APPLICATION BRIEF

Gas Chromatography

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Evaluation of Epoxy Quality by Determination of Volatile Organic Compounds (VOCs)

Introduction

Epoxy resins are used in multiple industries due to their application versatility, like their ability to be designed as adhesives or for coating applications. Epoxy resins are formulated to withstand high temperature applications, they are moisture and chemical resistant, have a high degree of flexibility, and strong adhesive strength properties for binding an array of materials together. For these reasons, epoxy resins are widely used in the production of lightweight automotive and aerospace components, thus it is important to analyze and understand the properties of epoxy resins.

An epoxy is a thermosetting polymer that possesses unique mechanical and resistance properties. Epoxy goes through a chemical curing process, which starts when epoxy hardeners and epoxy resin are mixed. During the curing process, cross-linking occurs, which can take place at room temperature or under high temperature. Cross-linking strengthens the integrity of the epoxy while reducing the amount of monomer off-gasing. When more cross-linking occurs, the cured epoxy is stronger and more rigid. When more cross-linking occurs during the curing process, less off-gasing results in the cured epoxy, therefore the amount of off-gasing is a way to measure the quality of a cured epoxy. In this application brief we determined the volatile organic compounds (VOCs) used in epoxies that will off-gas vapors during the curing process to evaluate cured epoxy quality.

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Experimental

VOC levels are utilized to evaluate the quality of cured epoxies; therefore, an easy, straightforward technique is required to quantitate the hundreds of different unknowns that may be present. This experiment utilizes the technique of calibration by reference which will require only one target compound calibration and its calibration equation. This is a preferable method to save time and money, versus using tens or hundreds of standards. This calibration curve will be used to determine the total sum of concentration of all unknowns in the sample. This technique is commonly used for total off-gasing of industrial materials, including automotive interior items as per VDA-278. P-xylene is the reference standard utilized for this study.

Hardware/Software

This application brief utilizes a PerkinElmer Clarus® 690 Gas Chromatograph (GC) with SQ8 T Mass spectrometer and TurboMatrix HS 110 Trap with TurboMass Software.

Method Parameters

This application brief utilizes a PerkinElmer Clarus 690 Gas Chromatograph (GC) with SQ8 T Mass spectrometer and TurboMatrix HS 110 Trap with TurboMass Software.

Table 1: Method Parameters

HS Conditions	
Instrument	PerkinElmer TurboMatrix HS 110 Trap
Vial and Column Pressure	24.6 psi
Carrier Gas	Helium
Temperature Oven	100 °C
Temperature Needle	105 °C
Temperature Transfer Line	110 °C
Trap Hold	5.0 min
GC Cycle Time	30.0 min
Thermostat Time	20.0 min

GC Conditions	
Instrument	PerkinElmer Clarus 690 GC
Carrier Gas	Helium
Column	Elite-5MS 30m x 0.25 mm ID x 0.25 u
Oven initial temperature	40 °C
Oven initial hold	4.0 min
Ramp	10 °C/min
Final temperature	300 °C
Injector Parameters	
Injector Temperature	PSSI 150 °C
Carrier pressure	21.6 psi
Carrier conditions	Split Flow: 0 mL/minute
Detector Parameters	
Type	PerkinElmer SQ8T Mass Spectrometer
Temperature Transfer Line	180 °C
Temperature Source	200 °C

Consumables	Description	PE Part.no.
Headspace Vials	20 mL Headspace Crimp Top Vials with Write on Patch (1,000/pack)	N9306290
Headspace Crimp Cap & Septum	Pre-Assembled Aluminum Crimp Cap with PTFE/Butyl Septa and Star Spring	N9306265
Vial Crimper	20 mm Hand Crimper	N9302785
GC Column	Elite-5MS 30m x 0.25 mm ID x 0.25	N9316282

Sample Preparation

Four different epoxies were analyzed in this application note. Sample preparation is simple and straightforward. 0.5-2.5g of each prepared epoxy was added to a headspace vial, allowed to cure according to the manufacturer's specifications, then capped and analyzed. A 100 ppb Benzene, Toluene, Ethylbenzene, and Xylenes (BTEX) standard was analyzed for reference before the samples were run.

Data

We have selected a mass spectrum of a prominent peak in each sample and matched this against the NIST mass spectral library for tentative compound assignments as shown in Figures 1 to 4.

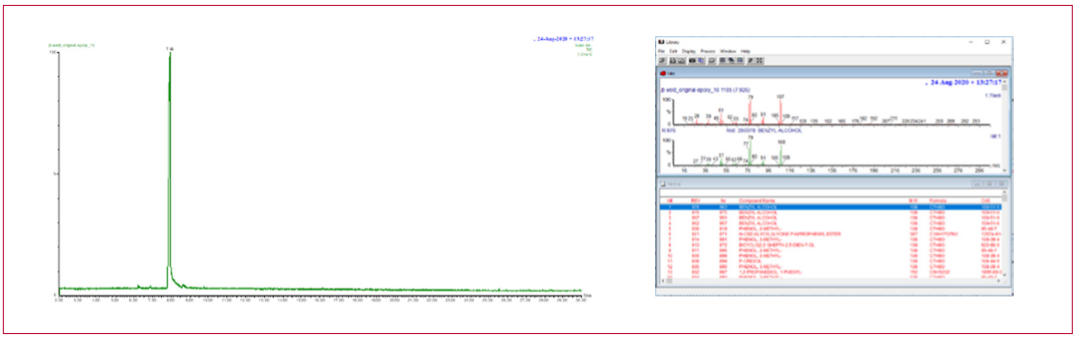

Figure 1: Manufacturer A - Epoxy Chromatograms with Benzyl Alcohol Mass Spectrum.

