CHEMTRACE 3563 Investment Blvd. E-6 Hayward CA 94545 Tel: 510 732 1400 Fax: 510 732 1515

SPECIAL PROBLEM ANALYTICAL REPORT

Requestor: Ed Waters
Company: W. M. Plastics, Inc.
Sample Receipt: 01/05/98
Report Date: 01/13/99

Work Order 961651
PO# 99-0003

Subject: ATD-GC-MS Analysis of Outgassing of Activated Gel

Background:
One sample of gel (90-0804) and its activator (90-0002) were submitted for analysis of organic outgassing of the activated gel. As requested, the activator was first mixed (2.5% wt) thoroughly with the gel and cured at room temperature (24 ± 2 °C) for 48 hours before analysis. Outgassing was performed at 50 °C according to Intel protocols.

ATD-GC-MS Analysis:
Outgassing was analyzed using a Perkin-Elmer ATD-400 automated thermal desorption system connected to an HP 6890 GC and an HP5973 MSD. The ATD was used to introduce outgassed components into a GC-MS system. About 30 mg of cured gel was accurately weighed and inserted into a Teflon tube and placed inside a stainless steel thermal desorption tube. The thermal desorption sample tube was heated at 50 °C in a flow of helium gas for 30 min. The volatiles were adsorbed on a Tenax cold trap (-30 °C), which was then heated to 325°C in a few seconds, allowing rapid transfer of volatiles to the GC capillary column through a heated fused-silica line.

Outgassed organic compounds were separated on a 30 mx0.25 mm and 0.25 μm HP-5 Trace Analysis column. The GC oven temperature was programmed from 40 °C (hold for 3.5 min) to 280 °C (hold for 10 min) at 10 °C/min. Chromatogram from 3.5 min and mass spectra in the range of 41-550 amu were recorded. Identification of each outgassing component was first attempted by searching through the NBSK library of 75,000 mass spectra. In cases of matches of high uncertainty, the mass spectra were interpreted to the best of our knowledge to give the estimated compounds or classes of compounds. Quantitation was achieved by using the response factor of the external standard *n*-decane, with an estimated method detection of 0.1 μg/g (ppmw).

It should be noted that the above experimental conditions were best optimized for analysis of outgassing of volatile (B.P. > 70 °C) and semivolatile organic compounds. Very volatile organic compounds, such as acetone and light hydrocarbon (< C_6), etc. may not be retained in the cold trap and eluted out before 3.5 min in chromatogram.

Results and Discussion:
A control experiment on a blank desorption tube with the Teflon insert was carried out at 50 °C and the chromatogram is shown in Figure 1. Only a few very small

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peaks of ATD contaminants were found in the control run, and these peaks are too small to be integrated with the same integration parameters used in sample analysis.

The outgassing results of activated gel are summarized in Table 1 and the chromatogram is given in Figure 2. In general, the early eluted organic compounds from 3.5 min to 11.5 min in Figure 2 are mostly hydrocarbons such as cyclohexanes and branched aliphatic alkanes. More than 85% of the total outgassing compounds are various aliphatic acid esters (C4 – C8) which eluted between 12 min and 15 min. The broad and tailing peak shape in this region suggests that some of the esters may be the decomposition products of some larger heat-labile outgassed organic molecules.

Kefei Wang, Ph.D.
Senior Research Chemist

Samantha Tan, Ph.D.
Technical Director

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Organic Outgassing Analysis by ATD-GC-MS

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Company: W. M. Plastics
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Table 1. Results of Outgassing of Activated Gel (50 °C for 30 min)

Sample	R.T. (min)[1]	Outgassing Compounds	Concentration in ppmw (ug/g)[2]
Activated Gel (Mixture of 2.5% wt 90-0002 and 97.4% wt 90-0804, cured at 24 °C for 48 hours)	3.96	Branched alkane (C10-C12)	3.7
	4.25	Cyclohexane, ethyl-	5.5
	7.87	Decane	5.6
	10.23	2-Propanol, alkoxyl substituted	3.2
	11.28	Pentanoic acid, ester	3.3
	11.86	Unknown (may contain nitrogen)	3.5
	12.24, 12.33, 12.50, 12.74, 12.84, 12.95	Aliphatic acid esters (C4-C8)	1.9×10^2
		Total Outgassing Compounds[3]	**~ 2.2×10^2**

Notes:
1. R.T. = Retention Time, +/- 0.2 min.
2. Quantitation was based on the response factor of the external standard *n*-decane, and the detection limit (DL) was estimated to be 0.1 ug/g (ppmw). Only major outgassing components (> 3 ppmw) were listed.
3. Total outgassing amounts were calculated from all the peaks as shown in Figure 2.

