

Military Simulant Linearity/Reproducibility with the Dynatherm 9300 TDA

This study details the analysis of the chemical weapon simulants Dimethyl Methyl Phosphonate (DMMP), 1,4-Oxathiane, and Triethyl Phosphate using the analytical technique of thermal desorption. Linearity, reproducibility, and carry over were examined.

A three compound standard mixture was used (30ng/ μ l of each simulant in methanol). Tubes packed with Tenax were spiked with 2,4,6,8, or 10 μ l respectively of the standard mixture. These volumes were equal to 60,120,180,240, and 300 nanograms respectively. Each concentration was spiked in triplicate. The Dynatherm 9300 TDA was interfaced to a GC/MS. The spiked standard sample tubes were thermally desorbed at 300 $^{\circ}$ C/5min. They were desorbed from the focussing trap at 275 $^{\circ}$ C/3min. The GC program was as follows: 60 $^{\circ}$ C/1min, 12 $^{\circ}$ C/min to 220 $^{\circ}$ C.

The three areas of each simulant concentration were averaged and plotted (area/nanograms). The correlation coefficient of each graphed compound was 0.99 (see figures 1-3). Linearity is clearly established for these compounds up to the 300 nanogram range.

A series of eighteen Tenax tubes were spiked with a 4 μ l (120ng) of each simulant each simulant. They were thermally desorbed using the above thermal desorption parameters. Areas were averaged for each simulant and

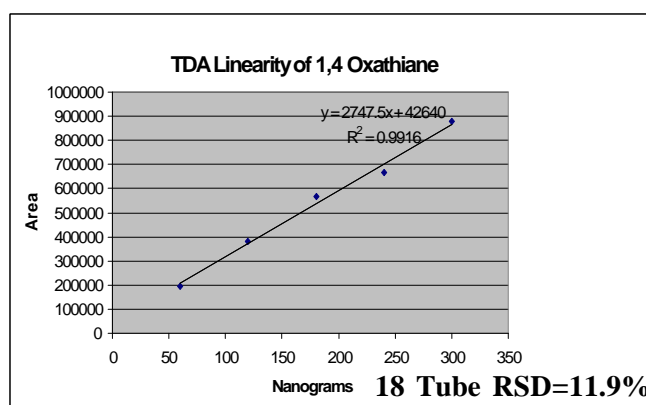


Figure 1

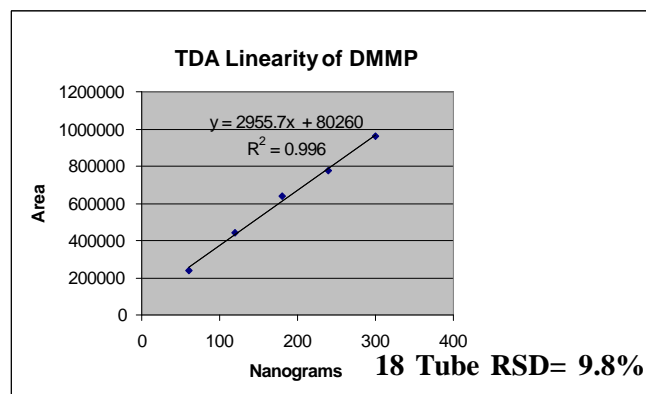


Figure 2

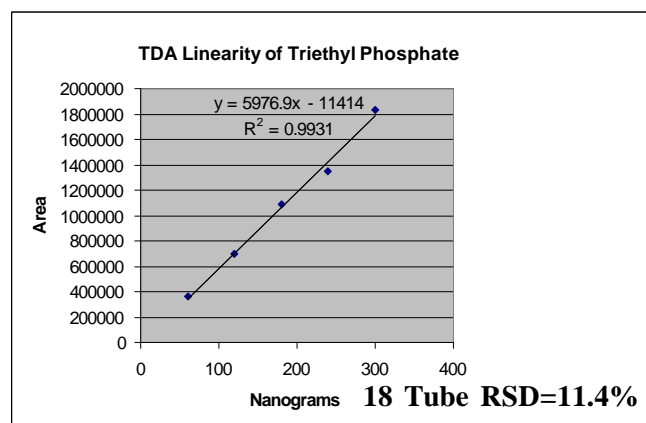


Figure 3

RSD's calculated. The 1,4-Oxathiane had an RSD of 11.9%, DMMP of 9.82% and TEP of 11.44%. Figures 4 and 5 show chromatograms of a duplicate run on the same tube. Figure 5 shows no observable carry over of compounds A (1,4 Ox) and B (DMMP). A negligible amount of C (TEP) is observed.

