M-CTE250(T)(i)™

Micro-Chamber/Thermal Extractor Operator Manual

Version 1.12

February 2012

CONTENTS **PAGE**

1. Preface

This manual provides detailed instructions on the use of the Micro Chamber/Thermal Extractor M-CTE250 system. It is suitable for users with little or no prior experience of the system, and details the software/hardware interface and procedure for first sample runs.

I. Warnings

- When moving the instrument ensure it is supported from underneath, it is not designed to be lifted by the covers.
- 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.
- Ensure that the plug (electrical isolator) can be easily and quickly accessed during use.
- This instrument must be earthed.

II. Technical Specifications

Physical		**Electrical**	
Height (cm/inches):	41.5/16.3	Maximum Power (W):	650
Width (cm/inches):	16/6.3	Line voltage:	115-230
Length (cm/inches):	52/20.5	Frequency (Hz):	50-60
Mass (kg/lbs):	15/33	Input inrush current (A):	40 (cold start)

III. Environment 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.

Temperature: Recommended operating ambient temperature range is 15 to 30°C.

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

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

IV. Safety alerts

Warning: Precautions must be taken when preparing liquid samples. These should be prepared away from the Microchamber, with care taken not to allow liquid to be split into the chamber manifold. It is recommended that liquid samples are placed into a PTFE sample plate, glass dish or aluminium boat and not directly into the Microchamber. Liquids for use in permeation devices should be injected into the permeation device, sealed and then placed into the Microchamber manifold. If a spillage does occur switch the Microchamber off. For further information or assistance please call Markes Technical support on +44 (0)1443 230935.

The 'CAUTION – HOT SURFACE' symbol indicates a burn hazard. Make sure the instrument is at room temperature before touching, or you may incur burn injuries.

If you have a version equipped with toggle valves for switching off unused chambers please take care not to trap your fingers when actuating the toggles.

V. 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.

Address:	Markes International,Gwaun Elai Medi Science Campus, LLANTRISANT, RCT United Kingdom CF72 8XL		
Website:	www.markes.com	E-Mail:	enquiries@markes.com
Telephone:	+44 (0) 1443 230935	Fax:	+44 (0) 1443 231531

CRITICAL INFORMATION - Please Read

Critical information with regards O-ring seals on your M-CTE250 Microchamber-Thermal Extractor

Two different types of pot-seal O-ring are available for the M-CTE250 Microchamber-Thermal extractor:

- **Black UltrA High Purity (UHP) O-rings**: suitable for temperatures up to 250°C – high cost
- **Brown O-rings**: only suitable for temperatures up to 200°C – low cost

Warning: DO NOT run the M-CTE250 at temperatures higher than 200 °C if you are using the ***brown*** low temperature O-rings as they will thermally degrade, stick to the lid and pot and will leave a deposit that requires physical abrasion or chemical means to remove.

M-CTE250 systems are supplied with five of the (***black***) UHP O-rings; they are easily distinguishable when packed as they come individually wrapped. Once opened however they can only be distinguished from the (***brown***) lower temperature O-rings by their colour.

Please note that there is a *significant price difference* between the two types of O-ring with the *brown* ones that are suitable for temperatures below 200 °C costing considerably less than the *black* UHP ones.

Care should be taken when closing the M-CTE250 lids to avoid pinching and damaging the supplied *black* UHP O-rings – which are suitable many thermal cycles.

When sampling is complete the lids should be opened immediately to allow the chamber and O-rings to cool. If the O-rings cool with the chamber sealed they can stick to the chamber lid and tear and also become distorted in shape.

Both types of O-ring are available from Markes International or your local Markes distributor, part numbers are as follows:

Part number	Description
M-MCHOR250HT	Pk 4 Ultra High Purity O-rings (suitable for use up to 250°C)
M-MCHOR250	Pk 20 Low temperature pot-seal O-ring (only suitable for use below 200°C)

Note: The appropriate o-rings must be fitted to all 4 of the microchamber pots before first use otherwise the system won't be leak tight.

