SHIMADZU

Application News

Gas Chromatography Mass Spectrometry No.M253

Analysis Using UV/Py -GC/MS System

Methods used for weather-induced degradation testing of polymeric materials include outdoor exposure methods and weather meters, however, measurement using such methods can take weeks or months to complete. Using a system consisting of a combination of a micro-UV irradiator (UV-1047Xe) and a GC/MS makes it possible to observe degradation products generated due to photo/thermal-oxidative degradation processes in a short period of time. Chemical changes in polymeric materials following these degradation processes can also be determined.

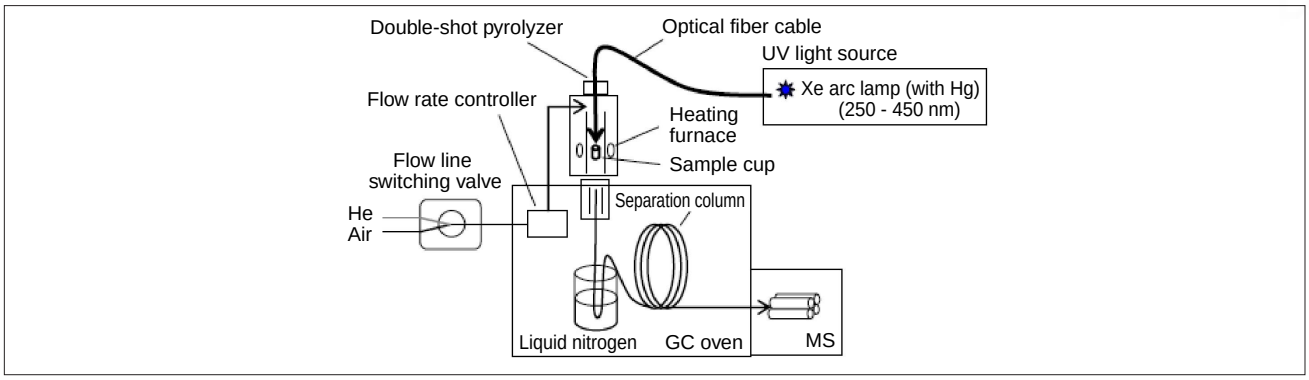

Fig. 1 UV/Py-GC/MS System

Fig. 1 shows the configuration of the UV/Py-GC/MS system.
UV light within a wavelength range of 280 - 450 nm is delivered from a xenon (Xe) arc lamp via an optical fiber cable to the center of the double-shot pyrolyzer furnace, and directly irradiates the sample placed in the sample cup of the pyrolyzer. An optional filter can limit the wavelength of the UV light to 300 - 400 nm. During UV irradiation, the atmospheric gas, temperature, and time are all selectable. The volatile degradation products are cryogenically-trapped by immersing the tip of the capillary GC column in liquid nitrogen; the volatile degradation products are then analyzed by TD-GC/MS. In addition, the residual deteriorated polymers in the sample cup can be analyzed by either EGA-MS or Py-GC/MS.

■ EGA Thermogram

After dissolving ABS resin (containing 1 % DBDE) in THF, a quantity corresponding to 50 µg was weighed into an eco cup, and after drying, this was used as the UV-irradiation sample. After 30 minutes of UV irradiation in air atmosphere, the volatile degradation products and Br products (decomposition products of DBDE) were selected for detection. The EGA thermogram results are shown in Fig. 2. Comparing the thermograms acquired without UV irradiation (referred to as "Before UV irradiation") and after 30 min of UV irradiation ("After UV irradiation"), while the other

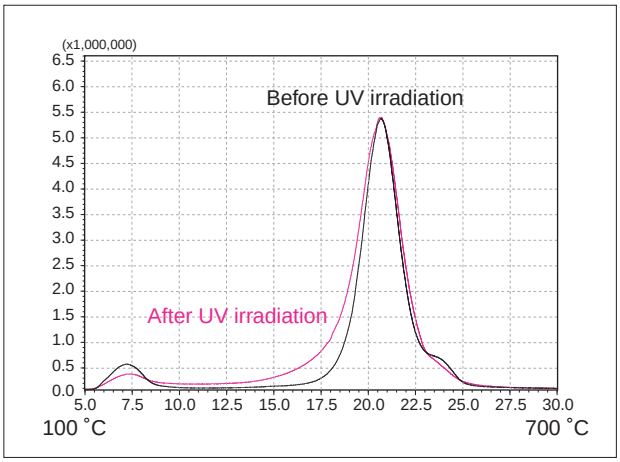

Fig. 2 EGA Thermogram

■ Analytical Conditions

Instrument	: PY-2020iD/UV-1047Xe/GCMS-QP2010 Plus		
Irradiation Temp.	: 60 ˚C	Column Temp.	: 300 ˚C
Atmospheric Gas	: Air	Irradiation Time	: 0.5 h
Separation Column	: UA-DTM (2.5 m × 0.15 mm I.D.)	Sample Quantity	: 10 µg
		Split Ratio	: 1 : 50

■ Analysis of VOCs Generated due to UV Irradiation

Fig. 3 (a) shows a chromatogram of the degradation products generated from this sample during 30 min UV irradiation while heating the sample at 60 ˚C in air atmosphere.
Bromomethane and 1-bromo-3-propanol are observed (Fig. 3 (b), (c)).
In comparison with the chromatogram of the pre-irradiated sample, many oxygen-containing compounds are observed (Fig. 3 (d)).

