

## **SUCCESS STORY...A005**

### **Deformulation of an Epoxy Hardener**

#### **Project Background**

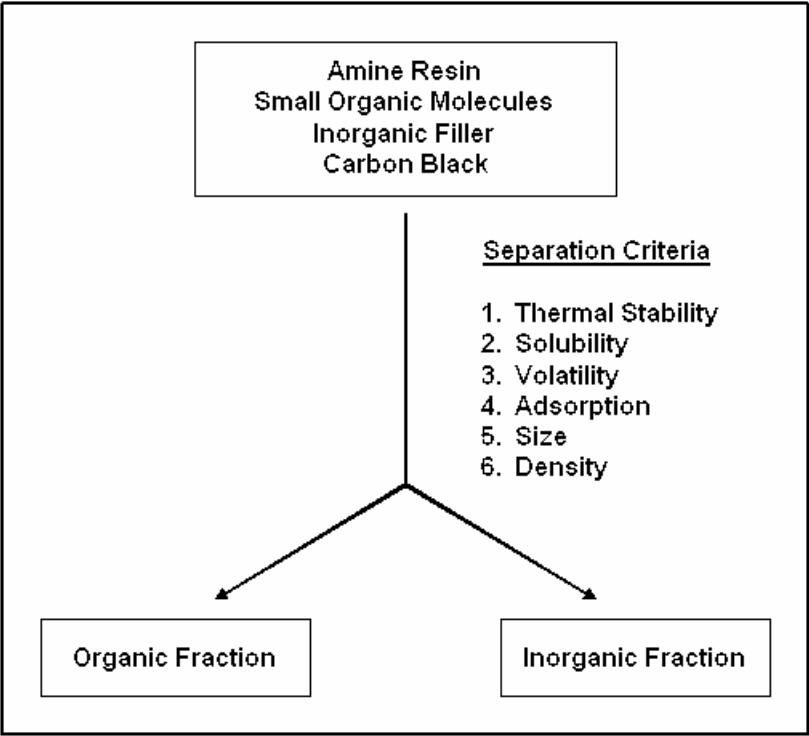
As more specialty chemicals are being imported into North America, there is an increasing need to demonstrate equivalency with materials that were once available locally. In some instances, the critical nature of the final application drives the need to verify the composition in significant detail.

#### **The Opportunity**

A U.S. based manufacturer of high tech products wished to determine the composition of an amine based hardener used with epoxy resins to make thermosetting adhesives. ANALYZE was asked to do a complete deformulation of the hardener.

#### **ANALYZE's Approach**

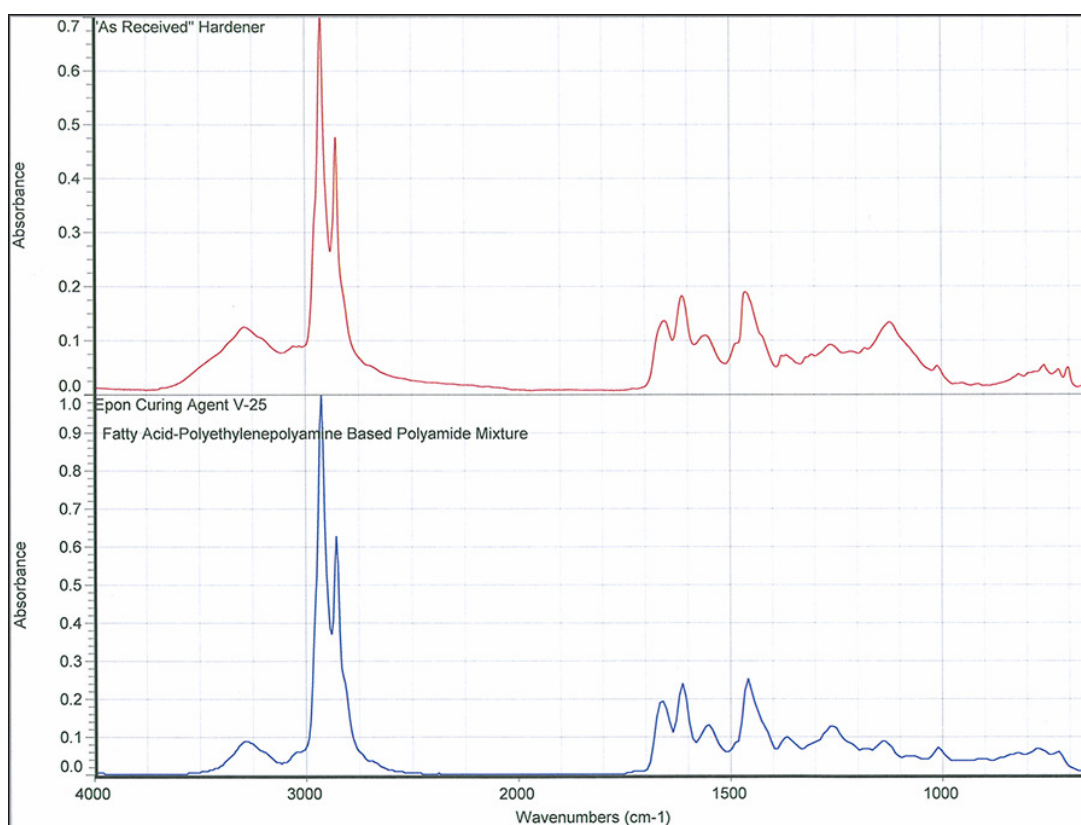
The hardener was reported to be an admixture of polyamine resin, small organic molecules, a mineral filler and carbon black. Deformulation of this product utilized multiple instrumental methods and laboratory procedures to determine the chemical composition. While there are analyses that can be done on the 'as received' material (e.g., FTIR), given the fact that the hardener is formulated from a number of raw materials, separation of the admixture using the criteria noted in the following analysis scheme was required.



