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**PYROLYSIS GAS CHROMATOGRAPHY
ANALYSIS OF 5 THERMO-LAG
FIRE BARRIER SAMPLES**

Performed For:

**Florida Power Corporation
Crystal River Energy Complex
15760 W. Power Line Street
Crystal River, FL 34428-6708**

P.O. No. N00989AX

10 May 1995

Distribution

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**NUCON: 06FP801 Master File (1)
Lab (1)**

NUCON 06FP801/01

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NUCON 06FP801/01

I. ABSTRACT

Inspection of the pyrograms of 5 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 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 five pyrograms for each of the five Thermo-Lag samples are shown in Figures 1, 3, 5, 7, 9. Figures 2, 4, 6, 8, 10 are extracted ion chromatograms using the acrylate base ion m/e of 55 common to ethyl acrylate (EA) and a m/e of 69 common to methyl methacrylate (MMA). The area ratios of these two peaks are shown in Table 2 for each sample. Following these two figures is attached a library search which identifies some of the major peaks for each sample tested and a summary area percent report.

V. DISCUSSION OF RESULTS

The average area ratio of $1.35 \pm 0.1 (\pm \sigma)$ for EA/MMA shown in Table 2 is consistent with the average ratio of $1.4 \pm 0.1 (\pm \sigma)$ obtained from other Thermo-Lag samples.

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The extracted ion chromatograms (Figure 2) for sample 0395-4A, a trowel grade sample, have an EA/MMA ratio of 1.28. Pyridine compounds identified in the pyrogram (Figure 1) are 3-methyl pyridine, 2, 5-dimethyl pyridine, 3-ethyl pyridine, 3-ethenyl pyridine, 3, 5-dimethyl pyridine, 2, 3-dimethyl pyridine, 2, 3, 5-trimethyl pyridine, 3-ethyl-5-methyl pyridine and 3-ethenyl-2-methyl pyridine, all consistent with other Thermo-Lag samples. Other key components identified are 2-phenoxy ethanol, pentanedioic acid diethyl ester, triphenyl phosphate, octicizer and trimethylphenyl phosphate.

The extracted ion chromatograms (Figure 4) for sample 0395-4B, a 1 hour rated conduit sample have an EA/MMA ratio of 1.25. Pyridine compounds identified in the pyrogram are pyridine, 3-methyl pyridine, 2, 5-dimethyl pyridine, 2, 3-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, all consistent with other Thermo-Lag samples. Other key components identified are 2-phenoxy ethanol, pentanedioic acid diethyl ester and octicizer.

The extracted ion chromatograms (Figure 6) for sample 0395-4C, a 3 hour rated conduit sample had an EA/MMA ratio of 1.40. Pyridine compounds identified in the pyrogram (Figure 5) are 3-methyl pyridine, 3, 5-dimethyl pyridine, 2, 3, 5-trimethyl pyridine and 5-ethenyl-2-methyl pyridine, all consistent with other Thermo-Lag samples. Other key components identified are 2-phenoxy ethanol, pentanedioic acid diethyl ester, and octicizer.

The extracted ion chromatograms (Figure 8) for sample 0395-4D a 1 hour rated panel sample, have an EA/MMA ratio of 1.50. Pyridine compounds identified in the pyrogram (Figure 7) are pyridine, 2, 5-dimethyl pyridine, 3, 5-dimethyl pyridine and 5-ethenyl-2-methyl pyridine, all consistent with other Thermo-Lag samples. Other key components identified are 2-phenoxy ethanol, pentanedioic acid diethyl ester, octicizer and trimethylphenyl phosphate.

The extracted ion chromatograms (Figure 10) for sample 0395-4E, a 3 hour rated panel sample, have an EA/MMA ratio of 1.34. Pyridine compounds identified in the pyrogram (Figure 9) are 3-methyl pyridine, 2, 5-dimethyl pyridine, 3-ethyl pyridine, 3, 5-dimethyl pyridine, 2, 4-dimethyl pyridine and 5-ethenyl-2-methyl pyridine, all consistent with other Thermo-Lag samples. Other key components identified are 2-phenoxy ethanol, pentanedioic acid diethyl ester and octicizer.

In conclusion, inspection of the pyrograms for these five Thermo-Lag samples indicates that they are consistent with other Thermo-Lag samples in terms of chemical composition.