SFL2

Method: Proton

Identification of Controlled and Non-controlled Substances by Nuclear Resonance Spectroscopy

Scope: General purpose (Proton)

Procedure:

Samples should be prepared using an appropriate solvent to dissolve the analyte of interest (ie: maleic acid in deuterium oxide (D2O)) containing 4,4-dimethyl-4-silapentane-1-sulfonic acid (DSS), tetramethylsilane (TMS), or trimethylsilylpropanoic acid (TSP) for 0 ppm reference at a recommended concentration ranging from 5-30mg/mL, but can be more or less depending on the sample. Vortex the sample for several seconds. If insolubles are present, additional solvent (not containing maleic acid or the reference compound) may be added. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate additional insolubles. Place sample into an NMR tube, cap it and place it in an NMR turbine properly.

Method Parameters:

Instrument: Varian/400MR
Minimum Spectral Width (sw): at least containing -1 ppm through 13 ppm
Minimum Delay Between Pulses (d): ≤45 seconds
Minimum Pulse Angle: ≤ 90 degrees
Minimum Acquisition Time: ≥ 2.5 seconds
Minimum Number of Scans: ≥ 1 scan (as necessary to enhance signal to noise)

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra, or spectra from another ISO/IEC 17025. Overall sample spectral pattern corresponds to that of the reference spectrum acquired using the same solvent. Prominent measured signals in the sample spectra were all within 0.2ppm of those in the reference spectrum. No unexplainable extraneous signals are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.