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1. Introduction

1.1 Notices

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Please note that the material contained in this document is subject to being changed, without notice, in future editions. Markes International shall not be liable for errors or for incidental or consequential damages in connection with the supply, use or performance of this document or of any information contained herein, unless a separate agreement between Markes International and the user should take precedence.

1.2 Warranty

The CIA Advantage-xr is designed for laboratory use only. It is not intended for use in domestic establishments or establishments directly connected to a low-voltage power supply network which supplies buildings used for domestic purposes. Where equipment is used in a field placement environment, care must be taken to ensure that the instrument is not exposed to detrimental conditions, i.e. rain, wind, or sun. Exposure may diminish the performance, cause damage to the instrument and/or cause the equipment to become unsafe to the user.

If the equipment is not used in a way specified by the manufacturer, the protection provided by the equipment may be reduced. System failures arising from such use may not be covered in standard warranty and service contract documents.

1.3 Regulatory Certifications

The instrument is designed and manufactured under a quality system registered to ISO 9001.

The instrument complies with the essential requirements of the following applicable European Directives, and carries the CE mark accordingly:

- Low voltage Directive 2014/35/EU
- EMC Directive 2014/30/EU
- ROHS Directive 2011/65/EU

The instrument conforms to the following product safety standards:

- IEC 61010-1/EN 61010-1
- Canada: CSA C22.2 No.61010-1
- USA: ANSI/UL 61010-1.

The instrument conforms to the following regulation on electromagnetic compatibility (EMC):

- IEC 61326-1/EN 61326-1.
1.4 Important Safety Warnings

Make sure you follow the precautionary notices presented in this manual. Safety and other special notices appear in boxes and include the following.

**WARNING** This is the general warning safety symbol and safety alert word to prevent actions that could cause personal injury.

**CAUTION** Highlights actions that may cause instrument damage. We use it to highlight information necessary to prevent damage to hardware, software, invalid test results, or to information that is critical for optimal system performance.

**NOTE** Emphasizes important information about a specific task.

1.4.1 Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Markes International Ltd. assumes no liability for the failure to comply with these requirements.

The **CAUTION – HIGH VOLTAGE** symbol indicates a mains voltage hazard.

The **CAUTION – HOT SURFACE** symbol indicates a burn hazard.

The **LIFTING HAZARD** symbol indicates that physical injury may occur if the correct lifting procedure for the instrument is not followed.

1.4.2 Mains Voltages

**WARNING** Contact with mains voltages can cause serious injury and even death. It is advised that a Markes International trained service engineer carries out all servicing of this instrument to ensure no safety risks are created for the engineer and/or the user, especially after the servicing is complete.

Some internal parts of the CIA Advantage-xr carry dangerous mains voltages. If the CIA Advantage-xr is connected to a power source, even if the power switch is off, potentially dangerous voltages exist on:

- The wiring between the power cord and the mains inlet.
- The wiring between the mains inlet and the power supplies.
- The mains inlet and power supplies themselves.
All parts carrying high voltages are shielded by covers. If the covers are in place it should be difficult to make contact with dangerous voltages.

The unit covers should only be removed if specifically instructed to do so, and should never be removed when the power cable is connected, even if the power switch is off. The safe state of the equipment must be verified following any service or repair.

1.4.3 **Power cord**

The instrument must be suitably earthed via the power cord.

Any cord set used with this equipment shall be previously approved and adequately rated according to the country of use.

Ensure at all times that the plug (electrical isolator) can be easily and quickly accessed during equipment use.

1.4.4 **High Temperatures**

Some parts of CIA Advantage-xr can be hot enough to represent a burn hazard. These parts are contained within the heated valve enclosure.

These zones are labelled with 'Burn hazard' labels similar to that shown below. Whenever possible cool these areas of the system to room temperature before working on it and ALWAYS operate the instrument with the covers in place to avoid accidental contact with these zones.

Due to the high temperatures involved in the flow path, other zones of the instrument will be at higher temperatures during operation. There may not on visual inspection be obvious to the user. These zones are: the insulation of the GC transfer line and the top and side covers (especially directly above the heated valves).

1.4.5 **Carrier Gas safety**

Previously approved and correctly rated external gas regulator should be used with this unit. Maximum inlet pressure must not exceed 60 psi.

1.4.6 **Cleaning and decontamination**

Please consult your local agent or Markes International for information on decontamination or the use of cleaning agents.

**NOTE** Incorrect cleaning/decontamination could result in damage to the instrument.

1.5 **Technical specifications**

1.5.1 **Dimensions and weight**

Select the laboratory bench space before your systems arrives. Pay attention to the total height requirements. Avoid bench space with overhanging shelves. Allow at least 20 cm clearance between the back of the equipment and a wall to dissipate hot air.
1.5.2 **Power Consumption**

The number and type of electrical outlets depends on the size and complexity of your system. Each TD unit will have a label next to the power cord connector that lists the line voltage requirements. The power consumption and requirements depend on the system ordered.

<table>
<thead>
<tr>
<th><strong>Electrical Properties</strong></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Power (W)</td>
<td>900</td>
</tr>
<tr>
<td>Line voltage (V)</td>
<td>100-240*</td>
</tr>
<tr>
<td>Frequency (Hz)</td>
<td>50-60</td>
</tr>
<tr>
<td>Inrush Current (A)</td>
<td>&lt;40 (cold start)</td>
</tr>
<tr>
<td></td>
<td>*Automatically selected</td>
</tr>
</tbody>
</table>

1.5.3 **PC Specification**

The recommended minimum PC specifications are:

<table>
<thead>
<tr>
<th><strong>PC Specification</strong></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Processor</td>
<td>1 GHz 64-bit dual-core or better</td>
</tr>
<tr>
<td>RAM</td>
<td>4 GB</td>
</tr>
<tr>
<td>Free Disk Space</td>
<td>2 GB</td>
</tr>
<tr>
<td>Graphics card</td>
<td>DirectX®9 or later</td>
</tr>
<tr>
<td>Operating System</td>
<td>64-bit Windows 7, 8.1 or 10</td>
</tr>
<tr>
<td>Minimum Resolution</td>
<td>1024 x 768</td>
</tr>
<tr>
<td>Peripherals</td>
<td>Windows compatible keyboard and mouse</td>
</tr>
</tbody>
</table>

CIA Advantage-xr requires a either a free serial port or a free USB port for communication with the PC.

1.6 **Environmental Operating Conditions**

It is advisable to operate the system in a clean laboratory environment, with minimal atmospheric concentrations of organic vapours. Performance can be affected by sources of heat and cold from heating, air conditioning systems or drafts.

The instrument should be protected from conditions that could cause exposure to frost, dew, percolating water, rain or excessive direct sunlight.

**Temperature**

Recommended operating ambient temperature range is 15 to 30°C.
**Humidity**

Recommended operating humidity range is 5 to 95% non-condensing.

**Altitude**

This product should not be operated above 2000m (~6500ft).

**CAUTION** For storage or shipping the allowable temperature range is -40 to 70°C and the allowable humidity range is 5-95% non-condensing. After instrument exposure to extremes of temperature or humidity, allow 2 hours for return to the recommended ranges before switching on.

### 1.7 Technical Support Contact Details

In the first instance please contact your supplier. If they are unable to resolve your query, please contact Markes International on the details below.

- **Website:** www.markes.com
- **E-Mail:** support@markes.com
- **Telephone:**
  - +44 (0)1443 230935 (UK office)
  - +49 (0)69 6681089-10 (German office)
  - +1 866 483 5684 (US office (toll-free))
  - +86 21 5465 1216 (China office)
2. Quick Start Guide

The information provided here is a brief guide to running your first samples on the CIA Advantage-xr connected to a UNITY-xr system. For more detailed information please refer to the subsequent sections in this manual.

2.1 Instrument overview

The key parts of CIA Advantage-xr are shown labelled below. They will be referred to throughout the Installation and User Manuals.

This section of the manual assumes that the instrument and software have been installed and that the appropriate cold trap has been installed in the UNITY-xr.

Whole air/gas samples e.g. continuous monitoring of air/gas streams or samples collected in whole-air containers such as canisters are introduced via one or more of the sample channels immediately to the right side of the instrument. See section 3.1 for a more detailed description.
2.2 Sequence of operation

Canister sampling methods follow this basic sequence of operation:

1. **Leak test (optional):** An automated, no flow leak test of the internal flow path is performed prior to sampling. Note the sampling line is not tested.
2. **Pressure release:** Removes residual pressure prior to sampling.
3. **Internal standard addition (optional):** Addition of a gas phase internal standard to the focusing (cold) trap.
4. **Sample purge:** Flushes system with sample.
5. **Sampling:** A metered volume of sample is collected on the focusing (cold) trap.
6. **Line flush:** The sample path is flushed with carrier gas to clean the system.
7. **Trap purge:** An ambient temperature purge of the focusing (cold) trap is performed to remove air, water and solvents before heating.
8. **Trap desorption:** The focusing (cold) trap is rapidly heated with a flow of carrier gas in the backflush direction, desorbed analytes are directed to the gas chromatograph (GC) via a heated transfer line and the GC run is triggered to start. There is an option to split the sample at this stage.

For a more detailed description of the modes of operation available on the UNITY–CIA Advantage-xr please see section 5.11.

2.3 Software overview

CIA Advantage-xr software can be started by clicking on the desktop icon or selecting **Start > Programs > Markes International > MIC 2.0.** This launches the Markes Instrument Control (MIC) homepage.

![Markes Instrument Control homepage](image)

Figure 3: Markes Instrument Control homepage

The different sections of the software can be accessed from the homepage, the key sections required for running a sample can be found under the ‘workflow’ heading.
2.3.1 Method Editor

Clicking on the Method editor tile in the home screen will open up the screen below.

Use the new method icon in the toolbar to select a template method to start from (i.e. TD MFC Sampling). Set the times, temperatures, flows and splits and click OK and a prompt to save the method will appear. Change the method name, add any comments and click OK to save. For more information on the parameters and templates available in the method editor please see section 5.

![Method editor](image)

Figure 4: Method editor
2.3.2 Creating a sequence

Select the sequence tile from the home screen to open the sequence edit view. Double click under the ‘Method’ heading and select a method from the ‘All Methods’ folder, update the sample type and comments column and enter the number of the channel you are going to use.

Depending on the configuration of the CIA Advantage-xr, there are either 4 or 14 sample channels. Select the sample channel to be analysed and the sample gas for each sample in the sequence.

![Creating a sequence](image)

Figure 5: Creating a sequence

2.3.3 Running a sample

- **Connect samples to the CIA Advantage-xr**
  All samples should be connected to the appropriate sampling port via non-contaminating sample tubing terminating in a 1/8” Swagelok nut.

- **Set up a method in the method editor**
  Set up the temperatures, times and splits (on or off) for the sample in the software.

- **Set up a sequence (even if you only want to run 1 sample you need to use the sequence view)**
  Create a sequence by changing the sample type, method, channel position and sample gas. Add more samples to the sequence using the tool bar icons when applicable.

- **Set up a sequence on the GC so that it is waiting for an external trigger**
  Set the GC up so that it is waiting for an external start signal – a green tick sign should be shown in the UNITY-xr status dialogue (see section 9) for the GC and the message “GC ready” will be visible.

- **Start your analysis**
  Press the ‘play’ button at the top of the edit sequence view.

2.3.4 Additional considerations

- Set temperatures that are compatible with the focusing trap sorbent and sample (refer to Application Note TDTS 5).
- If the sample is humid set the cold trap temperature to +30°C (refer to Application Note TDTS 26).
- Use a split during trap desorption where possible to achieve the best chromatography.
- If connected to a GC-MS ensure that all methods have the split on in standby option selected. Set the flow to a minimum of 10 mL/min.
3. Instrument Familiarisation

The UNITY-xr Thermal Desorber normally requires sample introduction by means of a standard sample tube. This could be a sorbent tube (used for diffusive or pumped sampling) or an empty tube into which solid or liquid samples are weighed for direct desorption. The CIA Advantage-xr accessory connects to UNITY-xr and extends the compatible sample range to include whole air/gas samples e.g. continuous monitoring of air/gas streams or samples collected in whole-air containers such as canisters or Tedlar bags. Tubes can still be desorbed using a UNITY-xr configured with the CIA Advantage-xr accessory.