2. Introduction

The Micro-Chamber/Thermal Extractor (M-CTE250™) from Markes International provides industry and researchers with a versatile and automated tool for testing materials.

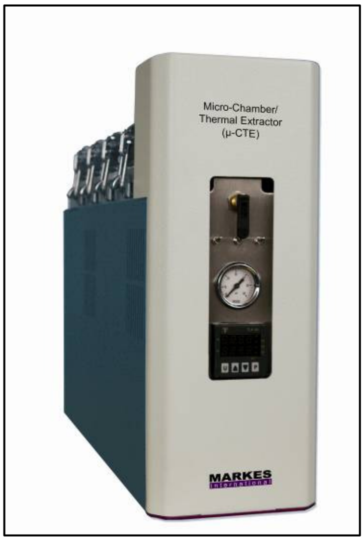
Key applications include:

- Testing emissions from material surfaces at low temperatures for:
    - correlation with data from conventional emission chambers/cells
    - intercomparison of products within a range (*e.g.* different colours/patterns)
    - testing prototype, low-emission materials
    - monitoring product uniformity in-between formal certification tests
- Testing VOCs and semi-VOCs (SVOCs) in bulk materials for routine quality control
- (content and emissions testing)
- Testing vapour permeation into and through materials at various temperatures
- Flavour and Fragrance profiling

The M-CTE250 is well suited to providing greater representation profile of emissions from heterogeneous samples, as well as low trace level component detection. This is because of the high temperatures that the chambers are capable of reaching, and large sample size that can be accommodated within each one.

2.1. Summary of Operation

The system (figure 1) comprises four microchambers (each 36 mm deep and 64 mm in diameter), which allow surface or bulk emissions to be tested from up to four samples simultaneously. The M-CTE250 is compatible with a range of sampling tubes and multiple standard analytical methods.



Conditioned sorbent tubes are attached to each microchamber and a controlled flow of air or inert gas is passed through all four chambers simultaneously. (S)VOC vapours are swept from the sample material in the micro-chamber and onto the attached sorbent tube. After sample collection, trapped vapours are thermally desorbed and analysed by GC(/MS)as per standard methods.

The thermal desorption analytical process is carried out off-line allowing a fresh set of samples to be introduced to the M-CTE250 even while analysis of vapours emitted by the previous set of samples is being performed. This also facilitates chemical analysis by third party laboratories.

Figure 1: The Micro-chamber/Thermal Extractor (M-CTE250)

Alternative analysers, combining thermal desorption with process MS or ENose detectors, are also applicable in some cases - particularly during quality control of fragrance/odour of foods and consumer products.

3. System Installation and Set-up

3.1. Pre-installation

3.1.1. Services Required

3.1.1.1. Power

The M-CTE250 is automatically compatible with all conventional mains power supplies ranging from 115 to 230 V and 50 or 60 Hz. It is not necessary to manually select or switch voltages. The maximum power consumption is 650 W.

3.1.1.2. Laboratory requirements

The M-CTE250 occupies minimal bench space, (52 cm x 16 cm) and requires sufficient space to allow unhindered access to all of the microchambers.

As the M-CTE250 is a powerful concentrator of VOCs and is often used to determine trace levels of organic analytes, it is advisable to store and operate the M-CTE250 in a clean laboratory environment with minimal atmospheric concentrations of organic vapours.

3.1.1.3. Gas supply

The M-CTE250 requires a pressure regulated supply of clean air, nitrogen or helium, between the ranges of 10-60 psi, as a carrier gas through the microchambers.

As the M-CTE250 is a concentrator, even trace level contaminants in laboratory gas lines can become significant interferents in the sample obtained. It is recommended that the gas line be constructed of refrigeration-grade copper tubing connected using approved swage-fittings. Laboratory gas line joins and connections must never be brazed. Position the gas supply as close to the M-CTE250 gas inlet (Figure 2) so that the gas lines are as short as possible. Use a high quality, stainless steel diaphragm cylinder head regulator for the gas supply. The cleanliness of the gas and supply can be validated prior to installation of the M-CTE250 (see section 3.3.1).

