

Packing Thermal Desorption Sample Tubes



Introduction

For a thermal desorption analysis to be successful, the adsorbent inside each tube must be properly packed. Any voids caused by settlement or contraction of packing may induce channeling of the gas flow during pumped sampling and thermal desorption. Such effects may also disturb the geometry of the “air gap” which is critical for predictable performance in diffusive sampling.

Several types of sample tubes are available for PerkinElmer® thermal desorption systems:

- Stainless steel, empty, without caps – pack of 100 (Part No. L4270129)

- Stainless steel, empty, without caps – pack of 10 (Part No. L4270128)
- Stainless steel, empty, with caps – pack of 50 (Part No. M0413596)
- Stainless steel, empty, with caps – pack of 10 (Part No. M0413595)
- Glass, empty, without caps – pack of 10 (Part No. L4071594)
- Glass, empty, with caps – pack of 10 (Part No. M0413598)
- Glass-lined stainless steel, empty, with caps – pack of 10 (Part No. M0413597)

All empty tubes with caps are shipped with Analytical End Caps (Part No. N6200119, pack of 20).

In the stainless steel tubes, the adsorbent is normally held in place using stainless steel gauzes (Part No. L4071034, pack of 100) with a gauze-retaining spring (Part No. L4071123, pack of 50) in the non-grooved end (see Figure 1 – Page 2). These tubes can be used for both diffusive and active (pumped) sampling.

With glass and glass-lined stainless steel tubes, the adsorbent material is held in place using glass-wool plugs (see Figure 2 – Page 2). Such tubes can only be used for active (pumped) sampling. Packing procedures for all types of sample tubes are very simple and guidance notes to help ensure success are presented in this technical note.

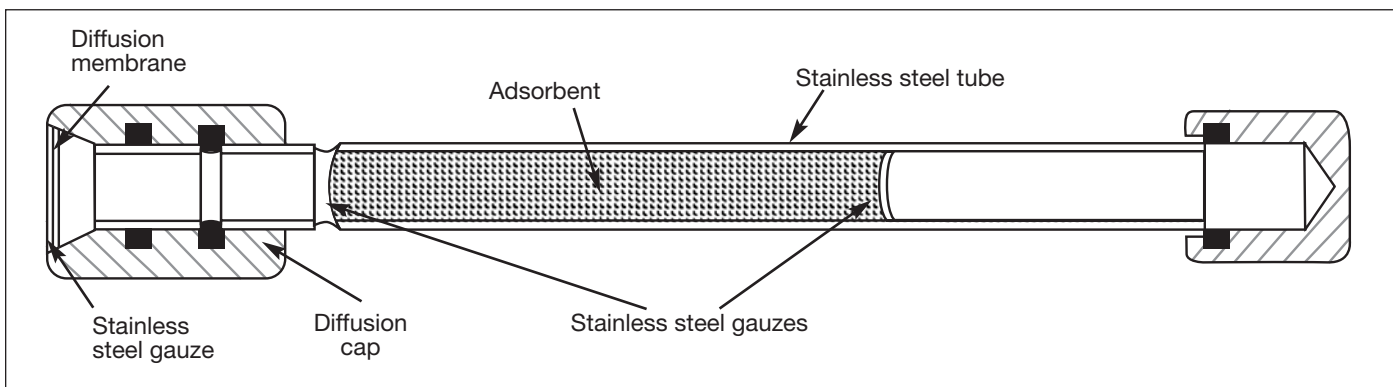


Figure 1. Packed stainless steel tube shown with a fitted diffusive cap.

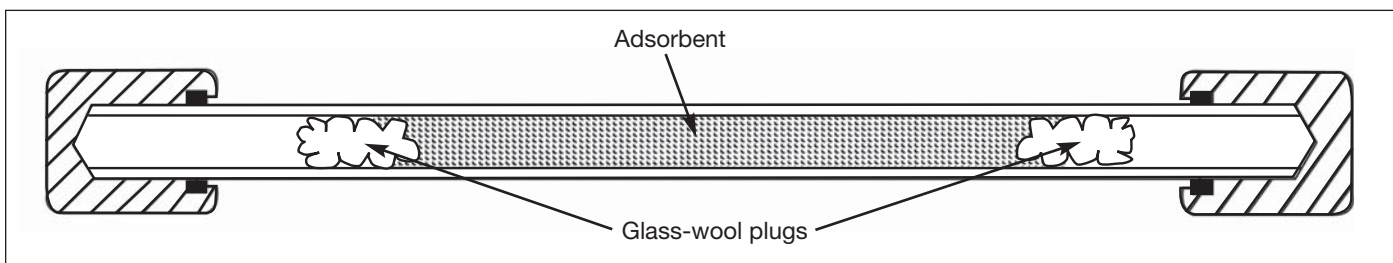


Figure 2. Packed glass or glass-lined stainless steel tubes.