3.1 CIA Advantage-xr

The CIA Advantage-xr contains a stream selection valve, switching valves and an interface to UNITY, all of which are controlled via Markes Instrument Control software.

The CIA Advantage-xr is compatible with gas-phase samples ranging in pressure from sub-atmospheric to 60 psig.

Depending upon the instrument configuration, between four and twenty seven inlet ports will be available.

3.1.1 Instrument Configurations

**CIA Advantage T-xr**

The CIA Advantage T-xr is a system for analysis of trace-level components in ambient air or gas streams. The system is able to introduce mass flow controlled samples, in volumes from 5 mL up to 15 L onto the cold trap within UNITY-xr. The CIA Advantage T-xr has four sample channels.

**CIA Advantage HL-xr**

The CIA Advantage HL-xr is a versatile system for the analysis of both high and low concentration samples and for screening unknowns. The CIA Advantage HL-xr is capable of introducing sample volumes from 0.5 mL using a gas loop, in addition to mass flow control for the widest sample volume range. The CIA Advantage HL-xr has 14 sample channels.

**CIA Satellite-xr**

The CIA Satellite-xr can be added to either CIA Advantage T-xr or CIA Advantage HL-xr to provide additional sample capacity. A CIA Satellite-xr connected with a CIA Advantage T-xr provides 17 channels, while in combination with a CIA Advantage HL-xr 27 channels are available.

**NOTE** CIA Satellite-xr cannot be interfaced directly to UNITY.

3.1.2 Serial Number Format

CIA Advantage-xr serial numbers take the following formats:

- CIA Advantage HL-xr: GB00H1nnnn and GB00H2nnnn
- CIA Advantage T-xr: GB00H4nnnn and GB00H5nnnn
- CIA Satellite-xr: GB00H7nnnn and GB00H8nnnn
**Location**

The model type and serial number of the instrument can be found on the back panel of the instrument (see Figure 6). The instrument serial number is also shown on a label on the top front panel (see Figure 1).

3.1.3 **Electrical Connections**

Summary of connections:

**Power**

The CIA Advantage-xr is supplied with a power cord and a plug appropriate for the country from which the order originates. Power cord lengths for the CIA Advantage-xr are approximately 2 m in length.

**Fuse**

The CIA Advantage-xr is fitted with a replaceable 12 A time lag fuse, with high breaking capacity. A replacement fuse is supplied in the CIA Advantage-xr shipping kit (SERZ-FS12A0).

**PC Connection**

Connection to the controlling PC is made using a RS232 null modem serial cable (SERZ-0189). The cable should be connected to an available serial port on the control PC. A USB-serial adapter (U-USBSR) is supplied for use if the PC in question does not have an available serial port.

**Remote Start Connection**

This connector can be used to provide a start signal for the CIA Advantage-xr. Typically this connection is not used.

![Figure 6: CIA Advantage-xr back panel connections](image-url)
3.1.4 Gas connections

Description of Gas Connections:

Pneumatic gas (Air/N2)

The pneumatics gas is used to switch several pneumatically operated valves. The pressure required for this gas supply is 50-60 psig. The supply can be shared with that on the UNITY purge gas although a separately regulated supply is recommended. A gas pneumatics accessory (U-GAS01) is supplied with every CIA Advantage-xr to facilitate this.

**NOTE** CIA Advantage-xr will not operate properly without this gas supply.

Carrier Gas

CIA Advantage-xr requires a regulated supply of carrier gas, in addition to the UNITY-xr carrier gas supply. The carrier gas should be 5.0 grade (i.e. 99.999%) or higher-purity helium or nitrogen, supplied at a pressure approximately equal to that of the UNITY carrier gas supply. As with the UNITY carrier gas supply, this pressure must not exceed 60 psig.

The gas pneumatics accessory (U-GAS01) supplied with every CIA Advantage-xr facilitates regulation of this supply.

**NOTE** CIA Advantage-xr will not operate properly without this gas supply.

**WARNING** Hydrogen is not suitable as a carrier gas for CIA Advantage-xr.

Internal Standard

If an Internal Standard is to be used, a pressurised cylinder containing an appropriate calibration gas can be connected. The cylinder must be equipped with appropriate (inert) step-down gas pressure regulation and should be connected to the CIA Advantage-xr using a length of 1/8-inch or 1/16-inch clean, stainless steel tubing. The pressure of the internal standard should be set approximately 2 psi below the pressure of the CIA Advantage-xr carrier gas, but must not exceed 30 psig.

A suitable internal standard would contain one or more gas-phase organic components that behave in a similar way to the target analytes, but are not found in the sample. For example deuterated toluene is commonly selected as an internal standard for BTX analysis, while the US EPA method TO-15 internal standard contains bromochloromethane, 1-bromo-4-fluorobenzene, chlorobenzene-d5 and 1,4-difluorobenzene.

Purge Gas

A purge gas that is different to the carrier gas can be used to facilitate clean-up after running very high concentration samples. Nitrogen or humidified nitrogen is recommended. If not used carrier gas will be used to perform flow path purging.

**NOTE** A humidified gas is more effective, than its dry equivalent, at removing contamination. For trace level analysis a humid purge gas is not required.

The supply pressure should be set approximately equal to the CIA Advantage-xr carrier gas pressure, but must not exceed 60 psig.
Canister Vent

The canister vent is opened occasionally during operation to prevent any sample being contaminated with a previous sample or diluted with carrier gas. The canister vent should be connected to a pump to be fully effective.

Sample Channels

The sample channels are located on the right hand side of the CIA Advantage-xr. On CIA Advantage HL-xr there are fourteen channels (see Figure 7) while on a CIA Advantage T-xr there are four channels.

NOTE

The pressure supplied to any one of these channels must not exceed 60 psig.

Gas Vents

There are two vents located on the top of the CIA Advantage HL-xr (see Figure 8); one is for the Internal Standard and the other is a sample vent. The CIA Advantage T-xr only has the Internal Standard vent. The CIA Satellite-xr has no vents.

At the end of sampling and after a purge of carrier gas to eliminate air from the trap, the UNITY-xr focusing trap heats in the normal way, transferring compounds of interest to the analytical system and triggering the measurement cycle. The outlet split at this point may be entered in the software and is controlled by the mass flow controller. Collection of the next sample can begin, if required, as soon as the cold trap has re-equilibrated at its trapping temperature.
3.1.5 **Nafion Dryer**

The Nafion dryer is a sample drying accessory which extracts water from the sample gas stream using a permeable membrane and a counter flow of dry gas. Some concurrent losses of polar species from the sample stream are also observed so the Nafion dryer is only recommended during the analysis of non-polar species. The Nafion dryer is installed onto the frame of the CIA-Advantage-xr and the sample gas stream diverted through the dryer prior to collection on the focusing trap.

3.1.6 **Kori-xr**

The Kori-xr (see Figure 9) is installed between the CIA Advantage-xr and UNITY-xr and consists of a cryogen-free trap that is placed into the sample flow path, upstream of the sorbent trap. The Kori-xr selectively removes water from the sample gas stream with virtually no loss of polar or non-polar species. It is ideal for GC-MS analysis of complex air samples.

The Kori-xr works in two steps:

- **Sampling phase**: the sample gas stream passes from the CIA Advantage-xr through the Kori-xr prior to collection on the focusing (cold) trap. The Kori-xr trap is held at a sub-zero temperature causing vapour-phase water in the air sample to be deposited as ice in the Kori-xr trap. This process doesn’t impact on the collection of VOCs onto the focusing (cold) trap.

- **Purging phase**: when sampling is completed, the Kori-xr trap is heated with a flow of carrier gas through it in order to purge any ice that has deposited during the sampling phase. This purge starts after sampling and finishes once the focusing (cold) trap has desorbed and cooled down.
3.2 UNITY-xr

3.2.1 CIA Advantage-xr transfer line

The CIA Advantage-xr connects to the UNITY-xr via one of two heated transfer lines. Analytes sampled from CIA Advantage-xr pass through this transfer line directly onto the focusing trap in the UNITY-xr.

The standard CIA Advantage-xr line interfaces through the UNITY-xr tube oven (left hand side of UNITY-xr), however if the system configuration includes an ULTRA-xr tube autosampler or the UNITY-xr tube oven is
required to carry out single tube desorptions then a different transfer line is used which terminates in a T-piece connection positioned between the split tube and heated valve on the right hand side of the UNITY-xr.

3.2.2 The focusing (cold) trap

The quartz cold trap contains a 2 mm diameter x 60 mm long bed of sorbent (30 to 100 mg depending on sorbent density) supported by quartz or glass wool. Note that the length of the first plug of glass wool is included in the total 60 mm sorbent bed. The full range of cold traps available can be found in the Markes International thermal desorption accessories and consumables catalogue.

UNITY-xr contains a 2-stage Peltier cell, which uniformly cools the entire 60 mm sorbent bed to a minimum of -30°C and a maximum of +50°C in ambient temperatures. No liquid cryogen is required. Dry air or nitrogen flows into the cold trap box creating a slight positive pressure and minimising entry of water from the laboratory atmosphere. If the cold trap box was not purged, ice would quickly build up around the Peltier cell, which is maintained at sub-zero temperatures throughout operation.

Once all the target analytes have been collected and focused in the cold trap, the trap oven heats rapidly reaching rates of 100°C/sec for the first critical stages of trap desorption. Uncompromised capillary chromatography is produced without on-column focusing and with desorption flows as low as 2 mL/min. This facilitates splitless operation with high-resolution capillary GC.

3.2.3 Trap filters and seals

As with the sample tube, the cold trap is sealed into the gas flow path of UNITY-xr via O-rings, which seal on the outer wall of the trap. At the cool non-valve end of the trap, the O-ring is backed up with a porous PTFE filter to prevent contamination of the pneumatics in the event of sorbent particles migrating out of the trap. The user has access to this O-ring seal and filter in the connector, but a service visit is required to access and change the trap O-ring seal in the heated valve. As the cold trap is only changed infrequently, the seals will rarely, if ever, need to be replaced. It is recommended that UNITY-xr is serviced once per year and that the valve-end seal be changed as part of this annual maintenance operation.

3.2.4 Split filters

There is a split filter tube packed with charcoal (P/N SERAAA-1600) on the split line upstream of the on/off solenoid and needle valve. This prevents the split portion of the sample from contaminating the valves and from reaching the laboratory air. The flow path up to the charcoal filter is heated and constructed of inert-coated tubing. The split filter itself is the same size as a standard sample tube and may be readily replaced by a clean sorbent tube if the split effluent is to be re-collected for repeat analysis. The split filter (or re-collection tube) is sealed into the split flow line using easy-connect, Viton O-ring seals. The sampling end/grooved end of the re-collection tube should point to the rear of the instrument.

Conventional charcoal split filters will become contaminated over time and should be reconditioned or repacked when required.
3.2.5  UNITY-xr Gas supplies

Pneumatic gas (Air/N2)

The pneumatics gas is used to switch several pneumatically operated valves and purge the trap box. The pressure required for this gas supply is 50-60 psig and it is essential that this gas is dry (dewpoint below -50°C).

Helium or hydrogen should never be used as the pneumatic gas.

NOTE  UNITY-xr will not operate properly without this gas supply.

Carrier Gas

UNITY-xr requires a regulated supply of carrier gas. The carrier gas should be 5.5 grade (i.e. 99.9995%) or higher-purity helium or nitrogen. Carrier gas supply this pressure must not exceed 60 psig.

NOTE  UNITY-xr will not operate properly without this gas supply.

