recommended to order required cables together with the Control Module.
  - Free serial port in the PC (fast 16550 UART) or computer connected to the same LAN as the autosampler.
- *Note:* Modern computers usually have only 1 (if any) serial (COM) port installed. To use more devices requiring the port, the **MultiCOM** adapter (p/n MC01) is available.

## **3 Installation Procedure**

## 3.1 Installation of Clarity Station

**CTC PAL** autosampler is not included in **Typical** installation configuration of the **Clarity** station.

Choose **Custom** configuration during installation and leave all the options unaltered (unless you want to make some other adjustments to the installation).

*Note:* If you have **Clarity** station already installed, run the installation once again according to the following steps. This will alter neither your configuration nor data files.

## 3.2 Installation procedure

### 3.2.1 Connections

The autosampler communicates with PC using straight full RS232-compliant DB9F-DB9M cable. Correct COM port settings on the PC side are **9600**, **n**, **8**, **1** (should be set automatically during configuration of the sampler).

Other possible connection can be made using LAN (Ethernet) network, it this case both computer and AS should be connected in the same subnetwork.

Besides the communication cable, the autosampler must be synchronized with the detector (e.g. INT7 card). This is achieved by means of connecting the **Start Output** of the autosampler (DB15F connector labelled INTERFACE) connected to the detector digital input configured as **Start Digital Input** in **Clarity** station. The pin connection is described in the following table:



Fig. 2: Autosampler IN/OUT connector

Signal Name	15 PIN Connector	25 PIN Connector
SW-Out1 NO	3	9
SW-Out1 COM	4	22
TTL-In1	7	1
GND	8	14-19

Tab. 1: CTC PAL pin connections

*Note:* Refer to the CTC manual for description of cables supplied with the autosampler. Cables are not part of **Clarity** Control Module. It is strongly recommended to order required cables (p/n SC02) together with the Control Module.

### 3.2.2 Autosampler Setup

Specific pins must be configured on both the CTC PAL sampler and **Clarity** station for synchronization of the sampler and pumps to work correctly.

• Use Control Terminal on the sampler body, navigate to Menu - Setup - Objects and choose values according to the following table:

Tab. 2: CTC PAL configuration of pins

Submenu	Parameter	Value
Out Signals - Injected	Destination	SW-Out1
	Pulse Time	1.0 s
Sync Signals - Start	Source	Immediate

## 3.3 Clarity Configuration

- In the *System Configuration* dialog press the *Add* (① on System Configuration picture) button to invoke the *Available Control Modules* dialog.
- Select the CTC PAL autosampler and press the Add 2 button.
- The CTC PAL Setup dialog will appear.

CTC PAL Setup	×
Sampler Name:	Sampler 1
COM Port:	COM1 ~
O Host/IP:	
Installed Syringe:	1.2ul V Detect
	Headspace
0	K Cancel Help

Fig. 3: CTC PAL Setup

• Choose the desired type of connection (COM or Host/IP), then fill in the *COM Port* or *Host/IP* and other autosampler options and press the *OK* button. The maximum number of COM port supported by CTC driver is COM8.

*Note:* Other fields from this dialog are described later in the manual (for more details see the chapter **CTC PAL Setup** on pg. **12**).

The CTC PAL Sampler will appear in the *Setup Control Modules* ③ list of the *System Configuration* dialog.

 Drag the sampler icon from the Setup Control Modules list on the left side to the desired Instrument tab on the right side () (or use the -> button () to do so).

System Configuration				×
Setup Control Mod	lules	Number	of Instruments: 4	
Name Used	S/N 3 ment 1 3469 ment 1	Instrument 1 Instrument 1 Instrument 1 Instrument Type LC Name Sampler 1 C Detector M. colindc Thermostat Valve Fraction Col Auxiliary	Instrument 2 Instrument 3 Instrument 2 Instrument 3 Instrument 3 Instrument 3 Instrument 3 Instrument 3 Instrument 5 Instr	ant 4
1 Add Remove About	Seup	Data Inputs & Output Ext. Start Dig. Input: Ready Dig. Output: Miscellaneous Setting Units Setu	Is Device Colibrick Colibridk p Method Option OK Cancel	Number 1 × 1 × s Help
Available Control Modules			– 🗆 X	
	Installed Only Filter:	: All V CTC	0	
Name	Status	Vendor	Comment Modul	
E 🐼 AS	installed	CTC	Combi PAL, Combi-xt PAL, Combi	
PAL3	installed	CTC	CTC PAL3 autosamplers controlled Testi	
ALOUS LC GC C Detector Balance C CT 100 C Valve II Fraction Collector C apillary Electrophoresi Auxiliary	installed	Watex	Column thermostat by UNI Ruby s	
			>	
Add Cancel			Help	

