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PYROLYSIS GAS CHROMATOGRAPHY

ANALYSIS OF 21 THERMO-LAG

FIRE BARRIER SAMPLES

Performed For:

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I. ABSTRACT

Inspection of the pyrograms of 21 Thermo-Lag fire barrier samples indicated that they are all similar in chemical composition.

II. OBJECTIVE

Pyrolysis Gas Chromatography (PGC) with Mass Selective Detection (MSD) was used to qualitatively compare twenty-one 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 twenty-one pyrograms (total ion chromatograms) for each of the twenty-one Thermo-Lag samples are shown in Odd numbered Figures 1-41. 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 even numbered Figures 2-42. The sample name at the top of each figure is the NUCON Log # I. D. Samples 0495-5A-F for Peach Bottom and 0495-6A-O for Limerick are further identified in Table 2 along with their respective EA/MMA area ratios and sample densities. 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 of EA/MMA of 1.30 $\pm$0.10 ($\pm\sigma$) for the Peach Bottom samples, of 1.28 $\pm$0.05 ($\pm\sigma$) for the Limerick Unit 2 samples, of 1.28 $\pm$0.06 ($\pm\sigma$) for the Limerick Unit 1 samples, of 1.29 $\pm$0.07 ($\pm\sigma$) for the Limerick Control Building samples and of 1.29 $\pm$0.07 ($\pm\sigma$) for all twenty-one samples is consistent with average EA/MMA area ratio of 1.4 $\pm$0.1 ($\pm\sigma$) obtained from other Thermo-Lag samples tested under the NEI generic testing program.

The extracted ion chromatograms shown in Figure 2 for sample 0495-5A a 3 hour rated panel sample, have an EA/MMA of 1.35. Pyridine compounds identified in the pyrogram (Figure 2) are pyridine, 3-methyl pyridine, 3, 5-dimethyl 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 (visual inspection), triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 4 for sample 0495-5B, a trowel grade sample, have an EA/MMA ratio of 1.25. Pyridine compounds identified in the pyrogram (Figure 3) are pyridine, 3-methyl pyridine and 2, 5-dimethyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromagrams shown in Figure 6 for sample 0495-5C, a 3 hour rated panel sample, have an EA/MMA ratio of 1.43. Pyridine compounds identified in the pyrogram (Figure 5) are 3-methyl pyridine, 2, 5-dimethyl pyridine, 3-ethyl pyridine, 3-ethenyl-pyridine, 3, 5-dimethyl 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, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 8 for sample 0495-5D have an EA/MMA ratio of 1.26. Pyridine compounds identified in the pyrogram (Figure 7) are pyridine, 3-methyl pyridine, 3, 5-dimethyl 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, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 10 for sample 0495-5E, have an EA/MMA ratio of 1.15. Pyridine compounds identified in the pyrogram (Figure 10) are 3-methyl pyridine and 3, 5-dimethyl pyridine. Other key components identified 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 12 for sample 0495-5F, a trowel grade sample, have an EA/MMA ratio of 1.38. Pyridine compounds identified in the pyrogram (Figure 11) are pyridine, 3-methyl pyridine, 3, 5-dimethyl pyridine, 2, 3, 5-trimethyl pyridine and 5-ethyl-2-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, octicizer, triphenyl phosphate and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 14 for sample 0495-6A, a trowel grade sample, have an EA/MMA ratio of 1.30. Pyridine compounds identified in the pyrogram (Figure 13) are pyridine, 3-methyl pyridine, 3, 5-dimethyl pyridine, 2, 3, 5-trimethyl pyridine and 5-ethenyl-2-methyl pyridine. Other key components identified 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 16 for sample 0495-6B, a 3 hour rated panel sample, have an EA/MMA ratio of 1.27. Pyridine compounds identified in the pyrogram (Figure 15) are 3-methyl pyridine, 2, 5-dimethyl pyridine, and 3-ethenyl-2-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, tris (methylphenyl) phosphate and octicizer.