3.3  ULTRA-xr

The ULTRA-xr is a 100 tube autosampler that can be connected to the UNITY-CIA Advantage-xr, allowing analysis of sample tubes as well as on-line sampling within the same sequence. For more details on the ULTRA-xr, please refer to the QUI-1120 UNITY-ULTRA-xr User manual.
4. Software Familiarisation

UNITY-CIA Advantage-xr is controlled by Markes Instrument Control software (MIC), this section details software familiarisation.

4.1 Markes Instrument Control installation

This user manual assumes the software has already been installed onto the instrument control PC by the installation engineer. For help with installing the software please contact your local agent or Markes International Customer Support.

4.2 Starting the software

Start the software by clicking on the Markes Instrument Control (MIC) desktop icon. Alternatively go to Start > All Programs > Markes International > MIC 2.0.

![Markes Instrument Control desktop icon](image)

Figure 11: Markes Instrument Control desktop icon

In order for the User Interface (UI) to start up the software must detect the configured instrument(s), a status bar will appear on screen during detection with the options ‘Exit’ and ‘Configure’ (see Figure 12).

![Connecting to instrument(s) status bar](image)

Figure 12: Connecting to instrument(s) status bar

During connecting a status bar will appear and the configured instruments listed above it. Once an instrument has been detected the ‘Offline’ text will change to ‘Ready’. If the instruments are turned on and configured you do not need to do anything at this stage, the software will detect them and open the homepage.

**Exit:** Cancels the connection to the instrument and exits Markes Instrument Control.

**Configure:** Opens the Instrument Configuration page.

If the configured instruments are not detected they will remain ‘Offline’, check that the instruments are turned on and communication cables have not become dislodged from the instrument or the PC. Open the configuration page and check that the correct COM ports are assigned to the instruments (see section 8.1.1). Contact your local agent or Markes International Customer Support for further assistance.

When the software starts default parameters for flow path temperature, trap temperature and standby split flow will be sent to the instrument.
4.3 MIC Homepage

![Markes Instrument Control Homepage](image)

Figure 13: Markes Instrument Control Homepage

The Markes Instrument Control home screen contains a number of tiles that lead to the different sections of the software; these are described briefly below and in more detail later in this manual.

**Instruments**

The instrument tile can be used to view the instrument status from the homepage and access the instrument utilities. See sections 4 and 10 respectively.

**Workflow**

- **Method editor:** For viewing and editing instrument methods. See section 5 for more information.
- **Sequence:** For creating sequences and running samples. See section 6 for more information.
- **Sequence history:** Contains reports for previous sequences. See section 7 for more information.

**Tools**

- **Instrument configuration:** Contains the current instrument configuration settings and hardware options. See section 8.1 for more information.
- **Settings:** Contains standby, sequence and reporting options.
- **About:** Contains information about this software including version number.
4.4 Instrument tile

The instrument tile can be found on the homepage and in the sequencer. The instrument tile shows the status of the instrument at a glance:

- **Offline:** One or more of the configured instruments has not been detected.
- **Idle:** The instrument is ready to run.
- **Active:** The instrument is running a sequence or being controlled via direct control.
- **Error:** The instrument is in an error state; it is not possible to run samples or access direct control until the error is resolved.

To see more detailed information on the instrument status left click anywhere on the instrument tile – the instrument status dialogue will pop out, see section 9 for a detailed description of the instrument status dialogue.

If there is a ‘simulated’ banner across the top of the instrument tile, as shown in Figure 15: Simulation mode instrument tile, the software is in simulation mode. This means that the software is simulating one or more of the configured instruments, it is not actually controlling this instrument and samples will not run however the software will act as if it is connected to an instrument. This mode is typically used for training or for conveniently viewing methods, sequences etc. when the instrument is offline. The simulated mode is selected from within the configuration tab, See section 8.1 for further details.
5. **Method Editor**

The method editor can be accessed by left- or right-clicking the method editor icon on the MIC home page (see Figure 16). The method contains the set of parameters that will be used by the UNITY–CIA Advantage-xr when performing an analysis.

![Method editor icon](image)

**Figure 16: Method editor icon**

### 5.1 Overview

The layout of the method editor is shown in Figure 17:

1. The title bar of the method window includes the name of the method currently displayed and whether it has been modified since it was last saved, this is indicated by the word [Modified] after the method name.
2. Toolbar icons at the top perform functions such as ‘create a new method’ and ‘save method’.
3. The collapsible methods pane at the left hand side of the window is a convenient way to explore saved and template methods.
4. At the bottom of the methods pane the method history is detailed.
5. A ‘Set’ button allows the method parameters to be sent to the instrument.
6. A split calculator is included in the bottom right corner to help with choosing flow rates in methods.
7. The template method (or operating mode) from which the method was derived is shown at the top of the parameter set.
5.2 Creating a new method

To create a new method, click on the icon in the top toolbar of the method editor. This will open a new method window containing a list of method templates (see Figure 18, more details on each template can be found in section 5.11). Select the desired template method and click <OK>.

The method editor will then be populated with the appropriate parameters.
5.3 Saving a method

A method can be saved by left-clicking on the in the top toolbar, or by clicking on <OK> at the bottom of the method editor page. A window will open (see Figure 19) where the user can choose the method name, enter the name of the author and the company, and enter any comments (nature of changes for example). Click <OK> to finish saving the method. If the same method name is chosen, the method will be up-issued. If ‘Read Only’ is selected, the user won’t be able to make any further changes and save it under the same method name.

NOTE Template methods cannot be up-issued and must be saved with a new name.

5.4 Opening an existing method

To open a previously stored method, left-click on the in the top toolbar in the method editor. A window which contains the list of all stored methods will open (See Figure 20). This list can be sorted by name or by date using the drop down menu at the bottom of the window. Select the method to open and left-click <OK>.
Alternatively, methods can be accessed via the “Methods” pane on the left hand side of the method editor page (see Figure 21). Click on the bar of the pane to expand/collapse it. Double click on one of the “All Methods” folders to expand the list of all the stored methods and double-left-click on the desired method to load it.

Once opened, the method can be adjusted and saved with a different name, or saved with the same name and up-issued (unless it is a Read Only method).

### 5.5 Method history and version numbers

To view the history of a method, open the “Methods” pane within the method editor. The different versions of the method will be listed in the bottom left of the pane, with the version number denoted in square brackets. To view the full method history back to the original template method which was used, double-left-click on “...+”.

Left-click on an entry in the history list to view the method details in the adjacent pane. To view the full set of parameters for a historic method, double-left-click on it to load it into the method editor. Here it can be reviewed, edited, and re-saved with a different name.

**NOTE** When running sequences, the latest version for a method will always be used.
5.6 Copy a method to a project folder

To copy an existing method to a project folder, right click on the method from the methods pane and select ‘Add to project folder…’ and the folder of choice (see Figure 23).

If a new project folder is required, select ‘New project folder’, in which case a new folder named “untitled 1” will be created and will appear in the folder list in the methods pane. The name of this folder can be edited at any time by left clicking on it.

5.7 Deleting methods

To delete a method, open the “Methods” pane from the method editor. Double-left-click on one of the “All Methods” lists, right-click on a method and select “Delete”.

5.8 Variable parameters

MIC supports variable or “unlocked” parameters as part of a method. Any parameter with an adjacent purple padlock symbol can be unlocked to avoid the need to create multiple methods for parameter optimization. To lock and unlock parameters, click on the padlock symbol. The padlock will be replaced with an orange pencil symbol, indicating the parameter is now unlocked. Proceed to save the method. When the method is selected in the sequence, each unlocked parameter will appear as a separate column that can be adjusted independently for each line in a sequence.

- denotes a locked parameter, set in the method
5.9 Exporting and importing methods

To export methods to a .bin file, open the “Method editor” and left-click on [ ]. Select the required method(s) from the list and click <Export>. Browse to a folder and click <OK> to complete the export process.

To import a method from a .bin file, open the “Method editor” and left-click on [ ]. Select the required file(s) and click <Open> to import the method(s). If a method already exists with the same name, the user will be prompted to enter another.

Old versions of the same method are also exported/imported to allow full traceability.

5.10 Method reports

The method report shows a formatted list of all the method parameters, along with the method overview, history and any comments entered when it was saved. There are two ways to view a method report:

- Open the method in the “Method editor” and left-click on [ ].
- Right-click on the method name from any location and select “Method Report”.

5.11 Template methods

There are a number of possible operating modes that the UNITY–CIA Advantage-xr can perform; each mode has a different set of parameters that define its particular sequence of operation. Methods in MIC are derived from templates that define the mode of operation.

5.11.1 MFC Sampling

MFC sampling is available for all CIA Advantage-xr configurations, and is the recommended method of operation when sample volumes of 5 mL or greater are required.

- **General**

  - **Apply presets for:** This option offers presets for popular or regulated methods (e.g. TO-15). Select a method from the drop down menu and the method parameters will change to the optimal or regulated values in order to efficiently analyse your sample.
**Standby split on:** Determines whether the split vent is opened when the TD system is in a standby state (i.e. not being used). Checking the box selects Standby split on. An unchecked box will turn the split off in standby.

The flow rate through the split vent is controlled by the split MFC and can be set to between 5 and 100 mL/min. Typical flows are 10-20 mL/min.

**NOTE** If the TD system is being operated with a mass spectrometer (MS) or an electron capture detector (ECD) on the gas chromatograph, it is strongly recommended that the split is on in standby. Without a positive gas flow through the split vent air can slowly enter the system producing an undesirable increase in background signal.

**Flow path temperature:** Temperature setting (in °C) for the entire thermal desorption flow path.

**Sampling line temperature:** Temperature setting (in °C) for the sampling line, i.e. line between the canister and the sample channel.

**Overlap:** Tick this box to allow overlapping of samples. Collection of the next sample will begin while the previous sample is acquiring.

**GC cycle time:** Use of an appropriate GC cycle time allows for the most efficient operation of the analytical system.

The software uses the GC cycle time to calculate when the next sample in a sequence can be collected such that the trap is ready to heat just as the GC becomes ready following completion of the previous analysis. This maximises the amount of time that the GC(MS) is in operation.

\[
\text{GC cycle time} = \text{GC run time} + \text{GC cool down time} + \text{GC equilibration time}
\]

**NOTE** If the GC cycle time is set to 0, the next sample in a sequence will not be started until the GC is ready.

**Minimum carrier pressure:** If the carrier gas pressure, as measured by the UNITY-xr pressure transducer, drops below this minimum pressure no further samples will be analysed until the pressure increases above this minimum threshold. The default value is 5 psi. This value may be reduced if a GC method is present that uses a low column head pressure.

**Leak test:** The leak test is optional. If the check box is ticked, then an automated, no flow leak test of the integrated UNITY–CIA Advantage-xr system is performed. If the leak test fails the sample will not be analysed and the sample remains uncollected.
Pre sampling

Sample purge time (min): 1.0
Sample purge flow (mL/min): 50

Add internal standard

Add internal standard using

Loop fill time (min): 1.0
Loop equilibration time (min): 0.1
Loop injection time (min): 1.0
Loop injection flow (mL/min): 50

Internal standard volume (mL): 50
Internal standard gas type: N2

Sample purge: Sample purge introduces sample into the CIA Advantage-xr and UNITY-xr flow paths prior to sample collection onto the cold trap. The purge flow is controlled by the UNITY-xr split MFC.

Add internal standard: Optional addition of internal standard.

Internal standard can be added to the cold trap prior to the sampling process. If this option is selected, the internal standard method parameters become available.

The user has the option between two types of addition: either using the internal standard loop (fixed volume of 1mL) or using the MFC for higher volumes (5-500 mL). Different method parameters become available depending on the addition type selected.

**Loop**

Loop fill time: the time for which the internal standard loop is purged with internal standard. The flow rate is vented through and controlled by the Internal Standard Vent needle valve (see Figure 8).

Loop equilibration time: the time that the internal standard loop pressure is allowed to equilibrate to ambient pressure, prior to transfer to the UNITY-xr cold trap.