Fig. 4: System Configuration

Set the *Start* and *Ready* input and output numbers for your acquisition card according to the wires used for synchronization.

# 4 Using the control module

New <u>Method Setup - AS</u> tab appears in the *Method Setup* dialog, enabling the setting of the AS control method.

## 4.1 Hardware Configuration

The *Hardware Configuration* dialog can be accessed using the *AS Status* button on the *AS* tab of the *Method Setup* dialog. In the full version, this dialog displays automatically detected communication port, selected parameters of the sampler and detected tray configuration.

Hard	Hardware Configuration						
Fir	mware Version:	<unknown></unknown>	>				
	Device Mode:	PAL					
	Driver Version:	1.6.0.1					
	Connection:	Demo					
Trav	Configuration:						
No.	Tray Name	Туре	Min. Vial	Max. Vial			
1	Tray01	DW96	101	196			
2	Tray02	MT96	201	296			
3	Tray03	VT98	301	398			
4	Tray04	VT54	401	454			
	<select td="" to<="" type=""><td></td><td></td><td></td><td></td></select>						
			OK	Cancel			
				Contect			

Fig. 5: Hardware Configuration (DEMO)

The *Tray Configuration* table is particularly important as it shows the mapping of vial numbers in **Clarity Sequence** table to physical vials on trays. Vials on CTC samplers are addressed by [tray name, vial] pairs, while **Clarity** sequence uses single vial number only. *Min. Vial* and *Max. Vial* columns thus display vial number ranges assigned to particular trays on the autosampler.

*Note:* In the **Clarity DEMO** version various tray configurations can be simulated by choosing number of trays and their types.

### 4.2 Method Setup - AS

On the *Method Setup - AS - Parameters* sub-tab injection method parameters can be viewed, while the *Method Setup - AS - Description* sub-tab contains text description of the method.

The *Edit Method* button allows the changing of the parameters in the injection method, while the *AS Status* button displays the <u>Hardware Configuration</u> dialog. Both buttons are common to both the *Parameters* and *Description* sub-tabs of the *Method Setup - AS* tab.

Method	Setup test			_		×
New	Open Save Save as Report setup	Audit trail S	iend method by e-mail			
Select Sa	ampler Sampler 1	V 🗹 Ena	bled			
	CTC F	AL Method				
	Parameters Description					
	Svringe	2.5ml-HS	•			
	Injection Program	LC-Inj				
	Air Volume (ul)	20				
	Pre Clean with Solvent 1 ()	0				
	Pre Clean with Solvent 2 ()	0				
	Pre Clean with Sample ()	0				
	Filling Speed (µl/s)	50				
	Filing Strokes ()	1				
	Inject to	LC VIv1				
	Injection Speed (ul/s)	100				
	Pre Inject Delay (ms)	500				
	Post Inject Delay (ms)	500				
	Post Clean with Solvent 1 ()	0				
	Post Clean with Solvent 2 ()	0	Edit Method			
	Valve Clean with Solvent 1 ()	0				
	Valve Clean with Solvent 2 ()	0	•			
	Demo Mode: Ready					
Event 1	Table AS Measurement Acquisition Integr	ration Calculation A	lvanced			
	IK Cancel				Send Me	thod
41	dancer					

Fig. 6: Method Setup - AS - Parameters

#### **Injection Program**

Injection program (also referred to as Cycle in CTC manuals) used by the method. Additional method parameters depend on the injection program selected -"Overview of parameters used in injection programs" on page 12 for the table of adjustable parameters specific for particular injection programs.