3.2. Unpacking the M-CTE250

Remove the instrument from its packaging and inspect the contents. Check every item against the packing list included. Retain the instrument packaging and re-use if ever the system is to be shipped using conventional carriers.

3.2.1. Installation - tools required

To complete the installation you will need the following tools: 7/16” wrench/spanner

3.3. Installing the gas lines

Unique technology maintains a constant flow of air or gas through each sample chamber at any given backing pressure, independent of sorbent tube impedance and whether or not a sorbent tube is attached. No pump or mass flow controller is required.

3.3.1. Checking the cleanliness of the gas supply

It is recommended that the gas supply and lines are checked for cleanliness prior to using the M-CTE250. This can be quickly and easily checked by sampling ~10 L of gas through a conditioned Tenax tube and then analysing the tube using a Markes UNITY GC(-MS/FID) system. Artefact levels of less than 30 ng total compounds are generally acceptable depending on the type of application to be undertaken. If the gas supply is not sufficiently clean then a suitable filter should be placed in-line immediately prior to the M-CTE250. The cleanliness of supply should be re-checked after fitting the filter.

3.3.2. Connecting the gas supply to the M-CTE250 inlet

The gas supply should be connected to the inlet found on the rear of the M-CTE250 (figure 2).

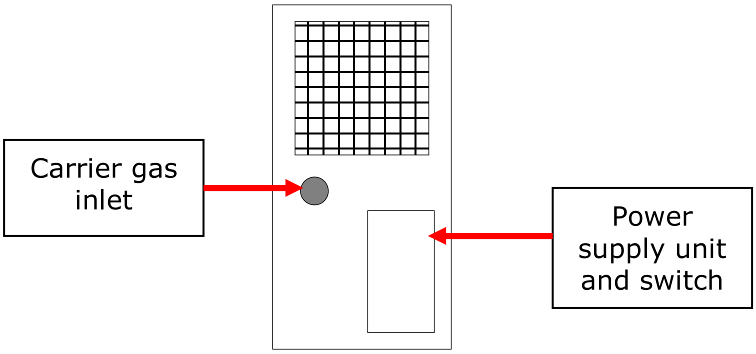


Figure 2: M-CTE250 rear view schematic

A common gas inlet controlled from the front of the instrument can accommodate two ranges (high, which is 10-500 $ml.min^{-1}$, and low, which is 10-70 $ml.min^{-1}$). In both cases, a gas backing pressure of between 10 and 60 psi must be maintained to ensure flow stability of the gas particularly at the lower flow rates.

The flow rate is determined by measuring the flow (with an appropriate flow meter) from the back of a sorbent tube inserted into the lid of the M-CTE250 with the lid closed (figure 3). The choice of sorbent tube used for setting flow rates will ideally be the same type of tube being used during sampling from the M-CTE250. The flow rate is adjusted by regulating the carrier gas pressure. The flow rate can be set with the M-CTE250 heated to the desired temperature if required.

Note: if you have a system equipped with toggle valves for switching off unused chambers (M-CTE250**T**(i)) the flow for the chambers in use MUST also be set/determined with the unused chambers switched off.

3.4. Connecting the M-CTE250

Connect the power cable supplied to the power socket on the rear of the unit (figure 2) and turn the power switch on.