Loop injection time: The time that the internal standard loop is purged with carrier gas to transfer its contents to the UNITY-xr cold trap.

Loop injection flow: the flow is controlled by the UNITY-xr MFC, and can be set to between 2 and 500 mL/min.

**MFC**

Internal standard volume: enter the desired volume (mL).

Internal standard gas type: select from the drop down menu the type of gas.
**Sampling**

- **Sample by time:** Untick the sample by volume check box to collect a sample for a specified length of time (range 0 to 999.9 in 0.1 min increments) at the defined sample flow rate (typically 50 mL/min).

- **Sample by volume:** Tick the sample by volume check box to collect the desired sample volume at the defined sampling flow rate (typically 50 mL/min).

- **Sampling flow:** This is the flow rate (in mL/min) at which sample will be collected onto the cold trap. Typically flows should be set in the range 10-50 mL/min.

  **NOTE** While sampling flow rates in excess of 50 mL/min can be used there is a greater chance of breakthrough of components of interests if such flow rates are used.

**Post sampling purge**

- **Use dedicated purge channel:** Determines whether the post sampling purge of the flow path is performed with the purge gas or carrier gas.

  If selected the post sampling purge will use the purge gas channel (located on the rear panel of the CIA Advantage-xr – see Figure 6).

  If not selected the post sampling purge will be conducted using the UNITY-xr carrier gas supply.

  **NOTE** Only select this option if a purge gas is connected to the CIA Advantage-xr.
Post sampling purge:
The flow path is purged with carrier gas (or purge gas) following sampling in order to clean the flow path in preparation for the next sample. A typical duration and flow would be 1 min at 50 mL/min but longer may be required following high concentration samples.

NOTE
If a Kori-xr is installed, a longer purge might be required in order to purge all the deposited water from the Kori-xr trap, especially if a very humid gas has been sampled or if a large sample volume has been taken.

Enable CIA post sampling purge:
If selected, this allows further purge (using the dedicated purge channel if selected above) of the CIA Advantage-xr flow path until completion of trap desorption. This may be recommended following high concentration samples.

Kori settings

| Kori settings          |  
|------------------------|---
| Kori trap low (°C)     | -30°C |
| Kori Trap High (°C)    | 300°C |

These settings are only available if a Kori-xr has been installed and configured.

Kori trap low: Temperature (°C) at which the Kori-xr trap will be held during the sampling phase in order to remove the water contained into the sample. This temperature should be low enough in order for the water contained into the sample to be deposited as ice onto the Kori-xr trap (recommended temperature is -30°C).

Kori trap high: Temperature (°C) at which the Kori-xr trap will be heated up to in order to remove any ice formed during the sampling phase. The temperature should be high enough in order to purge all the ice from the Kori-xr trap to prepare it for the next sample (recommended temperature is 300°C). The Kori-xr trap will be held at high temperature during the duration of post sampling purge, trap purge and trap desorb.

The flow of carrier gas during the purging phase must be set manually using the needle valve on the front panel of the Kori-xr. Recommended flow range is 50-100 mL/min.
**Trap settings**

- **Desorb trap:** This option, if selected, allows the user to desorb the trap following the sampling. If unselected, the user has the possibility to perform a “sample stacking”, i.e. to sample another (or several) sample onto the trap, before desorbing it.

- **Trap purge:** Purges the focusing (cold) trap with carrier gas in the sampling direction to remove any residual air, water or solvent prior to heating the trap. As this purge is performed in the sampling direction, anything purge from the trap is directed away from the analytical column.

  The time (in minutes) and flow rate (in mL/min) will need to be set depending upon the type of adsorbents in the cold trap and the amount of moisture in the samples collected.

- **Elevated trap purge:** This option allows purging of the trap prior to desorption at a temperature above the Trap low temperature to help removing excess moisture or unwanted solvent.

- **Trap low temperature:** Temperature (in °C) at which the cold trap will be held during all sampling phases.

- **Trap heating rate:** The rate (in °C/s) at which the trap will heated from the trap low temperature to the trap high temperature during the trap desorption stage. Selecting ‘MAX’ applies the fastest heating rate possible which is approximately 60-100°C/s.

- **Trap high temperature:** Temperature at which cold trap will be desorbed, transferring analytes to the GC(MS).

  - **CAUTION:** Do not set this temperature higher than the maximum recommended desorption temperature of the installed cold trap. Please refer to the Cold Trap Certificate supplied with the Cold Trap for further information.

- **Trap desorption time:** Time (in minutes) for which the trap will be held at the trap high temperature for trap desorption.
**Desorb split:**

Determines whether the UNITY-xr split vent is open during trap desorption.

If unselected the UNITY-xr cold trap will be desorbed without any split, transferring all of the sample collected to the GC(MS).

If split on is selected the split flow rate will be controlled by the UNITY-xr MFC and can be set to between 2 and 500 mL/min.

**NOTE**

If the UNITY-xr cold trap is to be desorbed without split, the GC column flow must be greater than 2.0 mL/min to ensure efficient trap desorption.

**Post desorption purge:**

The post desorption purge allows for further purging of the sample flow path, after completion of the trap desorption to aid system clean up. This should only be required for high concentration samples. This option is not available if a Kori-xr is configured.

The Post desorption purge time (in minutes) and flow rate (in mL/min) can be set.

The Post desorption purge will use the dedicated purge gas channel if the ‘Use dedicated purge channel’ option is selected in the Post sampling purge section of the method. If this option is not selected the purge will be performed using the UNITY-xr carrier gas supply.

5.11.2 Loop Sampling

Loop sampling is only available for CIA Advantage HL-xr configurations. It is the recommended method of operation for high concentration samples where low sample volumes (e.g. 0.5 – 5 mL) are sufficient. It is also the most suitable method for screening samples where the concentration is unknown.

**NOTE**

Loop sampling is only suitable for pressurised samples. Samples at low pressures (i.e. < 5 psig) cannot be analysed using a loop sampling method.

**NOTE**

If a Kori-xr is installed, the Kori-xr trap will be held at flow path temperature for the entirety of the run/sequence.
Standby split on: Determines whether the split vent is opened when the TD system is in a standby state (i.e. not being used). Checking the box selects Standby split on. An unchecked box will turn the split off in standby.

The flow rate through the split vent is controlled by the split MFC and can be set to between 5 and 100 mL/min. Typical flows are 10-20 mL/min

NOTE If the TD system is being operated with a mass spectrometer (MS) or an electron capture detector (ECD) on the gas chromatograph, it is strongly recommended that the split is on in standby. Without a positive gas flow through the split vent air can slowly enter the system producing an undesirable increase in background signal.

Flow path temperature: Temperature setting (in °C) for the entire thermal desorption flow path.

Sampling line temperature: Temperature setting (in °C) for the sampling line, i.e. line between the canister and the sample channel.

Overlap: Tick this box to allow overlapping of samples. Collection of the next sample will begin while the previous sample is acquiring.

GC cycle time: Use of an appropriate GC cycle time allows for the most efficient operation of the analytical system.

The software uses the GC cycle time to calculate when the next sample in a sequence can be collected such that the trap is ready to heat just as the GC becomes ready following completion of the previous analysis. This maximises the amount of time that the GC(MS) is in operation.

GC cycle time = GC run time + GC cool down time + GC equilibration time

NOTE If the GC cycle time is set to 0, the next sample in a sequence will not be started until the GC is ready.

Minimum carrier pressure: If the carrier gas pressure, as measured by the UNITY-xr pressure transducer, drops below this minimum pressure no further samples will be analysed until the pressure increases above this minimum threshold. The default value is 5 psi. This value may be reduced if a GC method is present that uses a low column head pressure.

Leak test: The leak test is optional. If the check box is ticked, then an automated, no flow leak test of the integrated UNITY–CIA Advantage-xr system is performed. If the leak test fails the sample will not be analysed and the sample remains uncollected.
Pre sampling

Optional addition of internal standard.

Internal standard can be added prior to the sampling process. If this option is selected the internal standard method parameters become available.

The user has the option between two types of addition: either using the internal standard loop (fixed volume of 1mL) or using the MFC for higher volumes (5-500 mL). Different method parameters become available depending on the addition type selected.

**Loop**

- **Loop fill time**: the time for which the internal standard loop is purged with internal standard. The flow rate is vented through and controlled by the Internal Standard Vent needle valve (see Figure 8).
- **Loop equilibrate time**: the time that the internal standard loop pressure is allowed to equilibrate to ambient pressure, prior to transfer to the UNITY-xr cold trap.
- **Loop injection time**: The time that the internal standard loop is purged with carrier gas to transfer its contents to the UNITY-xr cold trap.
- **Loop injection flow**: the flow is controlled by the UNITY-xr MFC, and can be set to between 2 and 500 mL/min.

**MFC**

- **Internal standard volume**: enter the desired volume (mL).
- **Internal standard gas type**: select from the drop down menu the type of gas.
**Sampling**

**Sample volume:** Enter the desired sample volume (in mL). The minimum volume is limited by the size of the loop (0.5 mL) and the maximum volume is 20 mL. If a greater volume is required, please choose the MFC Sampling mode.

**Loop fill time:** The time for which the gas sampling loop is purged with sample.

**Loop fill rate:** The flow is controlled by the CIA Advantage-xr MFC, and can be set to between 2 and 500 mL/min. This flow is vented through the CIA Advantage-xr Sample Vent (see Figure 8).

**Sample injection time:** The time that the sample loop is purged with carrier gas to transfer its contents to the UNITY-xr cold trap.

**Sample injection flow rate:** The flow is controlled by the UNITY-xr MFC, and can be set to between 2 and 500 mL/min.

**Post sampling purge**

**Use dedicated purge channel:** Determines whether the post sampling purge of the flow path is performed with the purge gas or carrier gas.

If selected the post sampling purge will use the purge gas channel (located on the rear panel of the CIA Advantage-xr – see Figure 6).

If not selected the post sampling purge will be conducted using the UNITY-xr carrier gas supply.

NOTE Only select this option if a purge gas is connected to the CIA Advantage-xr.
Post sampling purge:
The sample line is purged with carrier gas (or purge gas) following sampling in order to clean the flow path in preparation for the next sample. A typical duration and flow would be 1 min at 50 mL/min but longer may be required following high concentration samples.

Enable CIA post sampling purge:
If selected, this allows further purge (using the dedicated purge channel if selected above) of the CIA Advantage-xr flow path until completion of trap desorption. This may be recommended following high concentration samples.

Trap settings

<table>
<thead>
<tr>
<th>Setting</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Desorb trap</td>
<td>This option, if selected, allows the user to desorb the trap following the sampling. If unselected, the user has the possibility to perform a “sample stacking”, i.e. to sample another (or several) sample onto the trap, before desorbing it.</td>
</tr>
<tr>
<td>Trap purge time (min)</td>
<td>1.0</td>
</tr>
<tr>
<td>Enable elevated trap purge temperature</td>
<td></td>
</tr>
<tr>
<td>Elevated trap purge temperature (°C)</td>
<td>25</td>
</tr>
<tr>
<td>Trap purge flow (mL/min)</td>
<td>50</td>
</tr>
<tr>
<td>Trap low temperature (°C)</td>
<td>25</td>
</tr>
<tr>
<td>Trap heating rate (°C/s)</td>
<td>MAX (fastest) which is approximately 60-100 °C/s.</td>
</tr>
<tr>
<td>Trap high temperature (°C)</td>
<td>200</td>
</tr>
<tr>
<td>Trap desorption time (min)</td>
<td>3.0</td>
</tr>
<tr>
<td>Desorb split on</td>
<td></td>
</tr>
<tr>
<td>Split flow (mL/min)</td>
<td>25</td>
</tr>
<tr>
<td>Post desorption purge time (min)</td>
<td>0.0</td>
</tr>
<tr>
<td>Post desorption purge flow (mL/min)</td>
<td>50</td>
</tr>
</tbody>
</table>

Desorb trap:
This option, if selected, allows the user to desorb the trap following the sampling. If unselected, the user has the possibility to perform a “sample stacking”, i.e. to sample another (or several) sample onto the trap, before desorbing it.

