CAMSCO
Sample Quality

This document intends to answer some frequently asked questions regarding sorbent tube conditioning:

1. At what temperature should tubes be conditioned?
2. At what flow rate should tubes be conditioned?
3. How long should tubes be conditioned? Is 2-hour conditioning enough for every tube?
4. Why are used tubes cleaner/better than new ones?
5. What is the Memory Effect and how to overcome it?
6. Why background levels differ from manufacturer to manufacturer? And from batch to batch?
7. What really happens during conditioning? How clean could it get?

1. At what temperature should tubes be conditioned?

- CAMSCO's Sorbent Selection Chart lists the conditioning temperature, desorbing temperature, and maximum temperature for each sorbent; the chart is the most complete in the industry, and downloadable here.
- As a general rule, the conditioning temperature is either 20°C higher than the desorbing temperature or 10°C lower than the maximum temperature. If your desorbing temperature is much lower than the maximum temperature, we recommend you at least condition the tube once at the listed conditioning temperature.
- For tubes with more than one sorbent bed, the conditioning temperature is that of the least stable sorbent. This also means it's desirable to build multi-bed tubes with sorbents that have similar conditioning temperatures.
- Instruments, such as thermal desorbers and tube conditioners, report the temperature of the heating block, not the temperature inside a tube. Keep in mind that while the sorbent is heated up by the instrument, it's also cooled down by the carrier gas flow (the Joule–Thomson effect dictates that gases are cooler released than compressed). In reality we have measured a gap as large as 20~30°C on some instruments, the magnitude of the gap depends on instrument design as well as carrier gas flow rate (see Question 2) and the temperature of the gas. Thus, you may carefully increase the conditioning temperature for better results, but try not to exceed the maximum temperature.

2. At what flow rate should tubes be conditioned?

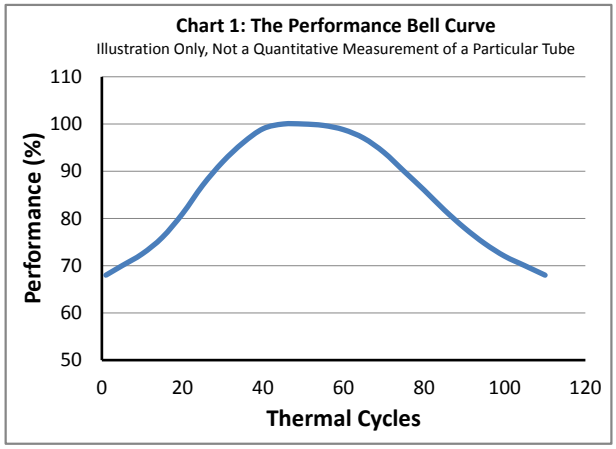
- The conditioning flow rate should be higher than the desorbing flow rate.
- For example, a typical set of numbers for a standard PerkinElmer steel tube could be: sampling flow rate of 60 ml/min, desorbing flow rate of 30~50 ml/min, and conditioning flow rate of 100 ml/min.
- Higher flow rate is not always desirable because: (1) it cools the sorbent too much, creating a larger temperature gap (see Question 1), (2) it may compress/break friable sorbents, (3) it may create small gaps in the sorbent bed.

3. How long should tubes be conditioned? Is 2-hour conditioning enough for every tube?

- Depending on how stringent you need to condition the tube, the time can vary from minutes to hours.
- We recommend 2 hours for general purposes, which means ppm level of airborne concentration or micro gram level of adsorbed sample. We recommend 4 hours for strong sorbents or multi-bed tubes.
- Longer time is needed to detect ppb level airborne concentration or nano gram level of adsorbed sample.
- Longer time is needed if the tube manufacturer does not pre-condition the sorbents, see Question 6.
- New tubes may require longer conditioning than used tubes: see Question 4.
- Sorbents that are prone to memory effect may need repeated conditioning, see Question 5.

4. Why are used tubes cleaner/better than new ones?

- Many users have found used tubes perform better than new ones.
- In Chart 1, "Tube Performance" on the Y-axis is a composite score of background cleanliness, adsorption/desorption efficiency and breakthrough volume; while a "Thermal Cycle" could be either a desorption or a conditioning process.
- Tubes that are very new or very old normally have higher background, lower efficiency, and smaller breakthrough volume; in short, not as good.
- The reason for the bell-curve is explained in Question 7.



