The goal of this task was to increase total PM mass on the filter.

Three sets of engine tests were performed on separate days. For Set 1 and Set 3, PM for each test cycle was collected on an individual filter. For Set 2, PM for five non-road transient cycles was collected on the same filter. For the ramped modal cycle, PM for four cycles was collected on the same filter.

Task 3. Analytical Test Procedures

Development

Both basic (pH 11.2) and acidic (pH 3.1) solvents were used for two-step extraction of acidic and basic compounds. Isotope labeled urea, cyanuric acid (CYA), and melamine (~250 ng) were added to the samples prior to extraction to monitor the analytical performance.

For the solid deposit samples with higher levels of compounds, one-step extraction was sufficient. Due to the extremely variable concentration of the compounds (from 95% to 5 ppm), multistep dilutions were used prior to the analyses.

Solid Urea Derived Deposits Results

Urea deposits were generated in the DEF tube decomposing urea. During the first set, the most abundant urea decomposition products were CYA and ammelide.

Three interesting phenomena were observed: a surprisingly high amount of CYA, a decrease of ammelide collected for the second set of PM filters, and a change in composition when PM was collected from several cycles on the same filter.

Results and Discussion

Urea and Urea Decomposition Compounds Analytical Method Performance

Analytical performance and reliability of the method were evaluated by determining its precision, linearity, limit of detection (LOD), limit of quantification (LOQ) and accuracy. Both intra-day and inter-day precision of peak area for the standards with known concentrations were between 1% and 6% of the relative standard deviations for all compounds quantifying in positive mode and 13% for CYA quantifying in negative mode.

PM Composition Results

Three compounds including urea, CYA, and ammelide were detected in all of the PM samples collected downstream of the SCR during the second set of samples, while only two compounds were observed on PM collected during the first set. The most abundant urea decomposition products were CYA and ammelide.

Three interesting phenomena were observed: a surprisingly high amount of CYA, a decrease of ammelide collected for the second set of PM filters, and a change in composition when PM was collected from several cycles on the same filter.

Conclusions

Major project objectives were met during this program.

• A validated method was applied for the analyses of deposits collected at different exhaust temperatures. Results demonstrated sufficient changes in chemical composition depending on temperature.

• Observations of decrease and increase in surface temperature provided a valuable tool for development of urea injection strategies.

• A new analytical method was successfully applied for PM on two types of the filters commonly used by engine developers. Cyanuric acid and ammelide were detected on all of the filters generated during the individual test cycles, thus increasing overall PM mass. This successful application filled an existing gap in particulate characterization.

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Deposit removal, deposit build up, and deposit removal

Urea deposits in DEF tube prior to collection

Quantitative mass trends correlated with known urea decomposition behavior

Comparison of experimental data (top) with TGA Data presented in literature (bottom) [Schaber, P. et al., “Thermal Decomposition (Pyrolysis) of Urea in an Open Reaction Vessel,” Thermochimica Acta 424, 2004]