CTC PAL sampler control from **Clarity** station comes with eight predefined injection programs:

- GC-Inj Standard GC Injection.
- LC-Inj Standard HPLC Injection.
- **HS- Inj** Standard Headspace Injection. When using this program, *Headspace* checkbox (in <u>CTC PAL Setup</u>) needs to be turned on. Also *GC Run Time* and *Cool Down Time* values need to correspond the real time of the analysis.

- **SPME** Heated Solid phase microextraction with heating.
- **SPME Agitated & Heated** Solid phase microextraction with heating and agitation.
- SPME Agitated & Heated- Fiber Conditioning Solid phase microextraction with heating, agitation and fiber conditioning between injections.
- **SPME Agitated & Heated Special** Same as SPME Agitated Heated, but with individual needle penetration and fiber exposure values.

*Note:* Take care that the fiber is not damaged by setting the parameters too high.

• SPME Out of Tray - SPME directly from the vial on tray.

For information on how to use other injection programs or how to create your own ones, "Custom Injection Programs" on page 16

#### Syringe

Syringe volume this method is designed for. The value set must match the syringe volume configured in <u>CTC PAL Setup</u> dialog and the syringe mounted on the autosampler, otherwise the method will not run.

Valid values: 10, 25, 100, 250 µl; 1, 2.5, 5 ml-HS; Fiber.

#### Additional method parameters

Please note that "." (dot) decimal separator should be used even in case your local settings are different.

#### Air Volume (µl)

Air volume aspirated after the syringe needle is moved out of the sample liquid.

Valid range: 0 - Syringe Maximum

#### Pre Clean with Solvent 1/2

Number of pre-injection syringe washes using wash solvent 1 / 2.

Valid range: 0 - 99

Pre Clean with Sample

Number of pre-injection syringe washes using sample. Valid range: 0 - 99

#### Fill Speed (µl/s)

Speed of sample aspiration.

Valid range: depends on the syringe, usually 10 - 500 µl/s

#### Filling Strokes

Number of filling strokes to aspirate sample

Valid range: 0 - 99

#### Injection Speed (μl/s) Speed of sample injection. Valid range: depends on the syringe, usually 10 - 500 μl/s

#### CTC PAL

#### Pullup Delay (ms)

Delay time before the sample is pulled into the syringe.

Valid range: 0 - 10000 ms

#### Pre / Post Inject Delay (ms)

Delay time prior / post sample injection.

Valid range: 0 - 99000 ms

#### Inject to

Name of the injector used for the injection.

Valid range: Choose from the list

#### Post Clean with Solvent 1/2

Number of post-injection syringe washes using the wash solvent 1 / 2. Valid range: 0 - 99

#### Valve Clean with Solvent 1/2

Number of valve rinse washes using the wash solvent 1 / 2.

Valid range: 0 - 99

#### Incubation Temperature (°C)

Sample incubation temperature.

Valid range: 30 - 200 °C

Incubation Time (s) Sample incubation time. Valid range: 30 - 86400 s

Syringe Temperature (°C) Syringe temperature. Valid range: *30 - 150* °C

#### (Pre Inc) Agitator Speed (rpm)

Speed of the sample agitation during incubation (prior to incubation). Valid range: 250 - 750 rpm

#### Flush Time (s)

The time during which the syringe is flushed with intert gas.

Valid range: 0 - 600 s

#### GC Runtime (s)

Duration of GC method. Used for calculation of sample incubation in headspace mode.

Valid range: 30 - 86400 s

#### Pre Incubation Time (s)

Time to wait until next vial can be inserted into agitator.

Valid range: 10 - 6000 s

#### Agitator On Time (s)

Time to start the agitation.

Valid range: 0 - 60 s

#### Agitator Off Time (s)

Time to stop the agitation. Valid range: 0 - 60 s

#### Vial Penetration in mm (µl)

Penetration of the needle to the vial using SPME programs.

Valid range: 22 - 31 mm

#### Vial Needle Penetration (mm)

Penetration of the needle to the vial using SPME Agitated & Heated - Special program.

