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PYROLYSIS GAS CHROMATOGRAPHY

ANALYSIS OF 10 THERMO-LAG

FIRE BARRIER SAMPLES

Performed For:

Texas Utilities Electric
Comanche Peak SES
FM56-5 Miles N.W. of
Glen Rose TX 76043

P. O. Number S 0164511601

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Distribution

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I. ABSTRACT

Inspection of the pyrograms of 10 Thermo-Lag fire barrier samples indicated that eight of the samples were similar in chemical composition. Two samples labeled "Flexi-Blanket" did not generate pyrograms typical of Thermo-Lag samples.

II. OBJECTIVE

Pyrolysis Gas Chromatography (PGC) with Mass Selective Detection (MSD) was used to qualitatively compare five Thermo-Lag fire barrier samples.

III. DESCRIPTION OF METHOD

The samples were compared by pyrolysis gas chromatography using ASTM D3452 as a general guide. A Hewlett-Packard model 5890 series II gas chromatograph equipped with a Hewlett Packard model 5972 mass selective detector was used to generate chromatograms of the pyrolysis products. Pyrolysis of the Thermo-Lag samples were performed with a CDS pyroprobe mounted in an independently heated interface attached to the injection port of the GC. Analysis involved weighing 1-3 mgs. of sample in a quartz tube and placement of the tube in the platinum coil element of the probe. The probe is then placed in the interface and pyrolysed ballistically for 2 seconds. Pyrolytic products are then swept by the carrier gas onto the fused silica capillary column where they are separated and detected with a MSD. Chromatographic and pyrolysis conditions are shown in Table 1. Prior to each analysis, the column is heated to 250°C to elute any volatiles which were not entrained in the polymer.

IV. PRESENTATION OF RESULTS

The ten pyrograms (total ion chromatograms) for each of the ten Thermo-Lag samples are shown in Figures 1, 3, 5, 7, 9, 11, 13, 15, 17 and 19. The extracted ion chromatograms using the acrylate base ion m/e of 55 common to ethyl acrylate (EA) and m/e of 69 common to methyl methacrylate (MMA) for each sample are shown in Figures 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20. The sample name at the top of each figure is the NUCON Log # I. D. Samples 0795-7A-J are further identified in Table 2 along with their respective EA/MMA area ratios. Each set of figures is followed by a library search, which identifies some of the major peaks from each sample's pyrogram, and a summary area percent report.

V. DISCUSSION OF RESULTS

The average extracted ion area ratio for EA/MMA of 1.23 ±0.09 (±σ) shown in Table 2 for the eight samples 0795-A-H is consistent with the average area ratio of 1.3 ±0.2 (±2σ) obtained from other Thermo-Lag samples tested under the NEI generic testing program. The EA/MMA ratio of 2.68 for sample 0795-7I and 2.48 for sample 0795-7J are not consistent with other Thermo-Lag samples tested.

The extracted ion chromatograms shown in Figure 2 for sample 0795-7A, a conduit sample, have an EA/MMA ratio of 1.29. Pyridine compounds identified in the pyrogram (Figure 1) are 3-methyl pyridine, 3, 5-dimethyl pyridine, 2, 3, 5-trimethyl pyridine, 3-ethyl-5-methyl pyridine and 5-ethenyl-2-methyl pyridine. Othe key components identified in the pyrogram are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 4 for sample 0795-7B, a panel sample, have an EA/MMA ratio of 1.25. Pyridine compounds identified in the pyrogram (Figure 3) are pyridine, 3-methyl pyridine, 4-methyl pyridine, 3, 5-dimethyl pyridine, 2-ethyl-6-methyl pyridine, 2, 3, 5-trimethyl pyridine, 3-ethyl-5-methyl pyridine and 5-ethenyl-2-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 6 for sample 0795-7C, a panel sample, have an EA/MMA ratio of 1.11. Pyridine compounds identified in the pyrogram (Figure 5) are 3-methyl pyridine and 3, 5-dimethyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole (visual inspection), pentanedioic acid diethyl ester (visual inspection), octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 8 for sample 0795-7D, a panel sample, have an EA/MMA ratio of 1.22. Pyridine compounds identified in the pyrogram (Figure 7) are 3-methyl pyridine and 3, 5-dimethyl pyridine (visual inspection). Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 10 for sample 0795-7E, a panel sample, have an EA/MMA ratio of 1.34. Pyridine compounds identified in the pyrogram (Figure 9) are pyridine, 3-methyl pyridine, 3, 5-dimethyl pyridine, 2, 3, 5-trimethyl pyridine and 3-ethyl-5-methyl pyridine.. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 12 for sample 0795-7F, a conduit sample, have an EA/MMA ratio of 1.09. Pyridine compounds identified in the pyrogram

(Figure 11) are 3-methyl pyridine and 4-methyl pyridine. Other key components are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester and octicizer (visual inspection).

The extracted ion chromatograms shown in Figure 14 for sample 0795-7G, a panel sample, have an EA/MMA ratio of 1.24. Pyridine compounds identified in the pyrogram (Figure 13) are 3-methyl pyridine and 3, 5-dimethyl pyridine (visual inspection). Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole (visual inspection), pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 16 for sample 0795-7H, a conduit sample, have an EA/MMA ratio of 1.36. Pyridine compounds identified in the pyrogram are pyridine, 3-methyl pyridine, 2, 5-dimethyl pyridine, 3, 5-dimethyl pyridine, 2, 3, 5-trimethyl pyridine and 3-ethyl-5-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 18 for sample 0795-7I, a "Flexi-Blanket" sample, have an EA/MMA ratio of 2.68. Pyridine compounds identified in the pyrogram (Figure 17) are 3-methyl pyridine (visual inspection) and 3-5-dimethyl pyridine (visual inspection). Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 20 for sample 0795-7J, a "Flexi-Blanket" sample, have an EA/MMA ratio of 2.48. Pyridine compounds identified in the pyrogram (Figure 19) are 3-methyl pyridine and 4-methyl pyridine. Other key components identified are pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

In conclusion, the results indicate that eight of the ten Thermo-Lag samples are consistent in terms of chemical composition to other Thermo-Lag samples tested as part of the NEI generic testing program. The remaining two, sample 0795-7I and 7J labeled "Flexi-Blanket" are not consistent with other Thermo-Lag samples.