Trap purge:
Purges the focusing (cold) trap with carrier gas in the sampling direction to remove any residual air, water or solvent prior to heating the trap. As this purge is performed in the sampling direction, anything purge from the trap is directed away from the analytical column.

The time (in minutes) and flow rate (in mL/min) will need to be set depending upon the type of adsorbents in the cold trap and the amount of moisture in the samples collected.

Elevated trap purge:
This option allows purging of the trap prior to desorption at a temperature above the Trap low temperature to help removing excess moisture or unwanted solvent.

Trap low temperature:
Temperature (in °C) at which the cold trap will be held during all sampling phases.

Trap heating rate:
The rate (in °C/s) at which the trap will heated from the trap low temperature to the trap high temperature during the trap desorption stage. Selecting ‘MAX’ applies the fastest heating rate possible which is approximately 60-100 °C/s.
5.11.3 Trap Heat

The trap heat method enables the UNITY-xr focusing (cold trap) to be desorbed without first collecting a sample. This can be used for conditioning a cold trap, or for confirming that there is no sample carryover on the cold trap following an analysis.

General

Apply conditioning presets for:

Standby split on

Flow (mL/min)

Flow path temperature (°C)

Minimum carrier pressure (psig)

This option offers presets for the most popular traps. Select a trap type from the drop down menu and the method parameters will change to the optimal values in order to efficiently condition the trap.
Standby split on: Set a standby split when using a mass spectrometer to ensure the system flow path is purged and background contamination limited. Typical flows are 10-20 mL/min.

Flow path temperature: Temperature setting (in °C) for the entire thermal desorption flow path.

Minimum carrier pressure: The Minimum carrier pressure (psi) is the pressure below which the TD system will not be allowed to operate.

**Trap Purge**

- **Trap low temperature:** Temperature (°C) at which the cold trap will be held during all sampling phases.

- **Trap purge time:** This determines the time (min) that the cold trap will be purged with carrier gas prior to desorption. If heating a trap for the first time, this purge time should be set to 5 min at 50 mL/min to ensure all oxygen is removed from the trap prior to initial heating. This is especially important if the trap contains molecular sieve type sorbents.

- **Trap purge flow:** The flow rate for this carrier gas purge can be set to between 2 and 500 mL/min.

**Trap desorption**

- **Heating rate:** The rate (°C/s) at which the trap will be heated from the trap low temperature to the trap high temperature during the trap desorption stage. Selecting ‘MAX’ applies the fastest heating rate possible which is approximately 60-100 °C/s.

- **Trap high:** Temperature (°C) at which cold trap will be desorbed, transferring analytes to the GC(MS) or at which the trap will be conditioned prior to use.

  **CAUTION** Do not set this temperature higher than the maximum recommended desorption temperature of the installed cold trap. Please refer to the Cold Trap Certificate supplied with the Cold Trap for further information.

- **Time:** Time (min) for which the trap will be held at the trap high temperature for trap desorption.
Split on: Determines whether the split vent is open during trap desorption.

If unselected the cold trap will be desorbed without any split, transferring all of the sample collected to the GC(MS).

If split on is selected the split flow rate (mL/min) will be controlled by the split MFC and can be set to between 2 and 500 mL/min.

**NOTE** If the UNITY-xr cold trap is to be desorbed without split, the GC column must be greater than 2 mL/min to ensure efficient trap desorption.

Other settings

<table>
<thead>
<tr>
<th>Other settings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wait for GC ready</td>
</tr>
<tr>
<td>Trigger GC</td>
</tr>
</tbody>
</table>

Wait for GC ready: If selected the instrument will wait for GC to be ready before starting the trap heat run.

Trigger GC: To select if data acquisition of trap heat run is required.

5.11.4 Utility: Canister Leak Test

The Canister Leak Test method enables the connections between the sample inlets on the CIA Advantage-xr and the sample canisters to be leak tested prior to starting an analytical sequence. These connections are not included in the system leak tests performed during the normal analytical sequence.

**NOTE** The canister valves should be closed throughout the leak test procedure to prevent sample dilution.

Standby settings

<table>
<thead>
<tr>
<th>Standby settings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standby split on</td>
</tr>
</tbody>
</table>

Standby split on: Set a standby split when using a mass spectrometer to ensure the system flow path is purged and background contamination limited. Typical flows are 10-20 mL/min

Leak test settings

<table>
<thead>
<tr>
<th>Leak test settings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow path temperature (°C)</td>
</tr>
<tr>
<td>Minimum carrier pressure (psi)</td>
</tr>
<tr>
<td>Leak test duration (min)</td>
</tr>
</tbody>
</table>

Flow path temperature: Temperature setting (in °C) for the entire thermal desorption flow path.

Minimum carrier pressure: The Minimum Carrier Pressure is the pressure below which the TD system will not be allowed to operate.
If the carrier gas pressure, as measured by the UNITY-xr pressure transducer, drops below this minimum pressure no further samples will be analysed until the pressure increases above this minimum threshold.

**Leak test duration:** Determines how much time the Leak test is going to be working.

5.11.5 *Utility: GC Blank*

This mode allows to trigger a GC blank during a sequence. No TD parameter is required.

5.11.6 *Utility: Sampling Line Flush*

This mode allows the flushing of the sampling lines.

### General

- **Standby split on:** Set a standby split when using a mass spectrometer to ensure the system flow path is purged and background contamination limited. Typical flows are 10-20 mL/min.

- **Flow path temperature:** Temperature setting (in °C) for the entire thermal desorption flow path.

- **Sampling line temperature:** Temperature setting (in °C) for the sampling line, i.e. line between the canister and the sample channel.

- **Sampling line flush:** Set the time (in minutes) for which each sample line will be flushed for.

5.11.7 *Utility: System Diagnostics*

The system diagnostics mode performs an automated check for system leaks and correct valve function. The method can be run at the start of a sequence, to ensure optimal system function prior to analysis. If no system deviations are detected, the sequence will continue to run and the successful completion recorded. If a system deviation is detected, the sequence will be stopped automatically and the user alerted to the deviation detected. In this event, detailed diagnostic logs can be found at:

C:\ProgramData\Markes International\TD\Logs\System Diagnostics [Date] [Time].txt
General

- **Standby split on:** Set a standby split when using a mass spectrometer to ensure the system flow path is purged and background contamination limited. Typical flows are 10-20 mL/min

- **Flow path temperature:** Temperature setting (°C) for the TD flow path.

- **Minimum carrier pressure:** The Minimum carrier pressure (psi) is the pressure below which the TD system will not be allowed to operate.

- **Trap setting:**
  - Trap setting
  - Trap low (°C)

The trap low temperature (°C) can be set either at room temperature or at the trap low temperature of the subsequent sample analysis.
6. Sequencing

The sequencer window may be accessed by clicking the sequence icon on the MIC home page (see Figure 24). The sequencer provides the means for listing the samples to be analysed, and the method to be used for each sample analysis.

![Sequencer icon](image)

**Figure 24: Sequencer icon**

The sequencer has two main screens, the ‘Edit’ view in which you can build and edit new sequences, even if the instrument is running, and the ‘Live’ view which shows you the currently running sequences and their status.

Navigate between the two views by left-clicking on the tabs at the top of the sequence screen.

![Sequencer Edit and Live views](image)

**Figure 25: Sequencer Edit and Live views**

The sequencer window is divided into three parts: the sequence table (Edit or Live), the instrument status bar and the instruments pane. The last two sections can be collapsed or expanded by clicking on the bars of the pane.

### 6.1 Edit view

The Edit view window is divided into three parts (see Figure 26): the sequence table, the instrument status bar and the instruments pane. The last two sections can be collapsed or expanded by clicking on the bars of the pane.
6.1.1 Edit view icons

<table>
<thead>
<tr>
<th>Icon</th>
<th>Name</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>🎧</td>
<td>Play</td>
<td>Opens the “Run sequence” dialogue so the sequence can be started.</td>
</tr>
<tr>
<td>🗑️</td>
<td>Clear sequence</td>
<td>Removes all samples from the sequence being edited.</td>
</tr>
<tr>
<td>🔐</td>
<td>Import sequence</td>
<td>Imports / opens a sequence from an existing file.</td>
</tr>
<tr>
<td>🇵🇺</td>
<td>Export sequence</td>
<td>Exports / saves the sequence to a file.</td>
</tr>
<tr>
<td>📋️</td>
<td>Number of rows to add</td>
<td>The number of rows to be added with the + or ‐ icons.</td>
</tr>
<tr>
<td>🔢</td>
<td>Undo</td>
<td>Undo the last command.</td>
</tr>
<tr>
<td>🔢</td>
<td>Redo</td>
<td>Re-apply the undone command.</td>
</tr>
<tr>
<td>➕️</td>
<td>Add rows</td>
<td>Adds the specified number of rows below the selected row and copies all fields.</td>
</tr>
<tr>
<td>➕️</td>
<td>Add rows and increment</td>
<td>Adds the specified number of rows below the selected row and increments numerical fields by 1. Non-numerical fields are copied.</td>
</tr>
<tr>
<td>➔️</td>
<td>Fill down</td>
<td>Fills the column / selected column section with the value in the first selected cell. If the whole column is selected fills with the value in the first cell.</td>
</tr>
<tr>
<td>➔️</td>
<td>Fill down and increment</td>
<td>Fills the column / selected column section and increments each line by 1.</td>
</tr>
</tbody>
</table>
6.1.2 Column headings

The column headings are divided in three categories:

- Permanent headings: always required in MIC sequencer, independent of mode, method or configuration.
- Configuration dependent headings: may be absent or present, depending on the instrument configuration.
- Mode dependent headings: may be absent or present, depending on the mode.
- Method dependent headings: may be absent or present, depending on the method.
- Unlocked parameter(s): only present if unlocked in the method.

If there are no samples in a sequence using a column heading, then that column will be hidden. When some samples don’t use a field that others do, the field will be disabled and crossed out.

If an ULTRA-xr is configured with a UNITY–CIA Advantage-xr, there is the option of having tube analysis and on-line sampling in the same sequence, as well as the option of re-collecting the sample from an on-line analysis onto a re-collection tube. Refer to the QUI-1120 UNITY-ULTRA-xr User Manual for more details on tube desorption.

<table>
<thead>
<tr>
<th>Heading</th>
<th>Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Row number</td>
<td>Permanent</td>
<td></td>
</tr>
<tr>
<td>Sample Type</td>
<td>Permanent</td>
<td>Specifies the type of analysis to run: sample, blank, unknown, standard or quality control.</td>
</tr>
<tr>
<td>Comment</td>
<td>Permanent</td>
<td>Free text field allowing user to store more information in sequence reports.</td>
</tr>
<tr>
<td>Method</td>
<td>Permanent</td>
<td>Specifies the instrument method.</td>
</tr>
<tr>
<td>Tube</td>
<td>Mode dependent</td>
<td>Specifies the tube to run.</td>
</tr>
<tr>
<td>Channel</td>
<td>Mode dependent</td>
<td>Specifies the number of the sampling channel.</td>
</tr>
<tr>
<td>Sample gas</td>
<td>Mode dependent</td>
<td>Specifies the type of gas of the sample.</td>
</tr>
</tbody>
</table>
| Re-collection Type | Configuration dependent | Specifies the type of re-collection required:  
|                  |                     |  
|                  |                     |  - None (no re-collection)  
|                  |                     |  - Tube (re-collects onto the tube specified in the re-collection tube column)  
| Re-collection Tube | Configuration dependent | Specifies the tube on which to re-collect the sample |

6.1.3 Creating and editing a sequence

**Populate first sequence line**

If a previous sequence is already loaded, it can be discarded by clicking on the delete button (\(\text{delete}\)) on the toolbar (note that the new sequence will not automatically overwrite the sequence that has just been deleted). To create the first line of a sequence, select first the type of sample to run by using the drop down menu in the Sample Type column of the sequence table (see Figure 27).
Select the instrument method to be used by double-left-clicking in the method field to open the “Select Method” window (see Figure 28). Select the required method and left-click <OK>.

