

QUI-0003 Version 6.3 – September 2013

User manual

TC-20 – Sample conditioning and dry-purge rig



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1. Introduction to the TC-20

The TC-20[™] may be used as a sorbent tube conditioning rig (to clean sorbent tubes prior to sampling) or as a dry-purge rig (to remove water from sampled tubes prior to analysis).

1.1 Tube conditioning

It is essential that tubes are carefully conditioned before they are used for sample collection. Conditioning parameters should be more stringent than those to be used for subsequent analysis. Typically, tubes are conditioned at 10°C below the maximum safe temperature of the least stable sorbent in the tube. Extra care should be taken when conditioning carbonised molecular sieve-type sorbents to ensure that all oxygen is eliminated from the tube before sorbent temperatures are allowed to exceed 250°C.

More details of recommended tube conditioning parameters are given in Application Notes TDTS 5 and TDTS 19 (available at <u>www.markes.com</u>).

After conditioning, a representative number of the cleaned tubes (usually 1 in 10) should be desorbed under analytical conditions to check artefact levels. Individual artefacts (peaks on the blank chromatogram) should be no more than 1 ng in toluene equivalents for Tenax® and below 10 ng toluene equivalents for other porous polymer sorbents. Artefact levels can be reduced to below 0.1 ng after stringent conditioning for most carbon sorbents.

NOTE: It is advisable to keep a record of blank chromatograms for individual tubes or series of tubes, so that trends in artefact levels can be monitored.

1.2 Dry-purging

Dry-purging is used to remove excess water from sorbent tubes, particularly when hydrophilic sorbents such as Carboxen[™] 1000 and Carbosieve[™] SIII have been used.

After sampling, clean, dry carrier gas is passed through the tube in the sampling direction (*i.e.* with gas arriving at the grooved end of the tube first).

For example, if mixed-bed universal tubes (which contain Tenax[®], a graphitised carbon black and a carbonised molecular sieve), are purged in this direction at a flow rate of approximately 50 mL/min for 10–20 min, the level of water found on the tube is significantly reduced. This helps to avoid potential difficulties with GC detectors such as mass selective detectors, FIDs and ECDs, where a large amount of water can adversely affect the detector. In addition to this, it extends the column lifetime.

2. Required services

2.1 Power supply

TC-20 requires either 240 V or a 120 V power supply. The voltage requirements for your unit are indicated on the serial number plate at the back of the instrument (Figure 2).

2.2 Supply of clean carrier gas

Markes recommends the use of clean dry nitrogen or helium.

It is strongly recommended that a large-capacity hydrocarbon filter is installed in the carrier gas line as close to the TC-20 as possible, to remove any trace level hydrocarbons from the carrier gas.



Figure 1: Front view of TC-20.



Figure 2: Rear view of TC-20.

3. Prior to operation

Prior to using the TC-20 for the first time, we recommend that the unit is operated overnight (for 6–8 hours), at a temperature of 350°C with carrier gas flowing through the unit. If possible, this should be carried out using 20 empty tubes *in situ*. This will ensure that the TC-20, associated connections and gas lines are scrupulously clean, following shipping and installation.

3.1 Attachment of cooling plate

The cooling plate is packed separately to the TC-20 and supplied with two screws for attachment to the top plate of the TC-20. Use the screws supplied and a posi-drive screwdriver to attach the plate, as shown in Figure 1.

3.2 Important notes relating to operation

Please read these notes **<u>BEFORE</u>** operating the TC-20.

- Ensure that the top manifold is always in the TC-20 (except when loading tubes) to keep the temperatures as equalised as possible.
- Do not block or cover the rear of the unit and allow at least 200 mm of clear space to the rear of the instrument. Note that the TC-20 emits approximately 250 W of heat when set at 400°C. Only operate the TC-20 in a well ventilated area.
- On reaching the end of the run time, keep the TC-20 and carrier gas flow switched on and place the manifold in the cooling plate. To avoid contamination of the clean tubes, remove them from the TC-20 as soon as they are cool enough to be handled. Reduction of the carrier gas pressure to 5 psi during the cooling process will also reduce the risk of contamination. (Note that it is important to keep the fan running while the TC-20 is hot).
- Operate with care at all times remember some parts of the TC-20 reach high temperatures. Observe normal good laboratory practice at all times.