General practices applicable to all tube types

1. An air gap of at least 1.5 cm must be left unfilled at each end of the tube as the desorption oven covers the central 6-cm portion of the 9-cm tube.
2. The adsorbent used must be 60-80 mesh or more coarse. Adsorbent materials with much finer particle size such as 80-100 mesh are NOT suitable. This is because the particles may pass through the retaining gauze. Such packings also produce too high a back pressure for conventional, personal monitoring pumps.
3. It is not necessary to compress the adsorbent material within the tubes. Once the packing has been tipped into the sample tube, it should be gently tapped to ensure that the particles have “settled” before pushing the second gauze or glass-wool plug into the tube.
4. For diffusive sampling, it is not usually necessary to weigh the adsorbent material into each sample tube. This is because analytes are only normally retained on the first few millimeters of packing. However, for pumped sampling, where the pressure drop across each tube should be identical, it is important to weigh the same amount of material into all the tubes. In these cases, the adsorbent material should also ideally be sieved to remove any fines (under-sized particles). This helps to guarantee that tubes containing the same mass of packing will exhibit identical back pressure and thus produce reproducible sampling flow rates with a given sampling pump.

Packing stainless steel tubes

As stainless steel tubes are compatible with the stainless steel gauzes (Part No. L4071034, pack of 100) the gauze loading rig (Part No. L4070023) can be used to help pack these tubes.

New stainless steel, empty tubes with caps, (Part No. M0413595) arrive with the initial gauze in position inside the tube. The exact position of this gauze is critical and is determined by a groove on the inside of the tube. The position of this inner groove is marked by a second groove on the outside of the tube (see Figure 1). This gauze is critical for diffusive monitoring as it establishes the 1.5-cm diffusion path. It also prevents the sorbent material from migrating into the cooler, remaining 1.5-cm portion of the tube when heated.

If this initial gauze becomes dislodged during use or when repacking older tubes, a new gauze must be inserted using the gauze loading rig as follows:

The adjustable stop should be set so that the underside of the knob is 3 inches above the barrel of the tool when the stop is in contact with the barrel. Fix the position of the “stop” using the appropriate Allen wrench.

- Place a clean, empty tube grooved end down on the gauze loading rig as shown in Figure 3.
- Place a gauze, convex side down, into the hole in the rig.
- Push the plunger gently down until the “stop” reaches the rig. At this point, the ball bearing underneath the tube should flex slightly. The gauze is now correctly positioned.

The gauze loading rig can also be used to insert the second gauze into a packed tube, if required:

- First tip the required amount of packing into the tube.

NOTE:

1. If the adsorbent is not to be weighed, it is normal to pour the material into the tube little by little and adjust the height by tapping gently to settle the packing until a gap of ~1.5 cm is left at the end of the tube.
 2. If an expensive or dense packing material such as spherocarb is used, a suitable sorbent bed length is typically considered to be around 2 cm, not 6 cm.
- Tap the tube gently to settle the packing material.
 - Insert the second gauze using the gauze loading rig but do not exert too much pressure or the adsorbent

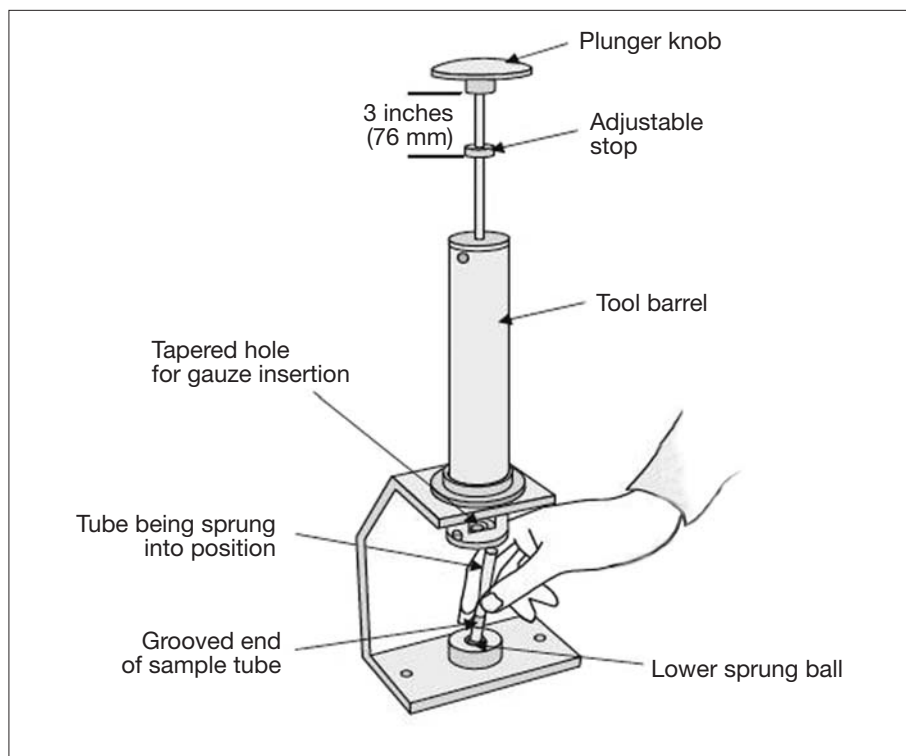


Figure 3. Use of the gauze loading rig.

bed will be compressed causing the tube back pressure to increase.

