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Letter to the Editor

# Experiences with HPLC-grade solvents

Chromatographers usually assume that the special HPLC-grade solvents available from several manufactures provide the level of quality and reproducibility that is needed for high-quality separations. After all, the label specifications spell out the level of merit, and the significant price of the solvent should justify the assumed excellence. Unfortunately, however, in some cases, the quality assumed for these solvents is not provided. I cite recent incidents that suggest that we should always be wary of solvents, particularly when unexplained problems arise.

## Acetonitrile

After several months of success, studies with a new prototype column used for the reversed-phase separation of synthetic peptides suddenly went sour. After a brief period with the usual acetonitrile–aqueous trifluoroacetic acid gradients, efficient, well-packed columns quickly developed high back pressure, much lowered plate numbers and poor peak shapes. After two weeks of frustrating work, we concluded that the prototype column packing was not the problem source. During this time the operator attempted to replace the column inlet frit of a column that went "bad". Much to his surprise, the porous stainless-steel frit was removed only with great difficulty, and when it finally released, about a millimeter of packing was stuck to it. This ridge of packing on the frit was very hard, and particles were essentially glued together. The remaining inlet of the column packed bed was in the same shape. We suspected that a polymeric material was building up in the column inlet. Having no further ideas as to the cause of this problem, we changed sources for water, trifluoracetic acid and acetonitrile. When acetonitrile from another manufacturer was used, the problem immediately disappeared.

In discussing the problem with the manufacturer of the original acetonitrile, we learned that this solvent now is purified by an adsorption process, rather than the previous (and traditional) distillation over permanganate. We speculate that the adsorption process, as exercised, failed to remove material (an unsaturate?) that polymerized upon contacting the inlet frit or the packed bed. This undesirable material apparently is removed by permanganate/distillation, and may be removed by adsorption performed properly. Shifting to a solvent made by another manufacturer solved this vexing problem.

## Dichloromethane

When using this solvent for normal-phase separations with unmodified silica, we suddenly observed problems with irregular peak shapes and varying retention times. The HPLC instrument was functioning properly, and other checks showed that the columns were appropriately packed. After much frustration, we were suspicious that the solvent may be problem (obtained from the same manufacturer that supplied the tainted acetonitrile above!). Some of this dichloromethane was shaken with an equal volume of water; the aqueous phase became strongly acidic. Based on this result, we speculated that this dichloromethane (tested from several fresh bottles) contained hydrochloric acid, likely

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formed by the hydrolysis of phosgene created by the oxidation/degradation of dichloromethane. Again, HPLC-grade solvent from another manufacturer immediately cleared up the problem. We also were able to suitably purify the contaminated solvent by treatment with Florisil.

The lesson learned by these experiences is that solvent purity should never be taken for granted. If problems occur for no definable reason, a change to solvents from another manufacturer may be the answer.

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