



**NUCON International, Inc.**

P.O. BOX 29151 7000 HUNTLEY ROAD  
COLUMBUS, OHIO 43229 U.S.A.

TELEPHONE: (614) 846-5710  
OUTSIDE OHIO: 1-800-992-5112  
TELEX: 6974415  
FAX: (614) 431-C.

## **PYROLYSIS GAS CHROMATOGRAPHY**

### **ANALYSIS OF 7 THERMO-LAG**

### **FIRE BARRIER SAMPLES**

**Performed For:**

**Florida Power & Light Company  
Turkey Point Nuclear Plant  
10 MI East/O Florida City,  
Palm Drive  
Florida City, FL 33034**

**P.O. No. B 95691-50027**

**22 June 1995**

#### **Distribution**

**Turkey Point: Frank Busch (1)**

**NEI: Biff Bradley (1)**

**NUCON: 06FL830 Master File (1)  
Lab (1)**

9507240040 950713  
PDR ADDCK 05000250  
P PDR

**NUCON 06FL830/01**

NUCON 06FL830/01



NUCON International, Inc.

P.O. BOX 29151 7000 HUNTLEY ROAD  
COLUMBUS, OHIO 43229 U.S.A.

TELEPHONE: (614) 846-5710  
OUTSIDE OHIO: 1-800-992-5192  
TELEX: 6974415  
FAX: (614) 431-0858

Original Issue

Prepared By

W. P. Freeman  
W. P. Freeman

23 June 1995  
Date

Reviewed By

T. S. Keller  
T. S. Keller

27 June 95  
Date

I. ABSTRACT

Inspection of the pyrograms of 7 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 seven 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 seven pyrograms (total ion chromatograms) for each of the seven Thermo-Lag samples are shown in Figures 1, 3, 5, 7, 9, 11 and 13. 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 and 14. The sample name at the top of each figure is the NUCON Log # I. D. Samples 0395-25A-G 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  $1.26 \pm 0.07$  ( $\pm\sigma$ ) shown in Table 2 is consistent with the average 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 0395-25A, a 1 hour rated conduit sample, have an EA/MMA ratio of 1.28. Pyridine compounds identified in the pyrogram (Figure 1) are pyridine, 3-methyl pyridine, 2-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, tris (methylphenyl) phosphate, octicizer and triphenyl phosphate.

The extracted ion chromatograms shown in Figure 4 for sample 0395-25B, a 3 hour rated conduit sample, have an EA/MMA ratio of 1.31. Pyridine compounds identified in the pyrogram (Figure 3) are 3-methyl pyridine. Other key components identified 2, 3, 4, 5-tetramethyl-1H-pyrrole, pentanedioic acid diethyl ester, tris (methylphenyl) phosphate and octicizer.

The extracted ion chromatograms shown in Figure 6 for sample 0395-25C, a 1 hour rated panel sample, have an EA/MMA ratio of 1.33. 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, pentanedioic acid diethyl ester, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 8 for sample 0395-25D, a 3 hour rated panel sample, have an EA/MMA ratio of 1.26. Pyridine compounds identified in the pyrogram are 3-methyl pyridine. Other key components identified are pentanedioic acid diethyl ester (visual inspection), triphenyl phosphate, octicizer and tris (methylphenyl) phosphate.

The extracted ion chromatograms shown in Figure 10a for sample 0395-25E, a trowel grade sample, had an EA/MMA ratio of 0.51. As this was not consistent with other Thermo-Lag samples tested, sample 0395-25E was retested. The extracted ion chromatograms for the retest sample shown in Figure 10b have an EA/MMA ratio of 1.21, which is consistent with other Thermo-Lag samples tested. Pyridine compounds identified in the pyrogram (Figure 9b) 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, octicizer and tris (methylphenyl) phosphate.

NUCON 06FL830/01

The extracted ion chromatograms shown in Figure 12 for sample 0395-25F, a 1 hour rated conduit sample, have an EA/MMA ratio of 1.13. Pyridine compounds identified in the pyrogram (Figure 11) are 3-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 14 for sample 0395-25G, a 1 hour rated panel sample, have an EA/MMA ratio of 1.31. Pyridine compounds identified in the pyrogram (Figure 13) are pyridine, 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, octicizer, triphenyl phosphate and tris (methylphenyl) phosphate.

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



$\frac{1}{2} \left( \frac{1}{2} \right) = \frac{1}{4}$



11

2

•

■

•

 1  
 2
 -3