Valid range: 7 - 43 mm

#### Vial Fiber Exposure in mm (µl)

Length of the fiber exposed in the vial.

Valid range: 10 - 25 mm

#### Injection Penetration in mm (µI)

Penetration of the needle to the injector using SPME programs.

Valid range: 44 - 57 mm

#### Injection Needle Penetration (mm)

Penetration of the needle to the injector using SPME Agitated & Heated - Special program.

Valid range: 20 - 43 mm

#### Injection Fiber Exposure in mm (µI)

Length of the fiber exposed in injector.

Valid range: 10 - 25 mm

#### **Extraction Time**

Duration of the extraction.

Valid range: 10 - 6000 s

#### Desorb to

Name of the injector used for desorbtion.

Valid range: Choose from the list

#### Desorbtion Time (s)

Duration of the desorbtion.

Valid range: 10 - 86400 s

#### Post FibCondTime (s)

Time of the fiber conditioning in the Fiber Conditioning Station between injections. Valid range: 0 - 10000 s

## 4.3 CTC PAL Setup

The CTC PAL Setup dialog sets the fundamental options of the control module.

*Note:* It is accessible from the *System Configuration* dialog by doubleclicking the *CTC PAL* item.

CTC PAL Setup	×
Sampler Name:	Sampler 1
COM Port:	COM1 ~
O Host/IP:	
Installed Syringe:	1.2ul V Detect
	Headspace
	K Cancel Help

Fig. 7: CTC PAL Setup

#### Sampler Name

Assigns name to the sampler, under which it appears in configuration, method setup, reports, etc.

#### COM Port

Sets the COM Port used for communication between the sampler and the PC.

*Note:* See chapter **3.1** for detailed description of the communication.

#### Host/IP

Sets the IP address of the AS when controlled via LAN network.

#### Installed Syringe

This version of CTC PAL control module cannot autodetect mounted syringe, its volume must be filled in manually. You may either choose value from the list or fill correct syringe volume manually. The number entered is an arbitrary value, most common volumes may be chosen from the drop-down list. When given syringe is not supported by the sampler, it will respond with "Syringe detection failed" message after closing the dialog.

#### Headspace

Turn on/off sample overlapping (preparing the new sample during the analysis of the previous one), when using headspace injections. Turn on only when using **HS-Inj** injection cycle, other cycles cannot work properly with overlapping.

*Note:* Setting the right syringe volume is crucial for correct operation of the sampler.

## 4.4 Overview of parameters used in injection programs

In the following table there is the list of parameters used in injection programs:

Parameter	LC	GC	HS	SPME Htd.	SPME Ag.+ Htd.	SPME FC.	SPME Spec.	SPME Out T.
Air Volume	×	×						
Pre Clean with Solvent	×	×						
Pre Clean with Sample	×	×						
Fill Speed	×	×	×					
Filling Strokes	×	×	×					
Injection Speed	×	×	×					
Pullup Delay			×					
Pre / Post Inject Delay	×	×	×					
Inject to	×	×	×					
Post Clean with Solvent	×	×						
Valve Clean with Solvent	×							
Pre Incubation Time			×	×	×	×	×	
Incubation Temperature			×	×	×	×	×	
Incubation Time			×					
Syringe Temperature			×					
(Pre Inc) Agitator Speed			×		×	×	×	
Flush Time			×					
GC Runtime			×	×	×	×	×	
Agitator On Time					×	×	×	
Agitator Off Time					×	×	×	
Vial Penetration				×	×	×		×
Vial Needle Penetration							×	
Vial Fiber Exposure							×	
Injection Fiber							×	
Extraction Time				×	¥	×	×	×
				Ŷ	× ×	Ŷ	× ×	Ŷ
Injection Depotration				Ŷ	× ×	Ŷ	0	Ŷ
injection Feneriation				^	^	^		^

CTC PAL		4 Using the	e control m	odule		
Injection Needle Penetration				×		
Desorbtion Time	×	×	×	×	×	
Post FibCondTime			×			

## **5** Sequence

To use the autosampler in the Clarity sequence, following conditions must be met:

- The sequence must be saved
- The sequence must be set as Active
- All methods used in the sequence must have the *External Start* enabled and the *Autostop* time set
- Method Setup AS dialog in all of the methods used must be filled

The required values for starting vial (SV), ending vial (EV), number of injections from vial (I/V) and injected volume are set in the sequence table.

