

CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Military Simulant Analysis Using Thermal Desorption

This reproducibility study details the analysis of chemical weapon simulants dimethyl methyl phosphonate (DMMP) and 1,4 oxathiane using Dynatherm thermal desorption equipment interfaced to a gas chromatograph equipped with flame photometric detectors. An auxiliary EPC was used to transfer the analytes directly from the Dynatherm to the head of the GC column, without splitting or using the injection port. Samples were placed onto the sorbent tube in methanol solution, using a microsyringe.

The Dynatherm IACEM Model 980 consists of two sets of adsorbent tubes, one of which was packed with a Tenax-TA/HaySep combination (for collection). The other tube, which served as the focusing trap to recollect the analytes for injection to the GC column, contained Tenax-TA. Side A of the Model 980 was used for the DMMP analysis with the analyte going to the GC and phosphorus detector. Side B was used for the 1,4 oxathiane analysis with the analyte going to the sulfur detector on the GC.

One microliter of simulant standard solution was used for each run. The concentrations were 5, 10, 15, 20, and 30 nanograms per microliter. TriPLICATE analyses were performed on both the DMMP and 1,4 oxathiane, and blank runs were made after each sample. Figure 1 shows the phosphorus detector chromatogram with the DMMP peak retention time of 5.4 minutes. Figure 2 shows the sulfur mode chromatogram of 1,4 oxathiane at a retention time of 5.1 minutes.

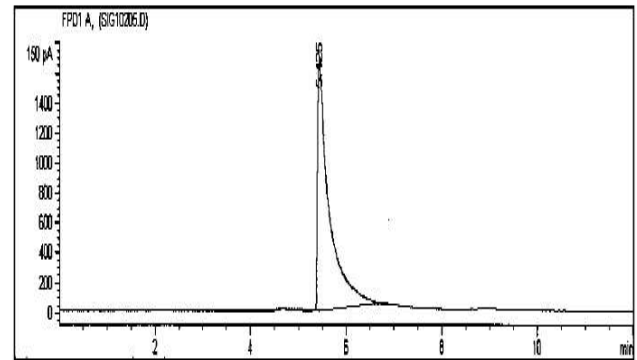


Figure 1. DMMP by FPD in phosphorus mode.

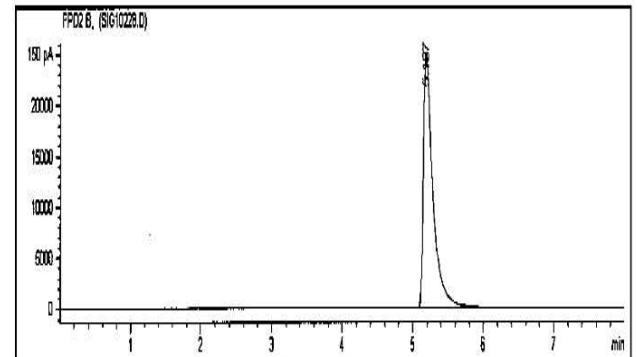


Figure 2. 1,4-Oxathiane by FPD in sulfur mode.

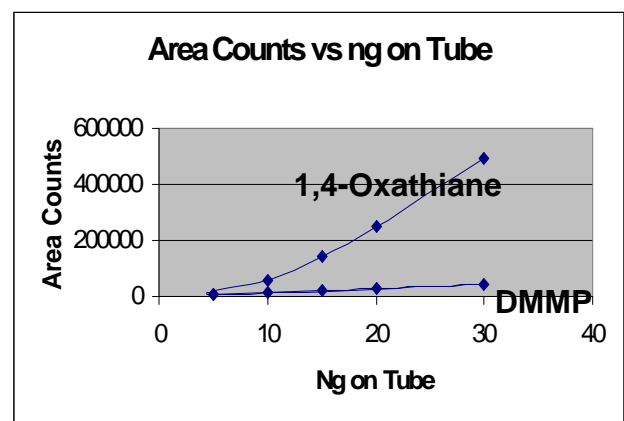


Figure 3.

In Figure 3, the graphs of DMMP and 1,4-Oxathiane peak areas versus concentration are shown. The regression coefficient for the DMMP is 0.9996 and shows good linearity. The curve of the 1,4-Oxathirane appears to be an exponential curve with an coefficient of 0.9964.

ANALYTICAL CONDITIONS

Thermal Desorption

Model: Dynatherm IACEM 980

Valve Oven: 150°C
Transfer Line: 250°C
Sample Desorb: 250°C/3min
Focus Desorb: 350°C/3min
Purge Flow: 30 ml/minute

Gas Chromatograph

Model: Agilent 6890

Carrier: He
Column: RTX 1701 (F), RTX 200 (B)
Oven Initial: 60°C
Ramp: 10°C/min
Final : 140°C
Detector: FPDs
Phosphorus and Sulfur modes



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