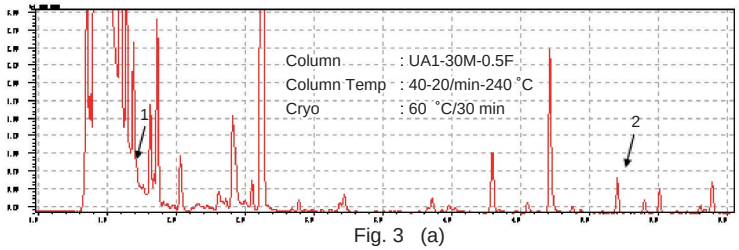

Fig. 3 (a)

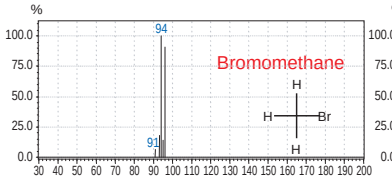

Fig. 3 (b) Peak-1

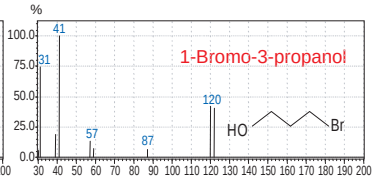

Fig. 3 (c) Peak-2

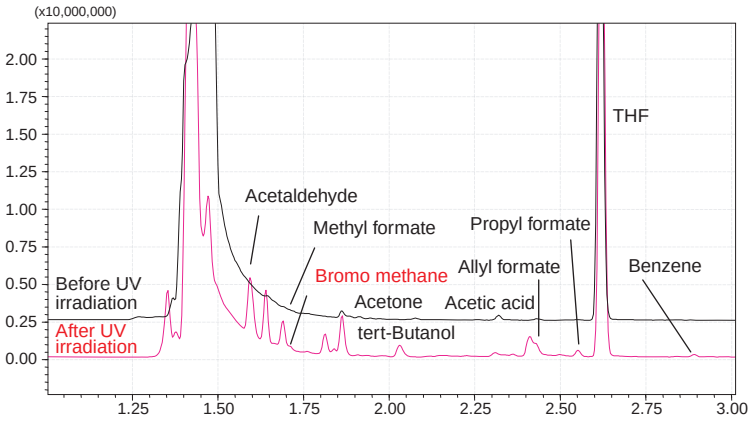

Fig. 3 (d)

■ UV Irradiation Time vs. Peak Area

Fig. 4 shows the relationship between the time of irradiation and the amount of bromomethane and 1-bromo-3-propanol that were generated. Both M+ (94, 120) areas show uniform increases, increasing in proportion to the exposure time.

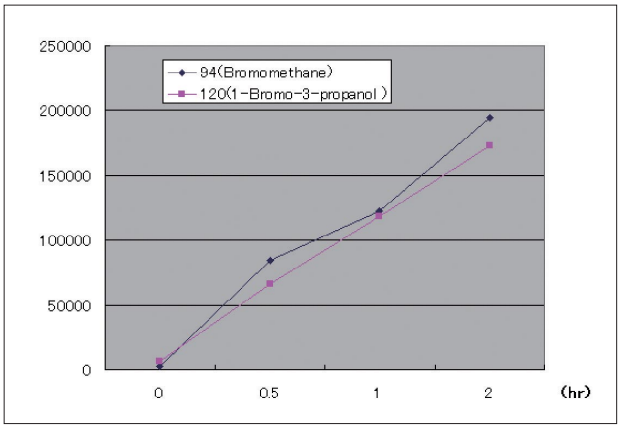

Fig. 4 Relationship Between UV Irradiation Time and Peak Area

■ Conclusion

An evaluation of the micro-UV irradiator was conducted using brominated flame retardant (DBDE) containing ABS resin. Detection of volatile products generated from ABS due to photo/thermal-oxidative degradation was demonstrated, as well as the detection of brominated compounds generated from decomposition of UV-sensitive DBDE. In addition, the correlation between UV irradiation time and the concentration of these substances was also confirmed.

SHIMADZU

SHIMADZU CORPORATION. International Marketing Division
3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

Printed in Japan 3295-03005-10H-IK