The sequence table will now show the permanent headings, the configuration dependent headings as well as any headings for unlocked parameters in the method (Sample Volume in Figure 29).
Adding and manipulating sequence rows

Additional rows can be added by left-clicking on ▶ to add, or on ◀ to add-and-increment numeric fields. The number of rows to add can be selected by altering the number adjacent to the ◀ icon (◀). Rows can also be inserted (above selected), deleted, cut, copied and pasted by right-clicking on the required row(s) and selecting the appropriate option.

Multiple cells in the same column can be highlighted by holding down <Ctrl> or <Shift> whilst left-clicking on the required cells, or by left-clicking and dragging a selection. Clicking on the column title will select the whole column. Right-clicking on a cell and left-clicking “Select” brings up some more advanced selection tools (see Figure 30) such as select “All mis-matches”, which will highlight all the cells in the current column which do not match the selected.

To fill-down the highlighted cells or columns with the value in the uppermost cell, left-click on ◀. Numeric cells can be filled-down-and-incremented by left-clicking on ▶.

![Figure 30: Cell selection options](image)

Select columns, rows or cells

**Column**

A whole column may be selected by left-clicking the column header. All cells in the column are highlighted dark green.

A range of columns may be selected by holding <Ctrl> and left clicking multiple columns. All cells in the columns are highlighted green, with the last selected column in dark green.

**Row**

A whole row may be selected by left-clicking the row number. All cells in the row are highlighted green.

**Individual cell**

Individual cells may be selected by left-clicking inside the cell. The entire row will be highlighted green with the selected cell highlighted dark green.
6.1.4 Saving a sequence

A sequence can be saved to a file using the saving button in the top toolbar ( ). The “Save sequence as” dialogue box will open (see Figure 31). To save the sequence, choose a folder and a file name (either a new one or an existing one) and left-click on <Save>. Note that it is not necessary to save a sequence before running it.

![Figure 31: "Save sequence as" dialogue](image)

6.1.5 Opening a saved sequence

To open a previously saved sequence, left-click on the “Import sequence” icon on the toolbar. The “Open sequence file” dialogue box will be displayed. To open the sequence, browse to the required folder, select the required sequence and left-click on <Open>.

6.1.6 Running a sequence

To run a sequence, left-click on at the top left of the sequencer. The sequence will be validated and any warning or error messages will be displayed. Following this, a number of options for running the sequence will appear (see Figure 32). Select “Run a sequence a total of” to run the sequence more than once, entering the number of times or “Continuous” as required.

To start the sequence at a specific time, select “Delay Sequence Start” and define the date and the time at which the sequence is required to start.

If another sequence is already running (see Live view section 6.2), the new sequence will be added to the end. To insert the new sequence higher in the queue “Priority Sequence” can be selected to start the new sequence after the current sample or after the current sequence as required. The original sequence will resume after the priority sequence has completed.
6.1.7 Reports

A method report and a sequence report can be generated by right clicking anywhere in the sequence table and selecting ‘Method Report’ or ‘Sequence Report’. The Report Viewer window will open, displaying the appropriate report and the user can choose to print or save it (see example in Figure 33).

A method report contains information such as date of creation, author, comments, mode type, method parameters, etc. Note that unlocked parameters will not appear in the method report as their values can be modified in the sequence table, thus, the values used will be reported in the sequence report instead. A sequence report will contain information found in the sequence table, i.e. sample type, sample name, method name, unlocked parameters values, etc.
6.1.8 Accessing the Method Editor

The method editor can be accessed from the sequence table by right clicking on the method name and selecting ‘View method’. Note that if any changes are made and saved under a different method name, the sequence table will upload that method in the cell originally selected to access the method editor.

6.2 Live view

The live sequence view shows the live sequence queue, the status of each sample and any live reports specified by the user in the “Reporting” tab in the MIC settings (see section 8.2.4).

Multiple sequences may be added to the live queue and these are separated by a title bar which allows each sequence to be collapsed or expanded. The current sequence repetition number is displayed in the sequence title bar. The total number of sequence repetitions can be altered by editing the “Repetition” field or (de)selecting the “Continuous” checkbox.

![Sequence title bar](Figure 34: Sequence title bar)

6.2.1 Column headings

The column headings in the live view are the same as those in the edit view (see section 6.1) with the addition of the sample status column and any reporting options chosen by the user in the settings (see details in section 8.2.4).

6.2.2 Modifying live sequences

Whilst it is not possible to edit fields in the live sequence view it is possible to manipulate a queued sequence without stopping it:

**Skip**

- It is not possible to completely delete lines in a queued sequence however lines may be ‘skipped’ by selecting the line, right clicking and selecting the ‘Skip’ option. Only queued sample may be skipped, if the sequence has started working on a line or moved beyond it, it cannot be skipped.

  This sequence line will not run and the sequence will move on to the next line. Note that this could cause discrepancies between the MIC sequence and any other sequences controlling the gas chromatograph or detector.

**Add**

- It is not possible to append lines to a queued sequence but the same may be achieved by creating an additional sequence with the extra lines required and pressing play – this new sequence will be added to the live queue after the current sequence.

**Insert**

- Lines cannot be inserted into the queue from the live sequence view, to achieve this use a priority sequence (see section 6.1.6).
6.2.3 Sample status

The sample status column and the colour of the sequence lines will update as the instrument works through the sequence, please see below for a description of each status.

<table>
<thead>
<tr>
<th>Status</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>This sample is waiting in the queue; the status remains blank.</td>
</tr>
<tr>
<td>Waiting</td>
<td>Instrument is busy finishing a previous action before loading this sequence line e.g. the previous sequence has been stopped immediately and is unloading a tube when a new sequence is submitted.</td>
</tr>
<tr>
<td>Active</td>
<td>This sample is active, this means that the TD has started working on it; the specific stage of operation is detailed in the status window. Note: when overlapping samples, the ‘active’ line in MIC may not match up with the currently acquiring sample in the GC (MS) software.</td>
</tr>
<tr>
<td>Complete</td>
<td>All actions have completed for this sample. Please check the deviations columns for any minor errors that may affect your data.</td>
</tr>
<tr>
<td>Skipped</td>
<td>The user has skipped this sample and no action has been taken with it.</td>
</tr>
<tr>
<td>Warning</td>
<td>A deviation from the normal operation of the system has been detected that did not result in the sequence stopping (e.g. tube not found, leak test failed, etc.). Please see the deviations columns in the sequence history for more information.</td>
</tr>
<tr>
<td>Stopped</td>
<td>The sequence has been stopped by the user using the ‘stop immediately’ option.</td>
</tr>
<tr>
<td>Error</td>
<td>A hardware error has occurred causing the sequence to stop immediately. Please see the deviations column in the sequence history for more information on the error.</td>
</tr>
</tbody>
</table>

6.2.4 Deviations

Deviations are recorded when errors are encountered during operation that do not require the instrument to shut down but may affect your sequence or results. Deviations are displayed in the live sequence and sequence history if selected in the reporting options, see section 8.2.4.

6.2.5 Stopping a sequence

A sequence that is in progress can be stopped by left-clicking on the button in the top toolbar of the sequencer in the live view. A “Stop” box will be displayed, presenting several options for stopping the sequence (see Figure 35). Select the required option and left-click on <OK> to confirm.

![Stop sequence dialogue box](image-url)
6.2.6 Completed sequences

When the sequence queue completes the live view is cleared and a pop up message appears giving the option to view the sequence history.

```
Markes Instrument Control

All queued sequences have completed.
View sequence history now?

☑ Don't ask me this again

Yes  No
```

Figure 36: Sequence Completed Dialogue Box

The settings for flow path temperature, split flow and trap low temperature will be retained from the final method in the sequence until the standby timeout has expired, then settings from the standby method will be sent to the instrument. See section 8.2.3 for more information on standby settings.

6.3 Instrument status bar

The collapsible instrument status bar at the bottom of both the edit and live sequence views contains a copy of the instrument status dialogue (see section 9 for a full description) and a live schematic of the instrument that updates as the instrument moves through the sample. This schematic may also be accessed from the instrument tile (see section 10).

Figure 37: Instrument status bar
7. **Sequence History**

Information from historical sequences can be accessed by clicking on the sequence history icon on the homepage, or from a pop up message when sequences complete.

![Sequence history icon](image)

**Figure 38: Sequence history icon**

In the sequence history a copy of the sequence is stored which includes information such as tube / re-collection tube number, instrument methods and a free comments section. Any reporting options selected in the settings will also be displayed in the sequence history, such as deviations, trap fire time and peak desorption temperatures. Please see section 8.2.4 for more information on reporting options.

![Sequence history example](image)

**Figure 39: Sequence history example**

By default, only the most recent sequence will be displayed. To display previous sequences, left-click as many times as required on the <Older> button on the toolbar to show the previous sequence(s) or by select a range of dates in the toolbar and click on Search button (see Figure 40). Each sequence is collapsible by clicking on the arrow button in the sequence title bar (✔️) for easier viewing.

![Sequence history search](image)

**Figure 40: Sequence history search**

The user has the option to export the sequence history by clicking on the <Export> button in the sequence title bar. A window will pop up for the user to choose the location and the name under which to save the sequence history.
8. Tools

8.1 Instrument configuration

This section contains information on the configuration of the instrument; this should be set up during installation and should only be edited under instruction from a service engineer. An overview of the instrument configuration section is included below; a more detailed description of the instrument configuration can be found in the installation manual.

8.1.1 Configuration

![Instrument configuration](image)

**Figure 41: Instrument configuration**

**Instrument configuration**

The current configuration is shown in the box on the top right of the configuration tab and should include all Markes TD instruments and any optional accessories that are installed. Do not make any changes to this section unless instructed to do so by a trained service engineer.

**Communication ports**

The instruments listed in the Communication ports section can be used in simulation mode by selecting ‘Simulate’ from the corresponding drop down box. This will mean that the software is not actually controlling this instrument and samples will not run; however, the software will act as if it is connected to an instrument. This mode is typically used for training or for conveniently viewing methods, sequences or sequence history etc. when the instrument is offline.

**NOTE**

The correct com port is setup at installation by a service engineer, when selecting ‘simulate’ mode for an instrument it is important to note this com port before changing it so it can be changed back when the instrument is ready to run.
Options

In this section, the user can adjust the CIA Advantage-xr options:

- Heated sampling lines: select this check box to be able to heat the sampling lines. If unselected, the sampling lines will remain at room temperature.
- Sample loop (only available for CIA Advantage HL-xr): enter the size of the sample loop fitted on your CIA Advantage HL-xr.
- Connection: if no ULTRA-xr is configured, the user has the option to connect the CIA Advantage-xr to either the UNITY-xr tube side (check box unticked) or the UNITY-xr split side (the check box ticked).

8.1.2 Gas and flows

The gas and flows tab shows any electronic mass flow controllers (MFCs) detected by the software and the gas types and flow ranges that they are calibrated for, if you do not have MFCs on your instrument the message ‘No MFCs detected’ will be displayed. The carrier gas type is also selected here – do not select a carrier gas that is not listed under ‘gases available’ for your MFC.

8.1.3 GC interface

The GC interface tab contains the settings required for communicating with the GC, for the TD to receive a ready signal from the GC and send a start signal to it. This should be set up by the installation engineer and it should not be necessary to change these settings unless advised by a trained service engineer.
8.2 Settings

The settings section of MIC allows the user to customise the displays and standby settings.

8.2.1 Global Settings

The reports section of the global settings tab allows the user to customise reports generated by MIC to include company name, address and an image such as company logo.