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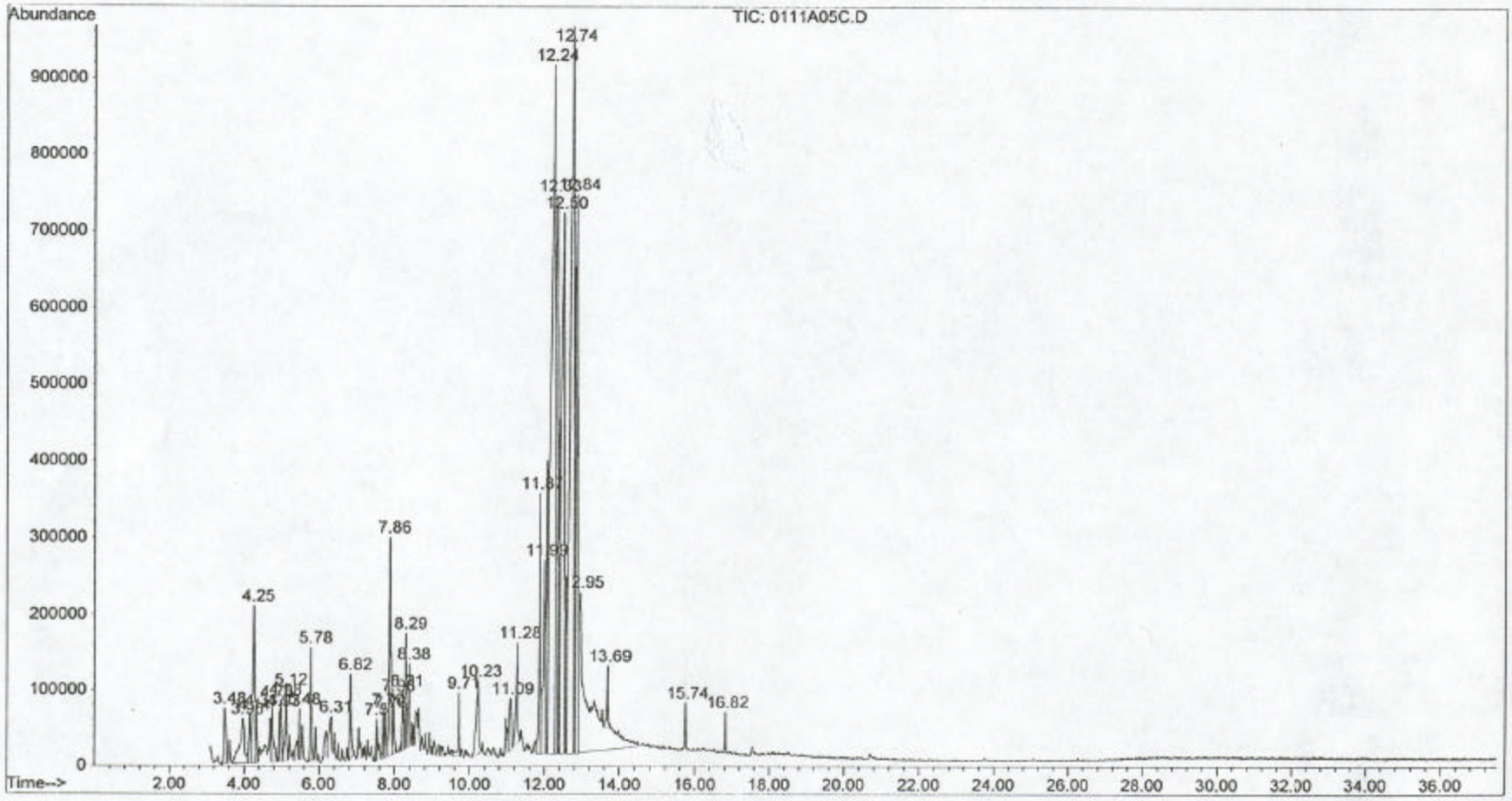
File : C:\HPCHEM2\2\DATA\9901\0111A05C.D
Operator : KW
Acquired : 11 Jan 99 19:33 using AcqMethod OUTGAS1
Instrument : GC6890/MS
Sample Name: 961651 Blue Seal mixed and cured 48 hrs
Misc Info : 50 C for 30 min
Vial Number: 5

Figure 2. GC-MS Chromatogram of 48 hr Cured Gel.



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Operator   : KW
Acquired   : 11 Jan 99  18:43     using AcqMethod OUTGAS1
Instrument :   GC6890/MS
Sample Name: SS Tube Blank w/ Teflon insert
Misc Info  : 50 C for 30 min
Vial Number: 4
```

Figure 1. GC-MS Chromatogra of Blank Tube (Control Experiment)