K5 Validation of Volatile Analysis Using Dual Column Capillary GC

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The authors will present data obtained during the validation of a dual column capillary gas chromatography (GC) procedure. The assay, which is routinely used by the New Mexico Department of Health for evidential ethanol testing and postmortem investigation, was validated in terms of precision, accuracy, matrix effects, carryover, linearity, limit of detection and limit of quantitation. A comparison of quantitative ethanol concentrations using postmortem and antemortem casework using both capillary columns (Restek BAC1 and BAC2), together with a comparison of capillary and packed GC columns is also described.

A targeted analysis is performed for methanol, ethanol, acetone and isopropanol using an Agilent HP 6890 GC equipped with a flame ionization detector (FID). Methanol, ethanol, acetone and isopropanol are identified based upon characteristic retention times relative to the two internal standards, n-propanol and t-butanol.

The limit of detection (LOD) in blood was 0.001 g/dL for all analytes tested. The limit of quantitation (LOQ) for ethanol, isopropanol and acetone was 0.005 g/dL and 0.010 g/dL for methanol. Precision using whole blood was evaluated by replicate analysis of in-house controls (n=8). Intraassay CVs for ethanol, methanol, acetone and isopropanol were 1.1, 1.1, 1.0 and 1.1% at 0.474 g/dL, 1.2, 0.9, 1.5 and 0.8% at 0.158 g/dL, 1.7, 1.6, 2.4 and 1.2% at 0.079 g/dL and 4.4, 3.5, 1.9 and 3.4% at 0.019 g/dL respectively. Intraassay CVs using a commercial whole blood control (BioRad) were in the range 2.2 - 3.1% (n=8). Accuracy was determined using internal and external controls. Accuracy using inhouse blood controls was 99-103% in the concentration range tested (0.039 – 0.379 g/dL). Accuracy using aqueous external controls (Cerilliant) was 96-102% and commercial whole blood controls (Utak Laboratories, BioRad) were within the acceptable limits defined by the manufacturer. Analysis of samples fortified with compounds at concentrations that were unknown to the analyst revealed concentrations of ethanol, methanol, acetone and isopropanol within 95 - 105% of the target concentration. No matrix effects were observed and the calibration was linear at 0.7 g/dL, the highest concentration tested. No carryover for any of the analytes was detected at this level. Quantitative ethanol concentrations in 128 postmortem and antemortem case samples were compared using both capillary columns. Linear regression analysis revealed an R2 value of 1.000 (y = 1.0138x – 0.0003), where BAC1 and BAC2 were plotted on the y and x-axis respectively. The mean interassay CV was 1.48% for casework samples that contained ethanol concentrations in the range 0.010 – 0.367 g/dL. Quantitative results using the new capillary GC procedure were compared with a previously used packed GCFID procedure. Analysis of 108 postmortem casework samples revealed an R2 value of 1.000 (y = 1.0188x – 0.00009), where blood ethanol concentrations using capillary and packed GC columns were plotted on the y and x-axis respectively. The mean interassay CV was 1.41% for casework samples that contained ethanol concentrations in the range 0.010 – 0.277 g/dL.

Headspace Analysis, Ethanol, Capillary GC

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