4. Operating instructions

- 1. Hold manifold assembly or place in a suitable position whilst inserting tubes.
- 2. For tube conditioning, push the non-grooved end of the sorbent tubes to be conditioned into the manifold block, with the sampling (grooved) ends pointing outwards. Any number of the 20 tube positions may be left empty if required.

For dry-purging, follow the instructions as for conditioning, but insert the tubes into the manifold assembly grooved end first, *i.e.* with the non-grooved end pointing outwards.

- 3. Using the handle, guide the assembly down into the heater block. The tubes should slot easily into the heater block. If there is any resistance, do not force the block down.
- 4. Set the required temperature for the conditioning programme (see Section 4.2).
- 5. Set the required time for the conditioning/dry-purging programme (see Section 4.3).

NOTE: At the start of the run the timer will commence and the heaters will switch on. Remember to extend the tube conditioning time to take account of the TC-20 warm-up time if necessary.

- 6. Switch on the carrier gas to the unit and adjust to the relevant pressure (see Section 6 for further information).
- 7. Press the $\langle U \rangle$ button on the timer (see Section 4.3).
- 8. To stop the TC-20 at any time during its programme, press the <U> button on the timer. Remember that it will take some time for the TC-20 to cool down to ambient temperature.
- 9. When the programme is complete or is aborted, leave the tubes in the heater block with the carrier gas flowing and the TC-20 switched on. Remove the manifold assembly and place it on the cooling plate. Remove the tubes from the manifold block as soon as they are cool enough to handle. Reduction of the carrier gas flow during the cooling process to 5 psi will reduce the risk of contamination. Do not switch off the carrier gas flow until the tubes have cooled sufficiently to comfortably handle them.

NOTE: To extend sorbent lifetime and avoid artefact formation, it is advisable to wait until the main heater block inside the TC-20 is reading below 100°C before loading a fresh batch of tubes.

10. Pull steadily to remove the tubes from the manifold assembly. If necessary, rotation of the tubes will aid removal.

4.1 Gas supply quick-release union

The manifold assembly and tubing is attached to the TC-20 *via* a quick-release union (Figure 3). The union can be disconnected, leaving the gas supply to the TC-20 sealed, so that the manifold assembly can be removed if required for ease of inserting/removing tubes.



Figure 3: Quick-release union.

To attach the manifold assembly and tubing to the TC-20:

- 1. Align the stem with the body.
- 2. Pull the body sleeve down and firmly insert the stem body until it bottoms-out.
- 3. Release the sleeve.
- 4. The sleeve should return to its original position.

To detach the manifold assembly and tubing from the TC-20:

- 5. Pull the body sleeve down.
- 6. Remove the stem from the body.

4.2 Temperature control

The TC-20 can operate between 50°C to 400°C using the temperature controller (Figure 4).



Figure 4: Temperature controller.

To select an operating temperature between 50°C and 400°C:

1. Press <P>. 'SP 1' should be displayed flashing on the top line of the display.

CAUTION: If the <P> key is pressed for longer than 2 seconds, the display will show 'PASS'. If this happens, press <P> again before repeating step 1. **Do not** attempt to make any changes if 'PASS' is displayed.

- 2. Use the Up or Down arrow keys to set the required temperature.
- 3. When the required temperature set-point is displayed, press <P> to confirm the new set-point. The display should revert to the normal operating mode within 10 seconds.

4.3 Digital timer control

The TC-20 run time can be set from 1 minute to 99 hours 59 minutes using the timer controller (Figure 5).



Figure 5: Timer controller.

To adjust the setting between 1 minute and 99 hours 59 minutes:

- 1. Press <P>. 't1' should appear on the display whilst the key is pressed, and the SET/CNT indicator at the top left of the timer will flash.
- 2. Use the Up or Down arrows to set the required time.
- 3. Press and release 'U' for display to revert to the normal running mode. The controller will revert to this display if no buttons are pressed for 10 seconds.