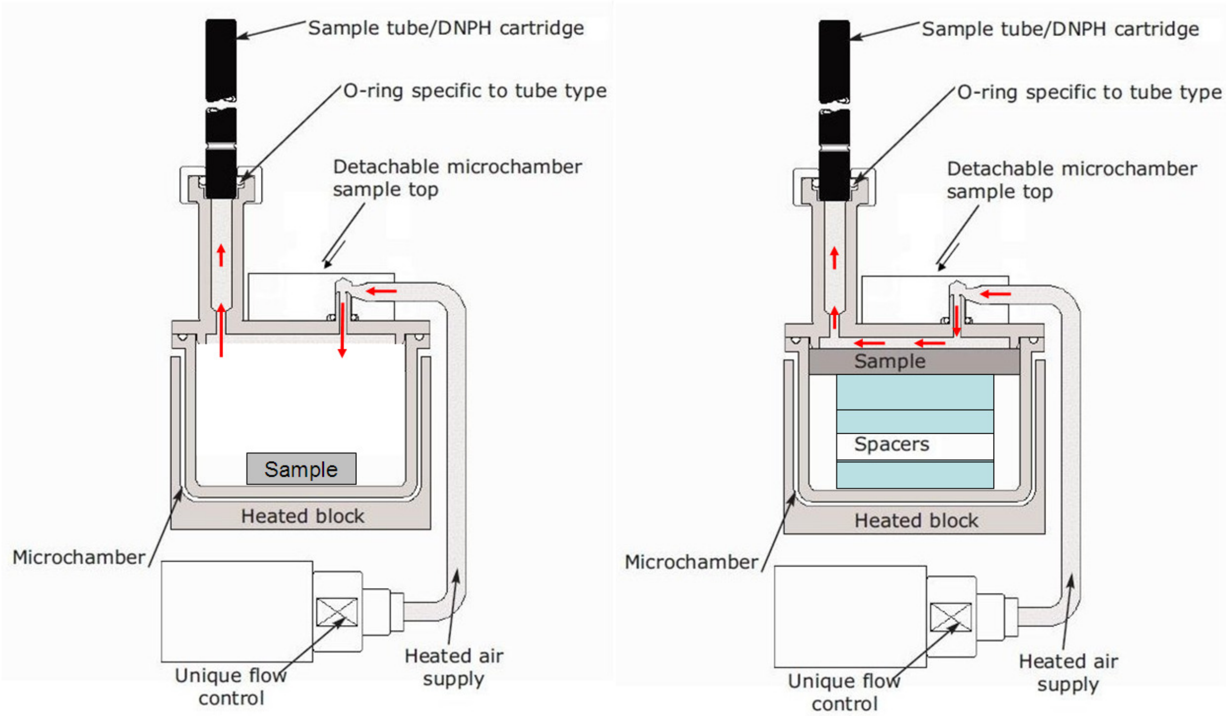


Figure 3: Schematic showing where to put tube and showing the setup for bulk emission testing (left) and surface emission testing (right)

3.5. Setting the temperature

Heating of all chamber components *e.g.* sample pans, chamber lids, air/gas supply tubing, is controlled by the temperature controller on the front panel of the instrument (figure 4).

Depending on the requirements of the application, microchamber sampling can be performed at ambient or elevated (up to 250°C) temperatures.

Once the M-CTE250 is switched on, the temperature controller should be lit up and displaying the current temperature set point in green and the actual temperature in red (figure 5).