ANALYTICAL CONDITIONS

Thermal Desorption

Model: CDS 9300 TDA

Valve Oven: 250°C

Transfer Line: 275°C

Sample Tube Desorption: 300°C/5min

Trap Tube Desorption: 275°C/3min

Aux Line; 300°C

Gas Chromatograph/Mass Spectrometer

Model: Varian 3800/Saturn 2000

Carrier: Helium

Column: Varian CP-Select 624
30M x 0.25mm ID x 1.4µm

Oven Initial: 60°C/1min

Ramp: 12°C/min

Final: 220°C

Split: Initial -on-20 @0.03min-off
@1.50min-on-20

Mass Range: 35-500 Daltons

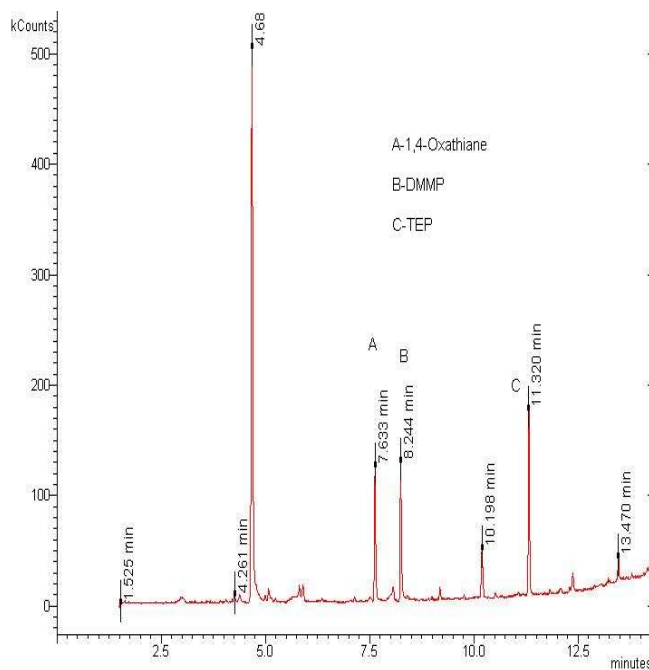


Figure 4

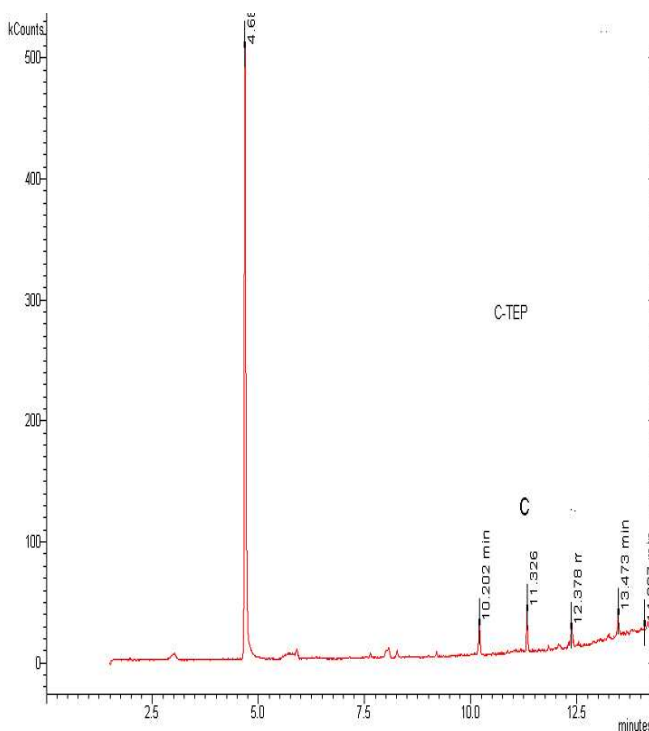


Figure 5

CDS Analytical, Inc. has been a leader in the design and manufacture of laboratory instruments for sample preparation and analysis since 1969. We are dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of pyrolysis, CDS manufactures the Pyroprobe® 5000, 5150, 5200 and 5250 autosampler for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of dynamic headspace instruments for the analysis of volatile organic compounds in environmental, pharmaceutical and food applications, including the model 8400 four-position autosampler for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet your needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, call us at 1 800 541 6593, or log onto www.cdsanalytical.com.