5. Service and general maintenance

5.1 Trip reset procedure

If the heat generated by the TC-20 does not dissipate sufficiently quickly, and as a result the internal temperature exceeds 150°C for any period of time, the trip switch is activated. To avoid injury, allow the unit to cool before resetting as follows.

- 1. Switch off unit and remove the mains power lead. (It is not necessary to remove the gas connection if the rear of the unit can be freely accessed).
- 2. Remove the four larger (M4) screws securing the fan assembly (Figure 2).
- 3. Withdraw the fan assembly. It is not necessary to remove the fan supply lead.
- 4. The red reset button is now accessible inside the unit. Push to reset.
- 5. Place the fan supply leads in the unit and replace the fan assembly, being careful not to trap the fan supply leads.
- 6. Replace the four screws removed in Step 2.
- 7. Replace the mains power lead and the gas connection (if removed).

5.2 Charcoal filter replacement procedure

Charcoal filters are positioned in front of the fan to trap the VOCs removed from the sorbent tubes. This ensures that the VOCs do not contaminate the laboratory air (see Figure 6).

Replacement filters are available in packs of four (part number R-FLTR4)

- 1. Switch off unit and remove the mains power lead. (It is not necessary to remove the gas connection if the rear of the unit can be freely accessed).
- 2. Remove the four larger (M4) screws securing the fan assembly (Figure 2).
- 3. Withdraw the fan assembly. It is not necessary to remove the fan supply lead.
- 4. Unclip the spring holding the filters in the assembly and replace and remove old filters.
- 5. Place two new filters in the assembly and replace the spring, ensuring that it is clipped securely.
- 6. Place the fan supply leads in the unit and replace the fan assembly, being careful not to trap the fan supply leads.
- 7. Replace the four screws removed in step 2 above.
- 8. Replace the mains power lead and the gas connection (if removed).





6. Recommendations for successful conditioning

• The carrier gas supply should be either high-purity helium or high-purity nitrogen. The recommended gas purity is 5N. Depending on the quality of the gas supply, it may be necessary to use in-line filters to remove trace levels of oxygen, moisture and hydrocarbons. Markes International recommend the use of the following filters:

0	Disposable indicating oxygen trap	Part number:	C-O2TRP
0	Refillable hydrocarbon trap	Part number:	C-HCTRP
0	Refillable glass moisture trap	Part number:	C-MSTRP

- Always ensure that there is carrier gas supply to the unit prior to starting the conditioning programme.
- Always ensure that there is sufficient carrier gas supply for the conditioning time chosen.

NOTE: The TC-20 will consume 900 mL/min of carrier gas (45 mL/tube connection point) at 15 psi (see Table 1).

• At the end of the conditioning programme, the unit will start to cool down. Ensure that the flow of carrier gas to the tubes is maintained until they have cooled back down fully to ambient temperature.

Pressure (psi)	Flow rate (mL/min)
10	30
15	45
20	60
25	75
30	90

Table 1: Flow rate versus pressure (approximate).

6.1 Recommended conditioning programmes

Freshly packed Tenax®: 2 hours at 320°C with 50–100 mL/min carrier gas flow, followed by 30 min at 335°C with 50–100 mL/min carrier gas flow.

Freshly packed Chromosorb[®] **106:** 2 hours at 220°C with 50–100 mL/min carrier gas flow, followed by 30 min at 230°C with 50–100 mL/min carrier gas flow.

Freshly packed SulfiCarb™: 1 hour at 100°C, 1 hour at 200°C, 1 hour at 300 °C and 30 min at 380°C with 50–100 mL/min carrier gas flow.

Previously used tubes: For tubes which are not freshly packed, *i.e.* those that have been used previously but need cleaning for future sampling, the final time and temperature given above will be sufficient. For example, used Tenax[®] tubes may be reconditioned at 335 °C for 30 min.

NOTE: For tubes which are to be used for trace-level analysis, additional conditioning may be required.

Further information on tube conditioning is given in Markes' Application Note TDTS 5.

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7. Recommended dry-purge programmes

- 1. Set the carrier flow to approximately 50 mL/min.
- 2. Either leave the temperature switched off or set it to a low temperature, *i.e.* just above ambient temperature (35–40°C).
- 3. Set the time run for about 10–20 min.

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