- Insert the gauze retaining spring and push and twist it down until it touches and supports the second gauze.

If the tube is to be packed by more than one adsorbent in series (pumped monitoring only), the different adsorbents must be kept in distinct, separate layers using stainless steel gauzes or glass-wool plugs (see Figure 4 – Page 4).

Packing glass or glass-lined tubes

As stainless steel gauzes cannot be used in glass or glass-lined tubes, the gauze loading rig is not appropriate to the tube packing procedure. The adsorbent material is simply held in place using glass-wool plugs which must not extend into the 1.5-cm air gaps at each end of the tube. If more

than one adsorbent is required in series, the different layers are again separated with glass wool.

Tube conditioning and storage

Once packed, all tubes must be conditioned using more stringent desorption conditions than those required for subsequent analysis, i.e. using faster gas flows and higher temperatures where possible.

NOTE: Care must be taken not to exceed the maximum temperature of the adsorbent.

Once packed and conditioned, the tubes should be capped and stored in as clean an atmosphere as possible. The standard Teflon® storage caps (Part No. N6200119, pack of 20) supplied with the tubes will prevent ingress of contaminants and a loss of trapped components from sampled tubes for approximately 1 week. For

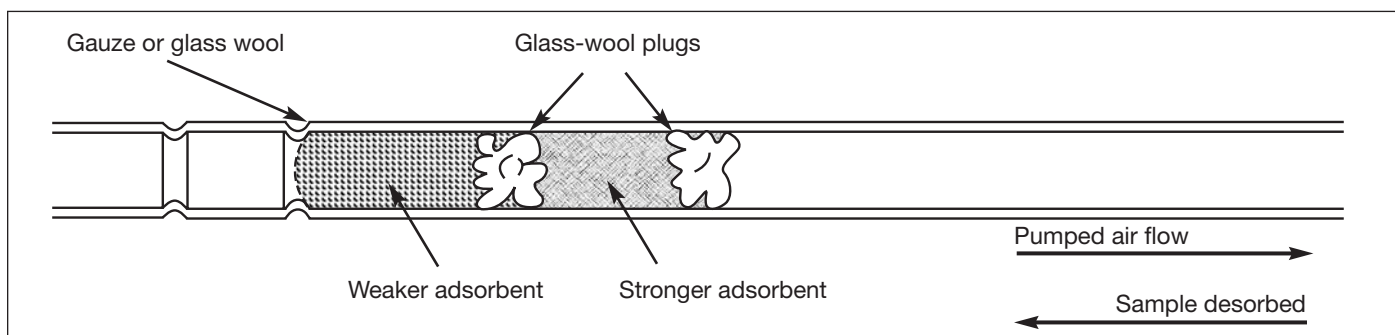


Figure 4. Packing a single sample tube with sorbents of increasing strength – Chromosorb 106 and charcoal are examples (applicable to all tube types).

longer term storage of conditioned or sampled tubes, the 1/4-inch brass, long-term fittings (Part No. 09908851, pack of 2, supplied with the tube) and one piece PTFE ferrules (Part No. L1003015, pack of 10) are recommended.

Such caps have been evaluated for storage periods greater than one year and are used to cap the PerkinElmer tubes supplied, as certified by the Bureau de Communité de Reference (BCR) of the European Community.

Tube impedance testing on the TurboMatrix 650 ATD

The TurboMatrix™ 650 ATD Thermal Desorber provides an automated method of characterizing

thermal desorption tube impedance to check packing integrity. Tube impedances may be tested against specified limits or against their initial values and existing manual QC procedures may be made more efficient. This technology should aid users in maintaining their tubes in optimum condition and improve confidence in the analytical data produced.

Further details of tube impedance testing using the TurboMatrix 650 ATD are available in the Technical Note entitled: “Tube and Trap Impedance Testing on the TurboMatrix 650 ATD Thermal Desorber” available on the PerkinElmer website at www.perkinelmer.com/GCLibrary.

Conclusions

The methods described in this document for packing thermal desorption stainless steel tubes as well as glass and glass-lined tubes will ensure that the appropriate techniques are employed to provide the most reliable packing for both single and mixed packing materials.

An extensive range of packing materials is available from our partner Supelco® (www.sigmaaldrich.com/supelco).

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