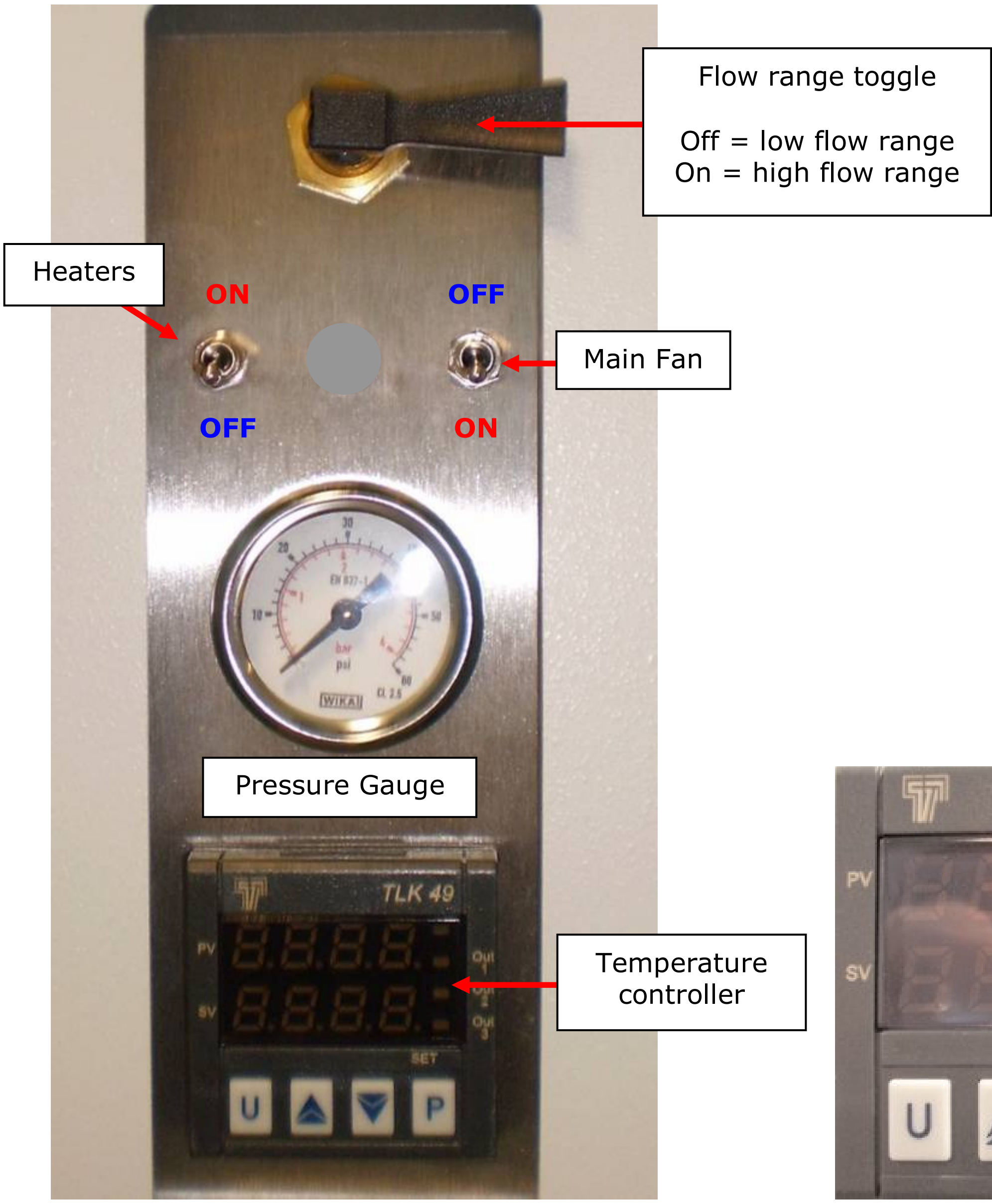
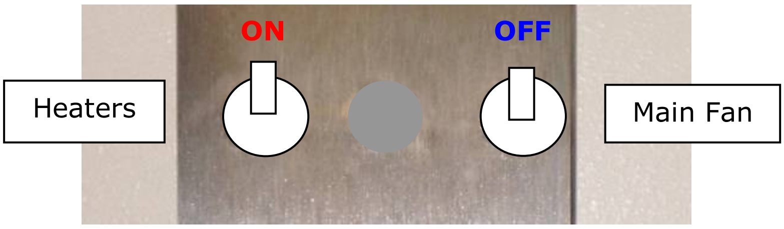


