PRECISION IN LC ANALYSIS

Many LC users are curious about the precision they can expect from an analysis. This question is often answered with specifications concerning flow rate precision, injection volume precision, etc. The chromatographer, however, is interested in the precision of his analysis which is influenced by all of the variables in the LC system, including operator error.

To address this issue, the 1979 ASTM E-19 Committee ran a cooperative program on the study of the quantitation of high performance liquid chromatography (1) which revealed that the technique is generally accurate and precise with relative standard deviations ranging from 6% to a worst case value of 16% for between-laboratory reproducibility of individual component analysis. Seventy-eight laboratories participated in the "round robin" test. A variety of reversed-phase columns was used (representing products from twelve manufacturers). Two solvent systems were used, either acetonitrile/water or methanol/water, in a number of ratios and in isocratic and gradient elution modes. UV detectors were used exclusively.

Two samples were each analyzed in triplicate. The first sample was an easily separated four-component mixture; the second was a more complex six-component mixture. Mean values of the analytical data submitted were consistent with the known concentrations of the components in each sample, indicating a highly satisfactory degree of overall accuracy. However, the spread of data, expressed as percent relative standard deviation, revealed analytical problems for some laboratories. Relative standard deviations for the whole data set ranged from 6% to 11% in the first sample. The problems were more serious in the more complex sample with relative standard deviations ranging from 9% to 16%.

A statistical method that identified outlier data eliminated the results of five laboratories' analyses (6%) from the data set of the sample whose components were readily resolved. It eliminated ten laboratories (13%) from the data set of the more complex and difficult-to-separate second sample. The outlier laboratories need to be concerned about the quality of their chromatography. Unstable baselines, noisy detectors, poor resolution of components, and occasional carelessness were evident in most of their data returns.

Removal of data from outlier laboratories reduced relative standard deviations in the first sample to a range of 3% to 5% and in the second sample to a range of 3% to 8%. This latter information was representative of the performance of about 90% of the participating laboratories.

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Reference:
J. of Chromatogr. Sci. (1981) 19 338.