B174 Quantification of Controlled Substances in Simulated Samples Using Attentuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) and Principal Components Regression

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After attending this presentation, attendees will understand how ATR-FTIR spectroscopy can be used in conjunction with multivariate statistics to develop and evaluate models for the quantification of controlled substances in submitted samples.

This presentation will impact the forensic science community by providing a rapid and non-destructive method to quantify controlled substances in samples using ATR-FTIR and multivariate statistical procedures.

ATR-FTIR is widely used for the analysis of submitted samples suspected to contain controlled substances. The ATR sampling accessory minimizes sample preparation and is relatively non-destructive, while FTIR is a Scientific Working Group for the Analysis of Seized Drugs Category A technique, enabling definitive identification of controlled substances in submitted samples.

In this research, Principal Components Regression (PCR) was investigated as an additional tool to aid in identification and quantification of controlled substances in sample mixtures analyzed by ATR-FTIR. PCR is a multivariate statistical procedure that combines Principal Components Analysis (PCA) and multiple linear regression. The former is used to calculate scores for the samples that are then used in the latter to generate a calibration curve that can be used for quantification purposes.

To test the utility of PCR for this application, two-component sample mixtures containing either amphetamine or methamphetamine as the controlled substance and caffeine as a cutting agent were prepared, containing 20%-100% (w/w) controlled substance, in 20% increments. These mixtures were used as the training set for the PCR model. A second set of mixtures was prepared, again using amphetamine and methamphetamine as the controlled substance and caffeine as the cutting agent. Mixtures in this set ranged from 10%-90% (w/w) controlled substance, in 10% increments. This set was used as the test set to evaluate the PCR model. All sample mixtures were then analyzed in triplicate by ATR-FTIR.

Prior to statistical analysis, data pretreatment procedures are often necessary to ensure that variation observed among the samples is chemically relevant. Examples of pretreatment procedures for spectral data include baseline correction, smoothing, scatter correction, and normalization, all of which were investigated in this research. The pretreated data were subjected to PCA and the effect of the pretreatment was evaluated based on the change in the association of replicates in the PCA scores plot compared to the untreated data. For this data set, the greatest improvement in the association of replicates was observed after applying a log transformation, followed by the baseline correction and smoothing functions available in the instrument software, and finally, standard normal variate normalization.

To develop the model, PCA was first performed on the pretreated training set data to generate scores and loadings matrices. The number of Principal Components (PCs) to include in the model was investigated using leave-one-out cross validation. The optimal model retained seven to nine PCs, with validation errors ranging from 2%-5%, indicating acceptable model performance. Multiple linear regression was then performed using the scores and PCs, in conjunction with the percent concentration of controlled substance, to generate a calibration curve. The resulting calibration curve had a correlation coefficient (r) of 0.98, indicating strong correlation between the predicted and actual concentrations of each controlled substance in the training set.

The performance of the optimized PCR model was evaluated based on the error in the predicted concentrations for the sample mixtures in the test set. Prediction errors ranged between 4% and 7%, which demonstrated the potential of PCR for quantification using spectral data. Thus, the ability to quantify controlled substances by applying PCR to spectral data collected using ATR-FTIR provides additional discriminating information about the samples.

ATR-FTIR, Controlled Substances, Multiple Linear Regression