Using a Standalone Bulk Headspace Sampling Vessel with a Pyroprobe Study Rubber Volatiles

Application Note
Rubber

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Extending the usefulness of the Pyroprobe, the bulk headspace sampler, which can hold items about the size of an orange, can be used for offline collection of headspace volatiles, to be desorbed and analyzed.

A bulk headspace vessel was used to collect outgassing of rubbers under different heating conditions (Figure 1). A piece of rubber was placed in the center of the heating vessel, on an overturned 50 mL beaker. The vessel was sealed and then heated at set temperatures at set times. A flow of helium was then added, to sweep contents onto a desorption tube filled with tenax, and then the tube was inserted into the interface of the Pyroprobe, ready for analysis.

EPDM rubber was heated to 150°C for 3 hours and 20 minutes, and to 165°C for 1 hour and 5 minutes. The rubber heated to a higher temperature but shorter time released more compounds, and the peaks were of a greater intensity (see Figure 2). Figure 3 has peak identifications.

Instrument Conditions
Pyroprobe

Interface: 300°C 4 minutes

Valve Oven: 325°C
Transfer Line: 325°C

Trap Rest: 50°C
Trap Heat: 300°C 4 minutes
Trap Material: Tenax TA

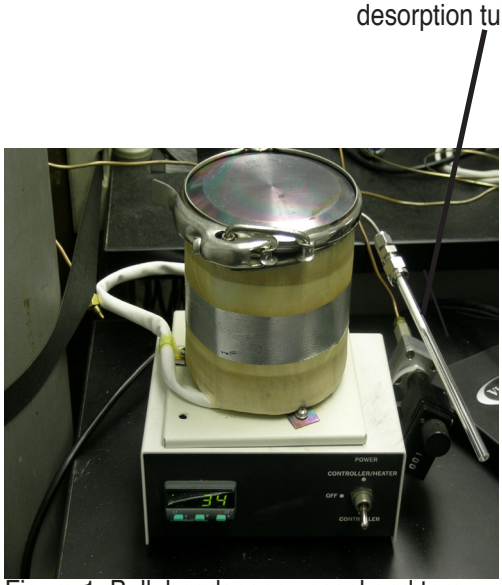

Figure 1: Bulk headspace vessel and temperature controller.

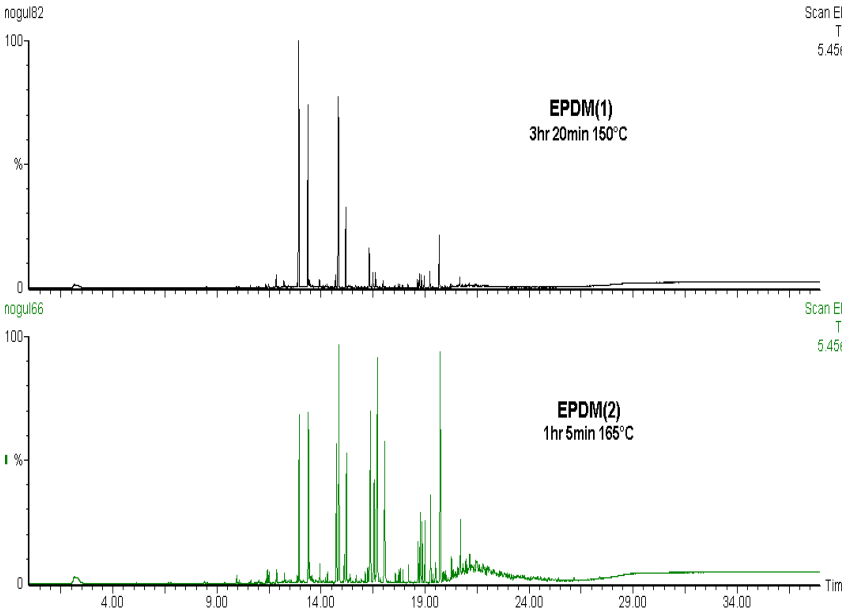

Figure 2: Headspace of two EPDM rubbers.

GC/MS

Column:	HP-5MS (30m X 0.25mm)
Carrier:	Helium, 50:1 split
Injector:	325°C
Program:	40°C for 2 min
	10°C/min to 300°C hold 10 min
Mass Ramge:	35-550

	EPDM(1)
RT	Peak ID
12.94	BENZENE, 1,3-BIS(1-METHYLETHENYL)-
13.38	BENZENE, 1,4-BIS(1-METHYLETHENYL)-
14.84	ETHANONE, 1-[4-(1-METHYLETHENYL)PHENYL]-
16.32	ETHANONE, 1,1'-(1,4-PHENYLENE)BIS-
16.62	ETHANONE, 1-[4-(1-HYDROXY-1-METHYLETHYL)PHENYL]-
18.63	BENZENE, (1-METHYLUNDECYL)-
18.73	BENZENE, (1-PENTYLOCTYL)-
18.82	BENZENE, (1-BUTYLNONYL)-
18.96	BENZENE, (1-PROPYLDECYL)-
19.24	BENZENE, (1-ETHYLUNDECYL)-
19.68	BENZENE, (1-METHYLDODECYL)-

	EPDM(2)
RT	Peak ID
12.95	BENZENE, 1,3-BIS(1-METHYLETHENYL)-
13.40	BENZENE, 1,4-BIS(1-METHYLETHENYL)-
14.87	ETHANONE, 1-[4-(1-METHYLETHENYL)PHENYL]-
16.39	ETHANONE, 1,1'-(1,4-PHENYLENE)BIS-
16.72	ETHANONE, 1-[4-(1-HYDROXY-1-METHYLETHYL)PHENYL]-
19.28	BENZENE, (1-ETHYLUNDECYL)-
19.73	BENZENE, (1-METHYLDODECYL)-
20.71	BENZENE, (1-METHYLTRIDECYL)-

Figure 4: Peak Identifications for rubber headspace.