The extracted ion chromatograms shown in Figure 18 for sample 0495-6C, a 3 hour rated conduit sample, have an EA/MMA ratio of 1.20. Pyridine compounds identified in the pyrogram (Figure 17) are 3-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 20 for sample 0495-6D, a 1 hour rated panel sample, have an EA/MMA ratio of 1.34. Pyridine compounds identified in the pyrogram (Figure 19) are pyridine 3-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, tris (methylphenyl) phosphate, and octicizer.

The extracted ion chromatograms shown in Figure 22 for sample 0495-6E, a 1 hour rated conduit sample, have an EA/MMA ratio of 1.30. Pyridine compounds identified in the pyrogram (Figure 21) 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 24 for sample 0495-6F, a trowel grade sample, have an EA/MMA ratio of 1.28. Pyridine compounds identified in the pyrogram (Figure 23) are 3-methyl pyridine, 2, 4-dimethyl 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, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 26 for sample 0495-6G, a 1 hour rated panel sample, have an EA/MMA ratio of 1.30. Pyridine compounds identified in the pyrogram (Figure 25) are 3-methyl 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 28 for sample 0495-6H, a 1 hour rated conduit sample, have an EA/MMA ratio of 1.31. Pyridine compounds identified in the pyrogram (Figure 27) are 3-methyl pyridine, 3, 5-dimethyl pyridine and 2, 3, 5-trimethyl 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 30 for sample 0495-6I, a 3 hour rated panel sample, have an EA/MMA ratio of 1.19. Pyridine compounds identified in the pyrogram (Figure 29) are pyridine, 3-methyl pyridine, 2-methyl pyridine and 3, 5-dimethyl pyridine. Other key components pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 32 for sample 0495-6J, a 3 hour rated conduit sample, have an EA/MMA ratio of 1.36. Pyridine compounds identified in the pyrogram (Figure 31) are pyridine, 3-methyl pyridine, 3, 5-dimethyl 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, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 34 for sample 0495-6K, a trowel grade sample, have an EA/MMA ratio of 1.30. Pyridine compounds identified in the pyrogram (Figure 33) are pyridine, 3-methyl pyridine, 3, 5-dimethyl 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, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 36 for sample 0495-6L, a 3 hour rated conduit sample, have an EA/MMA ratio of 1.28. Pyridine compounds identified in the pyrogram (Figure 35) are 3-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester and tris (methylphenyl) phosphate (visual inspection).

The extracted ion chromatograms shown in Figure 38 for sample 0495-6M, a 1 hour rated panel sample, have an EA/MMA ratio of 1.19. Pyridine compounds identified in the pyrogram (Figure 37) are 3-methyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole (visual inspection), pentanedioic acid dimethyl ester, (visual inspection), triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 40 for sample 0495-6N, a 1 hour rated panel sample, have an EA/MMA ratio of 1.32. Pyridine compounds identified in the pyrogram (Figure 39) are pyridine, 3-methyl pyridine, 3, 5-dimethyl 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, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 42 for sample 0495-6O, a 3 hour rated panel sample, have an EA/MMA ratio of 1.37. Pyridine compounds identified in the pyrogram (Figure 41) are 3-methyl pyridine and 3, 4-dimethyl pyridine. Other key components identified are 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

In conclusion, the results indicate that the twenty-one Thermo-Lag samples tested are consistent in terms of chemical composition with other Thermo-Lag samples tested as part of the NEI generic testing program.

TABLE 1

Chromatographic Conditions:

30 meter 0.25 mm narrow bore fused silica HP-5 CB capillary column.

Carrier Gas: Helium, 0.9 mL/min, split ratio 35:1

Column Conditions:

Initial Temperature: 50°C for 1 minute hold

Temperature Ramp: 8°C/min to 250°C

Final Temperature: Hold at 250°C for 10 minutes

Injector Temperature: 250°C

Detector Temperature: 280°C

Detector was an HP MSD in scan mode (30-550 amu)

Pyrolysis Conditions:

Pyrolysis Temperature: 650°C

Interval: 2 seconds

Ramp: 2°C/millisecond

Probe Type: Platinum Coil

Interface Temperature: 205°C