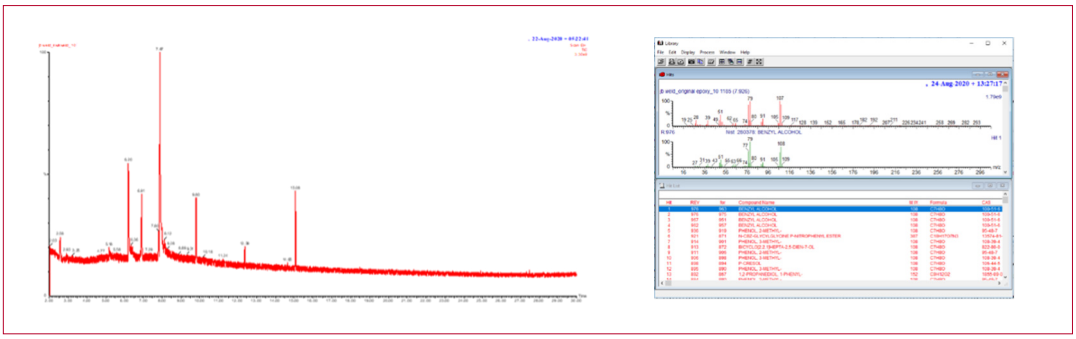

Figure 2: Manufacturer B - Epoxy Chromatograms with Benzyl Alcohol Mass Spectrum.

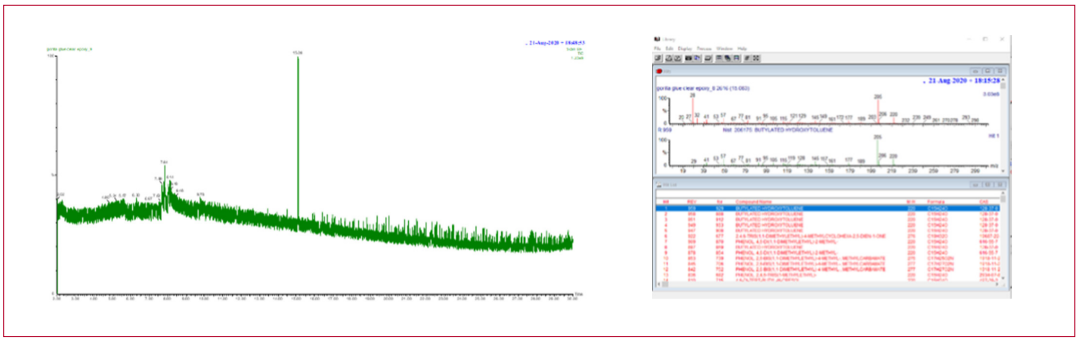

Figure 3: Manufacturer C - Epoxy Chromatograms with BHT Mass Spectrum.

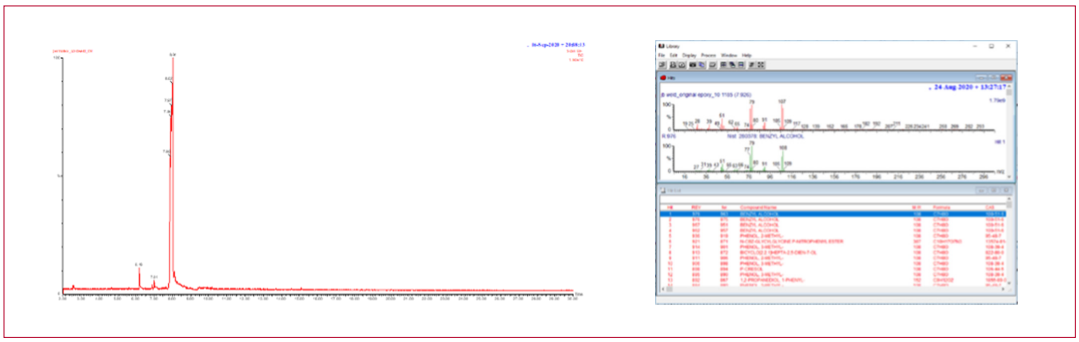

Figure 4: Manufacturer D - Epoxy Chromatograms with Benzyl Alcohol Mass Spectrum.

Results and Discussion

The results demonstrate that by utilizing the PerkinElmer GC/MS-HS solution, one can effectively identify and quantitate the various VOCs found in epoxies. All VOCs of interest were easily detected, even if they were not in the initial calibration; they can still be accurately identified and can be analyzed later with a reference standard. PerkinElmer GC/MS-HS is the perfect addition to any analytical laboratory to identify and quantitate many different organic compounds.

To enhance the data collected, we referenced VDA-278, a VOC and FOG emission test by thermal desorption, where calculations are utilized to semi-quantitate compounds that were detected but not calibrated. These are calculated using p-xylene as a reference which were analyzed at the start of the sequence.

Table 2: Semi-Quantative Results

Epoxy	Response	Sample weigh (mg)	Total VOC as o-xylene (mg)	VOC off-gas per gram (as o-xylene)
Manufacture A	290518432	2020	6248	3093
Manufacture B	36259288	1670	780	467
Manufacture C	5640708	2750	121	44
Manufacture D	440619136	1630	9476	5814

Conclusion

This application brief demonstrates that common epoxies can be effectively analyzed by GC/MS-HS as well as the identification and quantitation of the off-gasing that occurs. In this instance the amount of off-gasing that was measured correlated to the quality of the epoxy. Utilizing PerkinElmer GC/MS-HS is an effective analytical solution in which direct injection can still be utilized even when the GC/MS is configured for headspace sampling, increasing the number of applications the instrument can run.

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