Figure 4: Front of M-CTE250

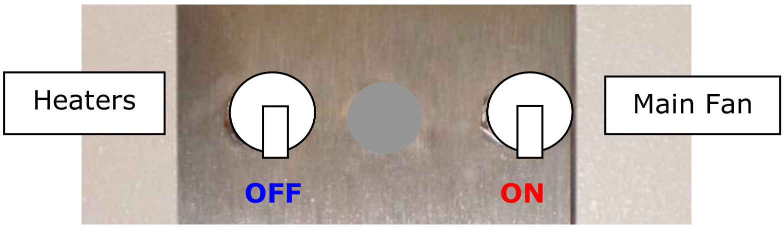
Figure 5: Temperature controller

To change the set point push the 'P' button once on the temperature control panel. The red display will change to a flashing 'SP 1' (Set Point 1). While 'SP 1' is flashing, the set point can be adjusted using the up and down arrows on the control panel. To confirm the new set point push 'P' once more. Alternatively, wait ten seconds after setting the new temperature and the controller will automatically save the new set point. If the temperature is changed, allow sufficient time for the temperature to equilibrate (green and red displays should both show the same value) before beginning sampling.

To being heating set the switches on the front panel as shown below for 'heating mode'. Once the experiment is finished the heaters can be turned off and the main fan activated to cool down the sample pots – 'cooling mode' below.



Heating mode



Cooling mode

Note: When sampling at >200°C, condition the Viton o-rings for at least 24 hours at 250°C.

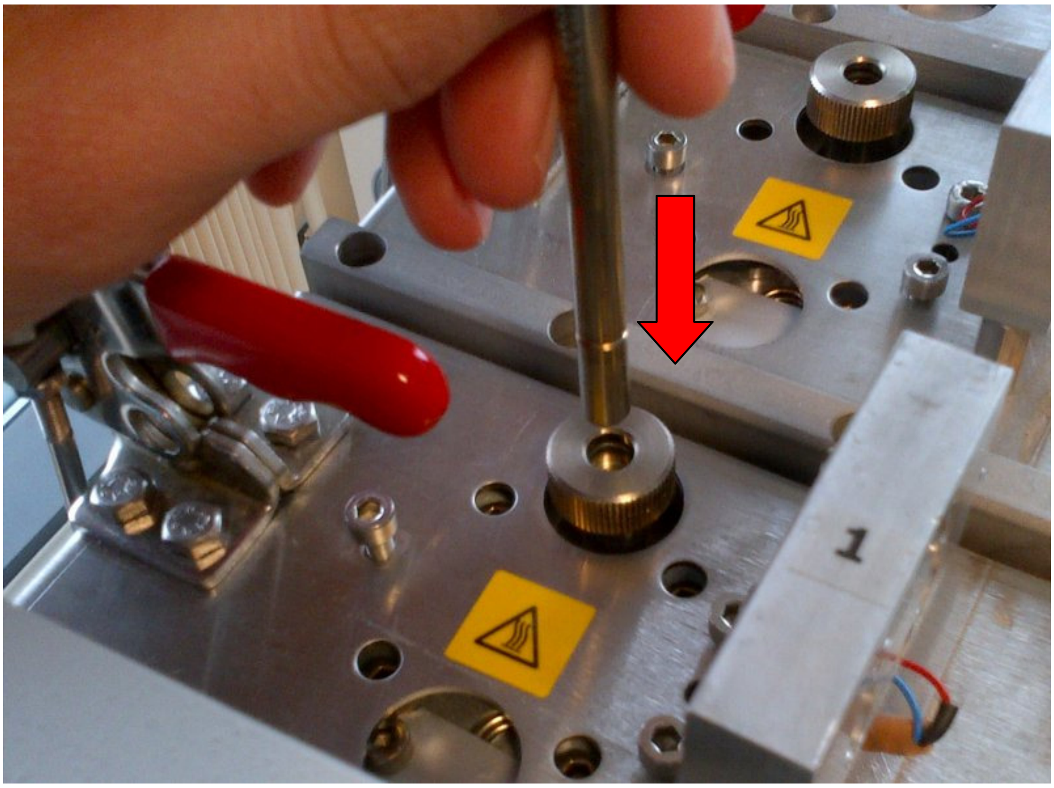
3.6. Removing microchamber pots

Use the supplied cup removal tool (p/n M-MCTL250) (figure 6) to remove microchamber pots, if they are hot.

