the Restek Advantage
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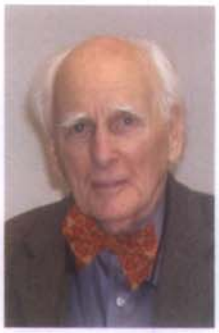
Erratum
Exempted drugs of abuse reference materials alprazolam (cat.# 34042), chlordiazepoxide HCl (cat.# 34044) and levorphanol (cat.# 34003) are at a concentration of 1mg/mL, not as listed in Advantage 2006v1.

Daily drawing winners at our PittCon® booth
Monday: Dr. S. Todd Swanson, University of Nebraska, Lincoln, NE
Tuesday: Dr. Jeffery Loo, General Motors, Milford, MI
Wednesday: Dr. Steven DuBose, Alcon Research, Fort Worth, TX
Thursday: Dr. Shawn Shanmugan, US Smokeless Tobacco, Nashville, TN
Congratulations, gentlemen, and thank you to everyone who visited our booth!



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Professor Emeritus, University of California, Davis
Co-Founder, J&W Scientific, Inc.
Co-Founder, AirToxics, Ltd.

Preventive Maintenance for GC

Far too many practitioners install a new column, then ignore the enclosures that came with that column, and blissfully proceed to analysis. For reasons discussed below, most authorities would agree that the "typical" or "generic" test chromatograms that accompany batch-tested columns are essentially useless and can be ignored, but if the enclosures include a test chromatogram *that is specific for that column*, this can be valuable. The user gains a distinct advantage by purchasing individually tested columns. In batch testing, one or more columns are tested and regarded as representative of the quality of that entire batch. But the quality of a given batch of columns - - whether N, N/m, bleed, or level of response to active analytes - - normally follows a Gaussian distribution, and individual testing allows the discriminating manufacturer to identify and discard those columns on the low end of any given quality curve. With batch-tested columns, those sub-standard columns become part of the stock shipped to customers. The individually tested column offers another advantage: the test chromatogram is specific for that column under a set of specified conditions. Manufacturers using the more expensive procedures of individual testing must maintain the QC testing chromatographs in pristine condition. Chromatographs possessing even trace residues in the injectors, detectors, or gas supply lines, or faulty temperature readouts can result in the condemnation of columns that are actually good. To prevent this expensive blunder, injectors, detectors, and gas supply lines must be routinely cleaned and/or replaced; in addition, oven readouts (which do drift) require periodic recalibration, and some manufacturers do this on a weekly basis.[1]

The test chromatogram for an individually tested column illustrates the chromatogram produced by a specific test sample under specified conditions in a meticulously maintained instrument. After installing a new column, it should be conditioned in accordance with the manufacturer's recommendations. One should then make an injection of that same test mixture under the same conditions and compare the results to the test chromatogram. *Differences in theoretical plate numbers, the relative responses of test solutes, retention factors or separation factors may indicate instrumental problems that should not be ignored.*

Brandies and some wines improve with age, but most other things undergo a time-related deterioration. Neither gas chromatographs (nor unfortunately, the author) are exceptions to this generality. Eventually problems invariably emerge - - for the GC, these can take the form of unsteady baselines, erratic signal, noise spikes, ghost peaks, higher detection limits, and higher bleed. Some users become addicted to a short-term solution: they simply install a new column and the problem disappears - - for a time. This solution is especially common for those analysts under pressure to produce results rapidly because more samples are coming in the door. However, column replacement is but a temporary solution, in that the same semi-volatile contaminants that destroyed the last column are now being trapped on the new column. Until they work their way through the column and to the detector, the analyst is lulled. However, eventually they do reach the detector, and the problem recurs. In the absence of corrective actions, the interval between the need for replacement columns continues to become shorter and shorter. This dilemma is exacerbated by "real world" or dirty samples, but it also occurs, albeit less frequently, for those analyzing pristine samples.

Where do these problems originate? Few samples are truly clean. It can be educational to place a few mL of the sample on a clean watch glass, allow it to evaporate, and note the residue. In addition to these semi-volatile and non-volatile sample residues, we should be concerned with gas-borne contaminants - - with FID, these would include carrier, combustion hydrogen, make-up, and air. Most chromatographers recognize that there are different purity grades for gases, and the wary analyst specifies "five-nines-purity" (99.999%). This does not negate the need for gas filters (or traps), but usually ensures that the filters will last longer. Removal of oxygen from the gas streams is important. Even traces of oxygen attack the siloxane chain (on which most GC polymers are based), cleave Si-C bonds, and leave terminal Si-OH groups at the points of cleavage. This quickly converts the column into a "bleeder", because the terminal silanols encourage "back-biting" reactions. These split out cyclic siloxanes, primarily trimers and tetramers of (-Si-O-), generating new terminal Si-OH groups at the points of cleavage. Oxygen traps normally pay for themselves because columns experience longer lifetimes. Water traps are also important, not for the stationary phase per se, but because water or water vapor will cause most oxygen scrubbers to deteriorate rapidly. Hence good judgment dictates that the carrier should be passed first through a water trap, then through a bulk oxygen trap, and finally through an indicating oxygen trap. Traps should be mounted vertically, never horizontally. Most are filled with particulate materials and in the horizontal position the particles can settle, leaving an overhead void that provides a path of lower resistance through which most of the gas will flow.

By comparing the performance of a replacement column with the test chromatogram specific for that column, paying attention to sample composition and cleanliness, conducting proper injector and detector maintenance, and using high quality gas purifiers even with high purity gases, column lifetimes can be extended, and down times become a rarity. A regular schedule of preventive maintenance does pay dividends.

[1] Temperature exercises an exponential effect on solute retention factors (k). One of the more precise methods of re-setting the oven readout is to reserve, solely for that purpose, a "recalibration column" on which solute k values at known temperatures have been predetermined. Temperature controls are manipulated to produce the proper solute retention factors, and the readout reset to the proper value.