

B84 Purity Determination of Reference Drug Standards and Quantitation of Illicit Drug Samples by NMR

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After attending this presentation, attendees will come away from the presentation with an understanding of how NMR is advantageous in the quantitation of seized drugs and determining the purity of reference standards.

This presentation will impact the forensic community and/or humanity by making the attendee aware of the power of NMR as a quantitative instrument.

Proton (¹H) Nuclear Magnetic Resonance (NMR) Spectroscopy is a rapid, sensitive, accurate, precise, reproducible, and versatile method for determining the purity of reference standards and analyzing illicit drugs and adulterants. No reference standard of the target compound(s) is required, and aqueous and organic deuterated solvents enable great flexibility in dissolving the drug(s) of interest.

For quantitative analysis, a weighed sample is dissolved in a deuterated solvent(s) with a high purity internal standard. The 7 minute NMR experiment employs 8 scans using a 45 second delay and 90° pulse. In the determination of reference standard purity, the number of quantitative values available is equal to the number of integrals of the compound. The relative standard deviation (RSD) of these signals is usually <1% for pure standards and results agree well with other instruments determining purity.

Unlike separation techniques such as gas chromatography (GC), high performance liquid chromatography (HPLC), and capillary electrophoresis (CE), NMR does not require a linearity study on every compound. NMR response is proportional to the number of nuclei in a given frequency range, not on a compound's functional groups and is not subject to stationary phase adsorption phenomena. Excellent correlation coefficients (>0.99995) were obtained using methamphetamine HCl dissolved in deuterium oxide (D₂O), with maleic acid as the internal standard. Average recovery was 99.4% with RSD of 0.7%.

In the analysis of seized drug samples, high spectral resolution usually results in at least one signal of the target analyte being free of interferences. In complex mixtures, multiple quantitation results of the target compound are possible in complex mixtures by subtracting the contribution of an interfering compound from an integral where the target compound also exists.

This method is also applicable to the determination of multiple target components in a seized drug sample. For many exhibits good agreement was determined by NMR and other techniques.

Nuclear Magnetic Resonance Spectroscopy, NMR, Quantitative