Figure 6: Removing a hot pot with the removal tool

3.7. Fitting sampling tubes

Sorbent tubes should be inserted into the chamber sampling ports by gently pushing then into the o-ring seals, as shown below. There is no need to adjust the screw caps, they are only removed when the sealing o-ring needs replacement (see section 5.2).



After the sampling is completed the tube can be pulled out of the sealing o-ring.

4. Sampling methods

4.1. Bulk emission testing

After setting the required gas flow rate and temperature, materials for bulk emissions testing, (or odour/fragrance profiling), may simply be weighed and placed directly into an empty microchamber (figure 3) - or into a custom made inert 'sample boat' which is placed directly into the chamber itself.

Allow the sample to equilibrate - typically 5 - 10 minutes - and then insert a conditioned sample tube into the M-CTE250 tube insert to commence collecting emissions. At the end of the sampling interval, remove the tube and cap with brass storage caps as described above.

Sampled tubes are then thermally desorbed and analysed by GC(-MS/FID). Alternative analysers, for example systems combining thermal desorption with process MS or ENose detectors, are also applicable in some cases.

Note: If sampling at elevated temperatures, sample tubes may be hot when they are removed from the M-CTE250.

Depending on the type of sample material analysed, after sampling the chamber may require cleaning before subsequent use. See section 5.1 for cleaning techniques.

4.2. Surface emissions testing

Surface emissions testing requires a 6.4 cm diameter circular sample specimen to be cut from the product/material so that it fits snugly into the microchamber on top of the supplied spacers. A collar projecting down from the chamber lid helps define both the exposed sample surface area and the depth of the air space above the sample (figure 3). When testing surface emissions approximately 24 cm^2 of sample surface area is exposed to the air/gas flow, and the air/gas volume above the sample surface is 7.4 cm^3.

The M-CTE250 is supplied with a complete set of sample spacers which are used to raise the surface of the test sample so that it is flush with the top of the microchamber pot. The spacers are placed inside the microchamber, underneath the sample, such that the emitting surface is presented to the air flow at the correct height, whatever its original thickness.

After setting the required gas flow rate and temperature, place the sample inside the microchamber and allow the sample to equilibrate - typically for 5 - 10 minutes – and then insert a conditioned sample tube into the M-CTE250 tube insert to commence collecting emissions. At the end of the sampling interval, remove the tube and cap with brass storage caps as described above.

Sampled tubes are thermally desorbed and analysed by GC(-MS/FID). Alternative analysers, for example systems combining thermal desorption with process MS or ENose detectors, are also applicable in some cases.

Note: If sampling at elevated temperatures, sample tubes may be hot when they are removed from the M-CTE250.

Depending on the type of sample material analysed, the chamber may require cleaning after sampling, before subsequent use. See section 5.1 for cleaning techniques.

5. Maintenance

5.1. Cleaning

The M-CTE250 sample pots, tube inserts and lids are constructed from stainless steel to minimise contamination and carryover effects. However, cleaning of the chamber pots and lids may occasionally be required depending upon the type of sample analysed and the conditions in which they were sampled.

Note: The inert microchamber (M-CTE250i) has sample pots, tube inserts and lids constructed of inert coated stainless steel to minimise breakdown of labile compounds.

The physical nature of some samples *e.g.* viscous compounds, molten polymers etc. will be more prone to contaminating the chamber than more rigid samples. As a result, cleaning of the sample pots and lids in between sampling such products may be necessary. For more rigid samples, cleaning will be required less frequently/rigorously.