The remainder of the global settings tab allows the user to decide whether they wish to be alerted by the software when performing certain actions.

For example, skipping a line in a sequence triggers a message reminding the user to adjust their data acquisition sequence accordingly. If you do not wish to see the message every time a sample is skipped, either click ‘do not show me this message again’ when the alert pops up or change the default action to ‘Always select: OK’ in the settings which prevents the message from being shown. Should you change your mind, this pop up message can be retrieved by changing the default action back to ‘Show Dialog’ in the settings.

![Global settings tab](image)

**Figure 44:** Global settings tab
8.2.2 Sequence Options

The Sequence Options tab contains options that effect how the sequence operates or is displayed. For UNITY-CIA Advantage-xr the options available are:

- To automatically export sequence report files (the files will be recorded as comma separated variable files in C:\ProgramData\Markes International\TD\Sequences).

- To set up the interval between samples. This option is applicable for on-line sampling only and allows the user to analyse samples at a specific interval.

  If the user chose the option ‘Specify time between sample lines’, a new column will appear in the sequence table where the user can enter the time interval desired.

  If the user selects the second option ‘Specify sampling start time’ then the user needs to select the interval time and the sampling start. For example, by entering a sampling start interval of 60 min and a sampling start time of 0 min past the interval, it means that a sample will be taken every hour on the hour. Another example, by entering a sampling start interval of 30 min and a sampling start time of 5 min past the interval, it means that two samples will be taken every hour at 5 min and 35 min past the hour.

- To ignore the GC cycle time when the GC becomes ready.

- To add additional column(s) into the sequence table (click on the button to add a column).

Figure 45: Sequence Options tab
8.2.3 Standby Settings

When MIC first starts up default instrument standby parameters are loaded to the instrument, these parameters set the flow path temperature, the trap temperature, turn the split on or off and if applicable set the split flow rate. The standby settings page allows the user to customise these settings.

Figure 46: Standby settings tab

**Standby timeout:** At the end of a sequence the instrument will retain the parameters from the final method in the sequence until the standby timeout has elapsed, then the standby settings will be applied.

**Use standby parameters from selected method:** Allows the user to nominate a method from the ‘All methods’ folder as the standby method; flow path temperature, trap temperature and split flow will be taken from this method.

**Use dedicated standby method:** Allows the user to specify standby parameters instead of selecting a method.

**Enable peltier timeout:** Turns the Peltier coolers off after the instrument has been in standby for the specified time period. This is a power saving feature that should be used when the instrument will not be used for several hours.
8.2.4 Sequence Reporting

The sequence settings tab contains a list of all the status information available for the configured instruments. From here the user is able to select the information most relevant to them to show in the live sequence and sequence history.

All of the information will be recorded regardless of this selection but these options allow the user to simplify the live sequence view and show only the information important to them. Example live reporting options include deviations, sampling start and end times and sample volumes temperature.

![Figure 47: Sequence Reporting tab](image)

8.2.5 Method Limits and Counters

The Method limits and counters tab allows the user to set lower and upper values for different method parameters and also offers a range of counters.

The user is not allowed to enter a value outside the valid range when creating a new method. In case of a method previously created with parameters outside the new valid range, the sequence will fail the validation and a warning message will pop up. The sequence will not run until the user decides to either carry on the sequence despite the outside parameter(s) or to abort the sequence.

Restricting the temperature range can be very useful for example to avoid overheating a sorbent (present in the trap or in the analytical tube) as it may result in contamination of the flow path. Restricting the upper limit of the trap high temperature and/or the tube desorption temperature will limit this risk.

The counters help track the need for maintenance or replacement of the trap. Setting up appropriate warnings will help scheduling a maintenance or ordering a replacement trap in advance, both limiting the risk of downtime. Note that the user will need to manually clear the trap counter every time a new trap is installed and that only a trained engineer can reset the maintenance counter.
Figure 48: Method limits and counters tab
9. Live status

The instrument status dialogue (see Figure 49) may be accessed two places: from the instrument status bar in the sequencer or by left clicking on the instrument tile. When left clicking the instrument tile the instrument status dialogue can be released by dragging the window to the desired location. If you wish to view the status dialog box above other panes right click anywhere in the status dialogue to lock the dialogue box in ‘always on top’.

The status of the instrument is displayed in the top right corner of the dialogue box, see tile status section 4.4 for detailed descriptions of these states.

The main body of the box shows live-updating textual reading of all the instrument parameters and their set points.

A dashed line in the set column represents a parameter that is not currently active or is not set-able, only monitored. For example the tube oven heated zone is not active in ‘idle’ so is shown as a dash. All pressures states within the system are read back only and will always be shown with a dashed line in the set column.

The title bar of the status dialogue shows more information on the overall instrument status (see Figure 50 - 54) and the GC ready status. A green tick next to GC ready indicates that the GC is ready to receive sample from the TD. A red tick indicates that it is not. Depending your method settings the TD may or may not ‘Wait for GC Ready’ before proceeding to certain states.

![Figure 49: Instrument status dialogue](image-url)
**Idle:** If the instrument has been in ‘idle’ for longer than the Peltier cooler timeout the instrument status dialogue will display ‘UNITY peltier timeout’, if the purge gas is turned off or at a pressure too low for the instrument to operate ‘UNITY PURGE GAS OFF’ will be displayed.

![Figure 50: Instrument status dialogue: Peltier timeout](image)

**Offline:** If the instrument tile status is ‘offline’ more information will be available in the instrument status dialogue as to which instrument the software cannot communicate with.

![Figure 51: Instrument status dialogue: offline](image)

**Active:** If the instrument tile status is ‘Active’ the instrument is either running a sequence or being controlled via the direct control mode (see section 10.2) for more information on the direct control mode. The instrument status dialogue will indicate give more information on which stage of operation the instrument is currently performing as well as giving live updates on the temperatures and flows.

In Figure 52 the instrument is active and performing the Trap Purge stage of operating mode, the elapsed time clock shows how long the instrument has been in the current stage.

![Figure 52: Instrument status dialogue: Active](image)

In Figure 53 the instrument is active and has been locked by the direct control mode. It is not possible to run a sequence until direct control mode has been closed.

![Figure 53: Instrument status dialogue: Direct Control](image)

**Error:** If the instrument tile status is ‘Error’ more information regarding the nature of the error can be found in the instrument status dialogue, for example if a problem with a heated zone has caused the instrument to enter the error state the heated zone at fault will be identified (see Figure 54).

![Figure 54: Instrument status dialogue: Error](image)
10. Markes TD Utilities

There are several TD utilities and routines available within MIC; these can be accessed by right clicking on the instrument tile and selecting from the menu.

10.1 View Schematic

Opens a new window containing a live-updating schematic of the UNITY–CIA Advantage-xr it is not possible to manipulate valves and flows from this schematic it is simply a representation of the current instrument state.

10.2 Direct Control

Opens the direct control utility, this allows the user to take direct control of the instrument without running a method. It is possible to open and close valves and set MFC flows from here.

Please note it is only possible to open direct control when the instrument is idle.

The direct control utility consists of a collapsible, live updating instrument status dialogue on the left hand side, an interactive instrument schematic in the centre and a set of action buttons on the right hand side.

Figure 55: Direct control utility layout

10.2.1 Instrument status dialogue

A set of instrument set points and read backs – please see section 9 for a full description.

10.2.2 Interactive instrument schematic

Figure 56 details the main components of the interactive instrument schematic. Green lines represent carrier gas flow.
Pressure transducer: Reads carrier gas pressure at these points, does not control pressure.

Solenoid valve: Opens and closes to control the flow of carrier gas around the instrument. Green lines through the valve represent carrier flow through the valve.

Mass Flow Controller: Controls the flow rate of gas through the split and trap vents.

Rotary valve: Open and close to control the flow of carrier gas around the instrument. Green lines through the valve represent carrier flow through the valve.

Stream selection valve: Set the stream selection valve to any position, or move it through one position at a time.

**Schematic actions**

**Solenoid valves:** Left click on solenoid valves to open and close them and allow carrier gas around the flow path of the instrument.

**Mass flow controllers:** Left click on a mass flow controller to set the flow, a dialogue box will pop out – enter the desired flow here and click <OK>.
**UNITY-xr heated valve:** The UNITY-xr heated valve has two independently moving pins that direct flow around the instrument and isolate other parts to achieve each stage of the operating modes. To move the pins left click on the heated valve and select an option from the dialogue box.

**Top In, Bottom Out**
Sets the heated valve for ‘standby’, the CIA Advantage-xr (or the sample tube if an ULTRA-xr installed and configured) is isolated from the carrier gas supply, the cold trap and the GC transfer line.

**Top Out, Bottom In**
Sets the heated valve for ‘tube desorb’, the CIA Advantage-xr (or the sample tube if an ULTRA-xr is installed and configured) is connected to the cold trap but isolated from the GC transfer line – allowing the sample to be trapped onto the cold trap.
**Top in, Bottom in**
Isolates the cold trap from both the CIA Advantage-xr and the GC transfer line.

**Top out, Bottom out**
Connects the re-collection tube, trap and GC transfer line when an ULTRA-xr is installed and configured. Allows carrier gas to flow from the focusing trap through the re-collection tube in the desorption direction to re-collect the sample onto the re-collection tube.

**Rotary valves:** Left click on rotary valves to switch between their two positions.

**Stream selection valves:** Left click on a stream selection valve to set its position, a dialogue box will pop out – select the position required from the drop down list or nudge the valve by one position using the icon. Click on <Cancel> when finished.

Figure 59: Stream selection valve dialogue
10.2.3 Actions list

Return to Standby: Returns the heated valve, all solenoid valves and MFC flow rates to the standby setting.

Exchange split tube off: Sets the valves so that the charcoal split filter may be removed and replaced without causing a leak.

Peltier cooler off: Turns off the Peltier coolers. Use this when the instrument will be in standby for an extended period.

Manual leak test

Pressurize 1/2: First step to pressurise the system for leak testing. Sets the valves to pressurise all the flow path apart from the CIA Advantage-xr canister vent.

Pressurize 2/2: Second step to pressurise the rest of the system for leak testing. Sets the valves to pressurise the CIA Advantage-xr canister vent.

Leak Test: Sets the valves for leak testing. In this state the pressure gauges shown in the instrument status should not rise or fall if the system is leak tight.

Depressurize: Drops the pressure in the instrument flow path through the split vent.

Check gas flows

Check trap flow: The solenoid valves open or close in order to get carrier gas flow through the trap only and the trap MFC is set to 20 mL/min. The user should check that the actual UNITY trap flow matches the set flow.

Check split flow: The solenoid valves open or close in order to get carrier gas flow through the split tube only and the split MFC is set to 20 mL/min. The user should check that the actual UNITY split flow matches the set flow.

Check CIA vent flow: Sets the valves to get carrier gas flow through the loops to the sample vent and the vent MFC is set to 20 mL/min. The user should check that the actual CIA vent flow matches the set flow.

Check IS flow: The IS solenoid valve opens to get carrier gas through the IS loop to the IS vent. The user needs to manually check that there is flow coming out of the vent and needs to adjust the IS needle valve to the required flow.

10.3 Diagnostics

The diagnostics section of the software is read only to users, an engineer user name and password is required to modify any files in this section. Diagnostics shows the calibration values for heated zones, motors, mass flow controllers and pressure transducers associated with all of the configured Markes TD instruments.
10.4 Flash Firmware

This allows the instrument firmware to be updated, this is another utility that should not be accessed unless under instruction by a trained service engineer.

**CAUTION** Flashing the instruments with the wrong firmware may result in a chargeable service engineer visit.

10.5 System Info

Contains information on the instrument configuration, firmware version and calibration values.
11. Routine maintenance

11.1 Removing the instrument covers

To remove the UNITY-xr instrument covers:

1. Remove the front top cover by pulling it up vertically.

2. Remove the heated valve shield by removing the two screws shown and then pulling it forwards and upwards.

3. Remove the back cover by sliding it backwards.
4. Remove the black pneumatics cover by removing the highlighted screws and pulling upwards.

