How can I make my GC column last longer

Once a column leaves the factory, there is no way of knowing for what sample type or experimental conditions it will be used – which will contribute to column stationary phase degradation and thus your column's life span. Therefore, if you want to maximize column life, it is important to understand what causes column stationary phase performance to degrade and what you can do to minimize that degradation.

The most common causes of GC column stationary phase degradation are:

- Column contamination from non-volatile and semi-volatile sample residues
- Oxygen damage at elevated temperatures
- Thermal damage by exceeding maximum temperature limits
- Chemical damage from inorganic bases, inorganic acids, and salts

While the demise of practically all capillary columns can be attributed to these four causes, the primary cause of system failure is contamination from injected sample.* This article will concentrate on performance tips that help minimize that damage. Tips for minimizing oxygen, thermal and chemical damage will be discussed in an article next month.

Column contamination – the primary cause of column failure

As stated above, contamination from the injected sample is by far the primary cause of column failure. Common symptoms of this contamination are higher bleed, peak tailing and reduced response to active analytes.

Non-volatile contaminants do not elute and consequently accumulate in the column. The column becomes coated with residues that interfere with proper partitioning of solutes both in and out of the stationary phase. These residues may also interact with active solutes and cause peak tailing or loss of peak size.

Accumulated semi-volatile contaminates eventually elute, but not until hours or days have passed. In addition to causing peak shape and size problems, semi-volatile residues are usually responsible for baseline problems, such as instability, drift and ghost peaks.

Three performance tips to minimize sample contamination

Use these performance tips to maximize the life of your column.

1. **Check for residues.** Use this quick and easy test to determine if your sample contains potentially damaging residues. Deposit about 20 µl of the sample onto a microscope slide. Set the slide over a heated injection port or on a hotplate until dry. If you can see any residue where the sample was deposited, you have a likely cause for chromatography problems and potential damage to your column's stationary phase.

2. **Use a guard column.** Guard columns protect your analytical column from contamination. Sample residues deposit in the guard column without coating the stationary phase of the analytical column. Using this simple preventative measure is cost-effective, as guard columns are often 5-10 meters in length to allow substantial trimming before the entire guard column has to be replaced. Depending on the application, guard columns last from one week up to six months; then baseline and peak shape problems are possible indicators for replacement.

3. **Limit bake-out times.** High temperatures over long periods may convert some residues into insoluble materials that shorten useful column life. Limit bake-outs to 1-2 hours at the isothermal temperature limit of the column.

Any column can fail from a single injection of a particular dirty sample, or it can last for years. The running record for a DB-1 column is **20 years** – performing petroleum (naphtha) analysis where samples were extremely clean and run temperatures were low. So there's clear evidence that column lifetime can be maximized when you take the necessary precautions.

Three additional causes:

how you can minimize the danger of column degradation from these three additional causes:

1. Oxygen damage at elevated temperatures
2. Thermal damage by exceeding maximum temperature limits
3. Chemical damage from inorganic bases, inorganic acids and salts

Oxygen damage begins at higher temperatures

A leak in the carrier gas flow path (e.g. gas lines, fittings, and injector) is the most common source of oxygen exposure. While the leaks don't cause damage at or near ambient temperatures, severe stationary phase degradation appears at elevated temperatures with oxygen concentration as low as 10 ppm. In less severe cases, the column may function at a reduced level, but more severe cases can irreversibly damage the column. Symptoms include: premature onset of excessive column bleed; peak tailing for active compounds; and loss of resolution.

The best way to extend column life is to maintain an oxygen- and leak-free environment by taking these steps:

- Use ultra high purity carrier gases
- Install a moisture and oxygen trap in carrier gas lines
- Perform periodic leak checks of gas lines and regulators
- Change septa regularly
- Change gas cylinders before they are empty

Exceeding maximum temperature limit causes thermal damage

Exceeding a column's upper temperature limit for a prolonged period of time or heating a column without carrier gas flow even slightly above the ambient temperature can result in degradation of the stationary phase and tubing surface damage. Worse, as polarity increases, the temperature at which this damage occurs gets lower. Thermal damage is greatly accelerated in the presence of oxygen. Overheating a column with a leak or high oxygen levels in the carrier gas can lead to rapid and permanent column damage. Symptoms of thermal damage are similar to those of oxidation and include poor peak shapes for active compounds, loss of retention, and elevated background signal.

When subjecting a column to elevated temperature (e.g. upper temperature limit) for a prolonged period of time, more stress is put onto the column. The column lifetime can be shortened compared to standard operation, although the mode of failure might be due to fatigue of fused silica tubing, rather than stationary phase degradation.

Tips to improve performance:

- You can maximize column life by setting the maximum GC oven temperature at or a few degrees below the manufacturer's specified temperature limit.
- If a column is already damaged, condition it for 8 to 16 hours at its isothermal temperature limit. Remove 10-15 cm from the detector end and reinstall it. Then you can continue to use the column, although lifetime and performance may suffer.
- After column installation, make sure carrier gas flows through the column before it is heated by injecting a small amount of an unretained compound. A well formed peak with a retention time of 0.5-2 minutes should be obtained.

Inorganic bases, inorganic acids, and salts cause chemical damage

Inorganic bases such as KOH, NaOH, and NH_4OH, and inorganic acids such as HCl, H_2SO_4, H_3PO_4, and HNO_3 are particularly damaging to stationary phases, causing peak tailing or broadening, adsorption of active compounds, or rising baseline.

Tips to improve performance:

- Use a guard column to minimize column damage, but keep in mind that frequent trimming may be required.

- Chemical damage is usually limited to the front of a column, so trim or cut 0.5 to 1 meter – or 5 or more meters in severe cases – from the column front to eliminate most chromatographic problems.

Column life is subjective

Lifetime projections are based on method performance criteria and each analyst's performance expectations, but how long you can actually continue to use a column is a subjective determination. A column that may last five years performing one method can be unusable for more demanding analyses after just one injection of a dirty sample or after one run at a high temperature with a leaky septum.

In regular use, much of what we inject will contain some amount of residue even for pure solvents or standards. It's also likely to find a small amount of oxygen native to the carrier gas and part-per-billion leaks in fittings, regulators, injector bodies, and septa. That means that once a column leaves the factory, there is no way of knowing for certain what the actual life span of each column will be – making the care you take with your sample, and instrument the most important factor in determining usable life span of a column.

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