5.1.1. Chamber lid removal

- Remove the screw cap and o-rings from the sample “chimney”
- Unscrew the 4 captive retaining screws using the hexagon driver provided
- Lift the stainless steel lid away from the aluminium heating block
- Remove the small o-ring from the protruding 1/16” tube

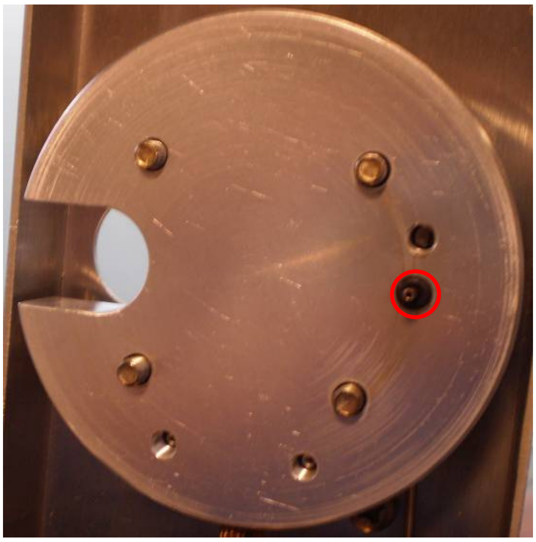


Figure 7: Position of 0-ring for removal

5.1.2. Cleaning methods

Having first removed all sealing O-rings, the microchamber pots, chamber lids and spacers can be cleaned by one of two methods:

1. The inner surface can be washed with detergent, followed by two separate rinsings with freshly distilled water. It is then rinsed again with isopropanol.

2. Inserting all components, with the exception of the chamber lids, into a vacuum oven. Individual microchamber pots can be removed from the M-CTE250 and placed directly into a vacuum oven at an elevated temperature (200-300°C) for approximately 2 hours. Before placing the sample pots into an oven, ensure that the O-rings have been removed from the rim of the microchamber pots, from the tube connector attached to the lids, and from the sample pot lid. See section 5.2 for further details on O-ring removal.

5.1.3. Lid reassembly

After cleaning, reassemble the lid ensuring that there is a fresh o-ring on the protruding tube, and that this tube engages with the conical hole in the lid. Tighten the 4 captive screws as shown below.

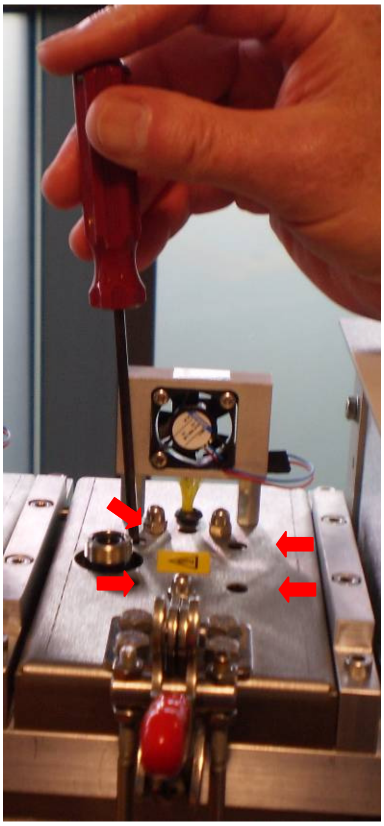


Figure 8: Position of 4 captive screws in microchamber lid for tightening

5.2. Changing O-ring seals

The microchamber pot seal O-rings can be found on the top rim of the pot. Two different types are available depending on your required operating temperature. Please refer to the CRITICAL INFORMATION on page 3 for advice on pot-seal O-ring selection and part numbers.

The tube insert O-ring, either size 010 for ¼" diameter tubes (p/n U-COV10 (pk 10)) or size 5.92 mm for 6 mm diameter tubes (p/n U-COV45 (pk 10)) can be found by unscrewing the retaining cap.

If the O-rings require removal they should be hooked out with the O-ring extraction tool (p/n SERZ-0351) available from Markes International.

To prevent unnecessary contamination, new seals should be pushed into position using the O-ring insertion tool (p/n SERZ-0285) available from Markes International, and gently nudged evenly into the seating. Also use the O-ring insertion tool to smooth around the inner diameter of the O-ring as it is being pushed into place to avoid distortion.