5. Remove the white front cover by pulling forwards.

6. Remove the side covers by removing the highlighted screws and sliding the instrument side panel towards the front of the instrument then lifting away.
11.2 Replacing the tube seals and filters

The interface link tube and split tube are sealed into the instrument flow path with size 010 Viton O-rings. Each O-ring should last for >1000 tube-sealing operations but when the system leak test starts to repeatedly fail then it is likely that these O-rings have deteriorated and will need to be replaced.

To replace the tube seals and filters:

1. Turn off the instrument, disconnect from the power supply and allow to cool if necessary. Once cool, turn off the carrier gas supply.

2. Gently remove the O-ring using the O-ring extractor tool supplied as part of the installation toolkit.

   **CAUTION**
   Be careful not to scratch the surrounding tube receiver, any damage may result in a permanent sealing problem that will require a service visit and replacement parts.

3. At this stage it is advisable to also replace the small PTFE filter that sits behind each O-ring, use the O-ring extractor tool to pull the filter out.

4. Inspect the tube receiver to ensure that it is clean and if necessary remove any small particles or fibres that may prevent a perfect seal with the new O-ring.

5. Using the O-ring insertion tool, replace the filter at the back of the tube receiver and the size 010 Viton O-ring, ensuring that it is neatly seated in its groove.

6. Insert the split tube and push the red lever down to seal the system.

7. Connect the instrument power supply, switch on, open the MIC software and perform a leak test to ensure the new O-ring seal is leak tight.

11.3 Replacing the charcoal split filter

When not re-collecting the split effluent for subsequent analysis, a charcoal filter tube will trap the split effluent to prevent it being vented to the atmosphere. These filters can become saturated and will require changing at routine intervals.

It is suggested that the filters are repacked with conditioned charcoal every three months. Charcoal filters are loaded in the same manner as tubes, with the grooved end (or fritted end of the glass tube) facing the back of the UNITY-xr.

For easy removal of the split tube, isolate the carrier gas flow by left clicking on the Exchange split tube button within Direct Control. Lift the black knob of the sealing mechanism and use the Tube Extractor tool to grip the charcoal tube and remove it from the seal.
11.4 The Focusing (cold) trap

The UNITY-xr quartz focusing trap is packed with a 6cm long sorbent bed made up of one or multiple sorbents as shown in Figure 60. A range of focusing traps containing different sorbents combinations are available to buy from Markes.com.

![Figure 60: Focusing (cold) trap](#)

The lifespan of a cold trap is dependent on its usage and the temperature at which it is routinely taken to, however it is recommended that the cold trap is replaced at least once a year. When a trap has reached the end of its lifespan or a different type of trap is required, the user is able to carry out this replacement.

11.4.1 Replacing the focusing (cold) trap

Scan this QR code to watch our video tutorial on how to replace the focusing trap on a UNITY-xr.

Or go to: [http://chem.markes.com/UNITY-Trap](http://chem.markes.com/UNITY-Trap)

1. Turn off the instrument, disconnect from the power supply and turn off the carrier gas supply.
2. Loosen the steel fitting attached to the cold trap.
3. Loosen the screw at the bottom of the trap pneumatics assembly, gently pull the assembly towards the front of the instrument.

**CAUTION** Keep a firm hold on the trap pneumatics whenever it is not secured in place with the screw - if it is allowed to twist it can snap the cold trap.

4. Once clear of the screw, pull the pneumatics up and off the brass guiding block and rotate to the right, clear of the cold trap.

5. Grip the glass collar and remove the cold trap by gently pulling, a rubber laboratory glove will provide a better grip.

This can sometimes prove difficult with a cold system – if so, switch the instrument back on to warm up before switching off again and reattempting to withdraw the trap.

**CAUTION** Be careful not to apply excessive force to the quartz cold trap tube.

6. Remove the new cold trap from its packaging and holding onto the trap as close as possible to the cold trap box, gently push the trap into the cold trap box.

If in doubt practice with the trap alignment tool supplied in the shipping kit.

**CAUTION** Never switch the UNITY-xr on with the trap alignment tool installed.

7. You will feel increased resistance as the cold trap pushes into the O-ring seal at the valve end. Once the trap is properly inserted the glass collar should be approximately 2 mm from the black plate.

8. Bring the pneumatic assembly back down onto the brass guiding block and relocate the screw in the slot, push the assembly gently back in the horizontal plane, taking care to align the trap and stainless steel connector.

**CAUTION** Misalignment can cause the end of the quartz trap to snap.

9. Apply gentle steady pressure to push the trap into the sealing O-ring located inside the steel trap connector. If the pneumatics assembly does slide easily onto the cold trap remove the trap pneumatics and check seating of the o-ring.

Keep hold of the pneumatics while re-tightening the screw firmly.

10. Gently tighten the steel fitting attached to the cold trap until slight resistance is felt.
11. Connect the instrument power supply, switch on, open the MIC software and perform a leak test to ensure the new O-ring seals are leak tight.

11.4.2 Removing/Replacing a broken cold trap

1. Turn off the instrument, disconnect from the power supply and allow to cool if necessary. Once cool, turn off the carrier and purge gas supply.
2. Remove the visible end of the cold trap using the method described in section 11.4.1.
3. Remove the split tube and any sample tubes / interconnects.
4. Remove the white front cover and the most convenient side cover from the instrument.
5. Remove the four (two on top and two below) purge gas lines that activate the heated valve by pulling the white/black tubes while pressing in the silver lip of the connector.

6. Disconnect the thermocouple/heater wire connectors of the heated valve assembly and the brass bypass line highlighted.
7. Loosen the 4 screws highlighted, then slide the whole heat valve assembly back in the frame to allow access to the brass connector. Hold in this position by tightening one of the rear screws.

8. Carefully peel back the insulation surrounding the brass connector and using a 7/16 " spanner if necessary, loosen it slightly to remove the broken trap with an appropriate tool.

9. Re-tighten the brass connector without the use of a spanner, as it does not actually seal the cold trap it is only required to be finger tight.

10. Feed the trap alignment tool through the cold trap box to ensure any broken glass is removed.

11. Loosen the screw holding the heated valve assembly in place and slide forwards again.

12. Retighten the 4 screws.

13. Place the trap alignment tool into the cold trap box.

14. The tool should pass smoothly up to the o-ring in the heated valve before further gentle pressure moves the tool another 1-2 mm.

   **CAUTION** If the tool does not move smoothly past the point where it enters the brass connector there may be a misalignment problem. If this is the case loosen the 4 screws again and ensure the assembly is pushed up against the cold trap box.

15. Reconnect the thermocouple/heater connectors and the brass bypass line.

16. Install a new cold trap as shown in section 11.4.1.
11.4.3 Replacing the cold trap seal and filter

At the heated valve end of the cold trap there is a Viton O-ring that should only be replaced by a qualified service engineer. However, at the pneumatics end of the cold trap there is a size 006 Viton sealing O-ring and a PTFE filter that can be easily replaced by the user when necessary.

1. Turn off the instrument, disconnect from the power supply and turn off the carrier gas.
2. Loosen the steel fitting attached to the cold trap.

3. Loosen the screw at the bottom of the trap pneumatics assembly, gently pull the assembly towards the front of the instrument.

   **CAUTION** Keep a firm hold on the trap pneumatics whenever it is not secured in place with the screw - if it is allowed to twist it can snap the cold trap.

4. Once clear of the screw, pull the pneumatics up and off the brass guiding block and rotate to the right, clear of the cold trap.

5. Fully unscrew the steel fitting on the pneumatics assembly and gently remove the O-ring and PTFE filter using the O-ring extractor tool supplied as part of the installation tool kit.

6. Using the O-ring insertion tool, replace the filter and the size 006 Viton O-ring, ensuring that it is neatly seated in its groove.

7. Bring the pneumatic assembly back down onto the brass guiding block and push the assembly gently back in the horizontal plane, taking care to align the trap and stainless steel connector.

   **CAUTION** Misalignment can cause the end of the quartz trap to snap.

8. Apply gentle steady pressure to push the trap into the sealing O-ring located inside the steel trap connector. If the pneumatics assembly does slide easily onto the cold trap remove trap pneumatics and check seating of the o-ring.

9. Keep hold of the pneumatics while re-tightening the screw firmly.

10. Gently tighten the steel fitting attached to the cold trap until slight resistance is felt.
11. Connect the instrument power supply, switch on, open the MIC software and perform a leak test to ensure the new O-ring seals are leak tight.

11.5 Replacing the fused silica insert of the transfer line

Compounds are transferred from the top of the UNITY-xr heated valve to the GC column through a length of inert fused silica (and protective sleeving) held at high temperature within the transfer line. When making new connections to a different GC column or to the heated valve, this silica requires trimming and over time can become too short and need replacing.

To replace the fused silica insert of the transfer line:

1. Turn off the instrument, disconnect from the power supply and allow to cool if necessary. Once cool, turn off the carrier gas supply.

2. Unplug the 2 transfer line connectors highlighted from the top deck of the instrument.

3. Remove the four highlighted nuts securing the transfer line.
4. Pull the transfer line up a few centimetres to reveal the transfer line nut shown. Remove this nut to disconnect the transfer line.

5. Disconnect the other end of the silica insert from the GC column and pull the insert (and its protective PTFE sleeve) through the transfer line into the GC oven.

6. Prepare the new transfer line insert by trimming the PTFE protective sleeve to ensure ~20cm fused silica is exposed at either end.

7. Connect the one end of the insert to the GC column with an appropriate connector. Push the other end of the insert up through the transfer line until it emerges the castellated end of the transfer line.

8. Place the 1/16 x 0.4 mm ferrule onto the union then thread the 1/16-inch stainless steel Swagelok type nut on loosely.

9. Pull ~ 20 cm of fused silica from the PTFE line casing. Cut off the first few mm of fused silica using an approved capillary cutting tool.

Make a mark 20 mm from the end of the fused silica using typing white-out fluid or an alternative marker.

Gently feed the fused silica through the 1/16” nut and ferrule until the mark is reached. Tighten the nut to trap the fused silica and then tighten a further 1/2 turn using one of the 8 mm wrenches (spanners) provided in the shipping kit.
10. Carefully bring the clamp plate, PTFE plate and shield tube down into the position with the shield tube covering the union nut.

The shield tube should be positioned such that the 1/16\(^{th}\) side tubing projects through one of the cutouts.

11. Secure the transfer line to UNITY-xr using the four nuts shown.

12. Plug the 2 transfer line connectors highlighted into their respective sockets on the top deck of the instrument.
### 11.6 Useful part numbers

<table>
<thead>
<tr>
<th>Part description</th>
<th>Part number</th>
<th>Pack size</th>
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</thead>
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<tr>
<td>Charcoal filter (split tube for UNITY-xr)</td>
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</tr>
<tr>
<td>Charcoal filter (glass split tube)</td>
<td>SERAAA-1600</td>
<td>1</td>
</tr>
<tr>
<td>Fused silica transfer line insert</td>
<td>SERUTD-5093</td>
<td>1</td>
</tr>
<tr>
<td>Humidifier, for humidification of purge gas</td>
<td>U-HUMID</td>
<td>1</td>
</tr>
<tr>
<td>Heated sampling line (1.8 m length) for CIA Advantage HL and CIA Advantage Satellite</td>
<td>U-HTLNKT</td>
<td>1</td>
</tr>
<tr>
<td>Heated sampling line (1.8 m length) for CIA Advantage T</td>
<td>U-HTLNKT-T</td>
<td>1</td>
</tr>
<tr>
<td>PTFE filter disc (for UNITY-xr), 5.1 mm</td>
<td>U-DISK1</td>
<td>10</td>
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<tr>
<td>PTFE filter disc (for CIA ports), 3.2 mm</td>
<td>U-DISK4</td>
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<td>Routine maintenance kit for CIA systems</td>
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