#### Standard sequence operation

After the sequence start, the AS control method will be sent to the sampler and the datastation will wait for the injection signal from the sampler. After receiving it, the run starts. When the run is finished, the method for next injection will be sent to the sampler.

*Note:* Autosampler control is not available during Single Run operation.

# 6 Advanced

## 6.1 Custom Injection Programs

If you have already controlled the sampler from PC using some other software, you can use your own injection programs. To do so:

- 1. Close Clarity station.
- 2. Run your other controlling software.
- 3. Find the setting called e.g. Instrument Data Location in its options, which specifies where the injection programs reside on your hard drive. Optionally, you may search your hard drive for files with .CYX suffix.
- 4. Copy all those .CYX files into folders **CLARITY** INSTALL FOLDER\CTC\_ SDK\INSTRUMENTDATAX, where X can be 1, 2. etc., depending on how many samplers you have connected.
- 5. Re-run **Clarity** station, injection programs should be available in *CTC Method Editor* dialog.

In case you need to create your own injection programs, you need to purchase Cycle Editor software from your CTC vendor.

## 7 Troubleshooting

In case there is no communication with the sampler on serial line without any apparent reason, re-activating the com port in the hand-held controller may help.

When the cause of the problem could not be discovered easily, the recording of communication between **Clarity** and the detector can significantly help the **DataApex** support to discover the cause.

The recording can be enabled by adding or amending the COMMDRV.INI file in the **Clarity** installation directory (C:\CLARITY\CFG by default). The file can be edited in any text editor (e.g. **Notepad**).

Following lines should be added (COM-connected AS):

[COM1] echo=on textmode=on filename=CommDrvCOM1\_%D.txt reset=off

For LAN-connected AS use:

[TCP\_IP address:port] echo=on textmode=on filename=CommDrvTCP\_%D.txt reset=off

Then you should try to invoke the error once more. The created LOG files will greatly help in diagnosis of unrecognised errors and problems in communication.

- *Note:* %*D* (or %*d*) in the filename parameter means that the log will be created separately for each day. The *reset=off* parameter disables deleting the content of the log each time the station is started during the same day.
- *Note:* It is not recommended to record the communication related to instrument control for prolonged periods.

*Note:* In case the autosampler cannot be added in **Clarity** *System Configuration* window, it is recommended to reinstall the **Clarity** to a new (empty) folder.

#### Overlap mode limitations:

Certain limitations are imposed when using Overlap mode (**HS-Inj** injection cycle):

Violation of these limitations can lead to interruption of the sequence.

- When the AS is synchronized via *Ready Input*, it is necessary to ensure that other devices on *Instrument* will be in *Ready* state within 6 seconds after sample preparation is finished. Otherwise, the autosampler will abort its internal sequence, Clarity will produce "*Unexpected device reset*" message and abort its sequence too.
- Second (and following) samples in *Sequence Table* cannot use preparation time longer that the first sample in the table (which was used for optimization calculations).
- The maximal number of rows in the *Sequence Table* should not be higher than 20.

Violation of these limitations can lead to the interruption of the sequence.

## 7.1 Specific Problems

An error message "011" appears after successful autodetect on the autosampler. This error is accompanied by a beeping sound of the CTC PAL autosampler.

Solution: Make sure that the autosampler has setup wash. If that is not the case, create "Wash" station and name it "Wash1". Once configured, after the new initialization the error message should disappear.

In case your installation has frozen, it has been most probably caused by the fact that .NET Framework (dotNetFx40\_Full\_x86\_x64.exe as displayed by the picture below) has failed to install.



Solution:

1) Using Windows Task Manager, end the installation process.

2) Turn OFF Windows automatic updates and leave it turned OFF for the time being of the installation.

3) Start Clarity installation again.

4) After Clarity has been successfully installed, turn Windows automatic updates back ON.