5. What is the Memory Effect and how to overcome it?

- Tubes with Memory Effect can be thoroughly conditioned, tested clean, and tightly sealed. But after a few days in storage, some small peaks appear as increased background.
- Sorbents that have larger surface area (i.e., $>500 m^2/g$), smaller pore size (i.e., micro-pore) and closed pores are more susceptible to Memory Effect due to their enhanced Van Der Waals Force, capillary effect, and dimensional limits on diffusion. Using such sorbents to monitor trace amount of VOC may require repeated conditioning with a few days in between until the Memory Effect is gone.
- What happens is one conditioning process only gets rid of the adsorbed VOCs near the surface, and you get a chromatogram that's seemingly clean. It takes days for deeply buried VOCs to migrate to the surface by diffusion. For example, most Carboxen sorbents may emit sulfur dioxide which needs repeated conditioning to diminish.

6. Why background levels differ from manufacturer to manufacturer, and from batch to batch?

- Many people think tubes should have very similar if not identical backgrounds because different manufacturers use the same Tenax TA. In reality that's not the case.
- First of all, some manufacturers, like CAMSCO, pre-condition all their sorbents before filling the tubes, and some don't. At CAMSCO, we found this pre-conditioning process critical to keep your/our instruments healthy, and you will be surprised how much can be cooked out of 100g raw sorbent – it could coat a big ball of glass wool with dark brown stuff, or it could make the whole building smelly if not ventilated.
- Second, manufacturers can cook the sorbent more or less in the pre-conditioning process. According to the bell-curve in Question 4, somebody could over-cook the sorbent before assembly, making their sorbent tubes cleaner/better than anybody else's; but the trade-off is the client only get half of the lifespan of a regular tube. At CAMSCO, we only get rid of the mass contaminants to protect your instrument.
- Third, some manufacturers have no front glass wool plug. The risk is sorbent particles sitting directly on a metal mesh screen could break and leak through, damaging the instrument. At CAMSCO we consistently put a front glass wool plug in place to prevent sorbent leakage unless the tube is designed for passive sampling. Glass wool itself may contain organics leftover from the manufacturing process and lead to a higher background.
- Finally, sorbent quality changes from batch to batch all the time. CAMSCO is the world's largest consumer of Tenax TA and HayeSep, we buy large quantities of sorbents and homogenize them to yield higher consistency over time.

7. What really happens during conditioning? How clean could it get?
    - Most people think the conditioning process simply sweep the adsorbed VOCs from the sorbent surface and make it available for next sampling. That's largely true but things can get more complicated and it's good to know more.
    - There are three basic categories of sorbents: Porous Polymers (PP), Graphitized Carbon Black (GCB) and Carbon Molecular Sieves (CMS). In general, PP have the highest background, after conditioning, a representative number of the cleaned tubes (i.e., 1 out of 10) should be desorbed under analytical conditions to check artifact levels. Individual artifacts (peaks on the blank chromatogram) should be no more than 1 ng in toluene equivalents for Tenax. For other porous polymer sorbents artifact levels may be as high as 25~50 ng and as such these sorbents are not recommended for trace level monitoring. Artifact levels can be reduced to below 0.1 ng after stringent conditioning for most carbon sorbents.
    - Different chemistry happens during the conditioning process. For PP, it's mostly oxidation, take Tenax TA for example, oxidized by-products can be detected during conditioning process and the color of the sorbent darkens as the tube ages, but this oxidation process may provide more surface area available for adsorption. For other sorbents like CMS/GCB, conditioning may open up closed/clogged pore and make the interior accessible to analytes, generating the bell-curve in Question 4.
    - Oxidation may not affect Tenax TA as badly as it does to some CMS sorbents, so it is a good idea to install an oxygen trap before your desorber when you try to condition CMS, such as Carbosieve S-III.

It is essential that tubes are carefully conditioned before they are used for sample collection. If you have further questions please do not hesitate to contact CAMSCO for technical support.

6732 Mayard Road
Houston, Texas 77041
Phone +1 (713) 983-0800
Fax +1 (713) 983-7200
solutions@camsco.com
www.camsco.com