## The Result

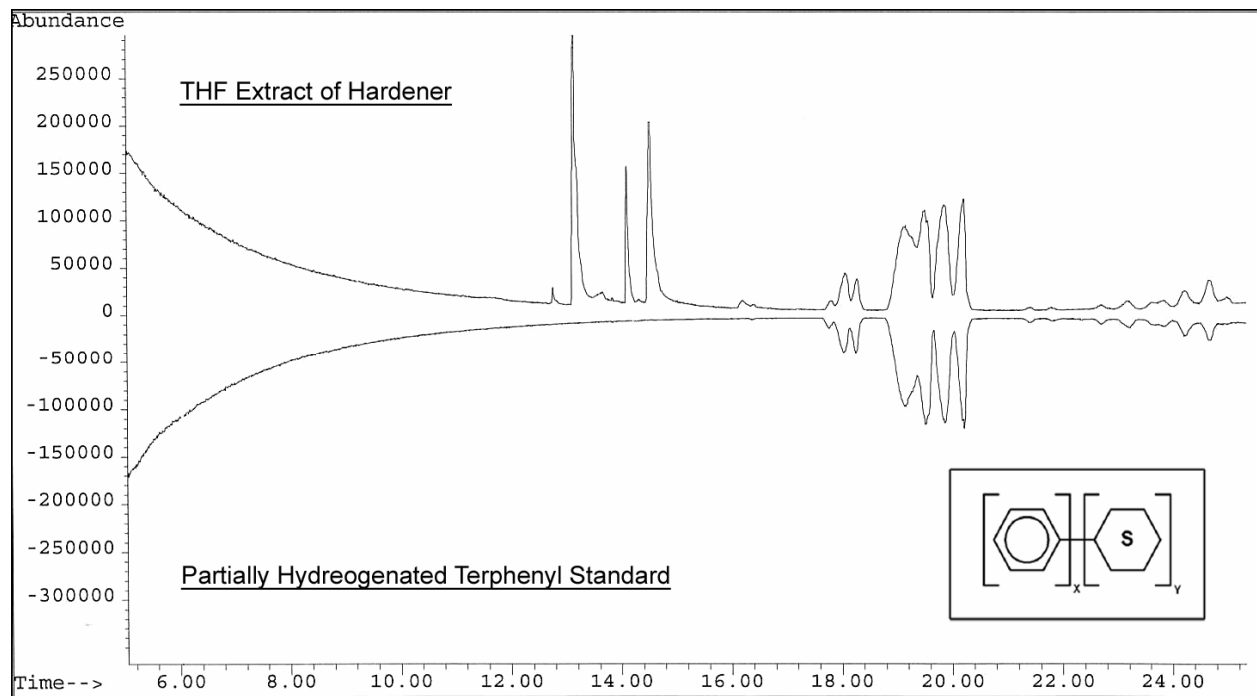
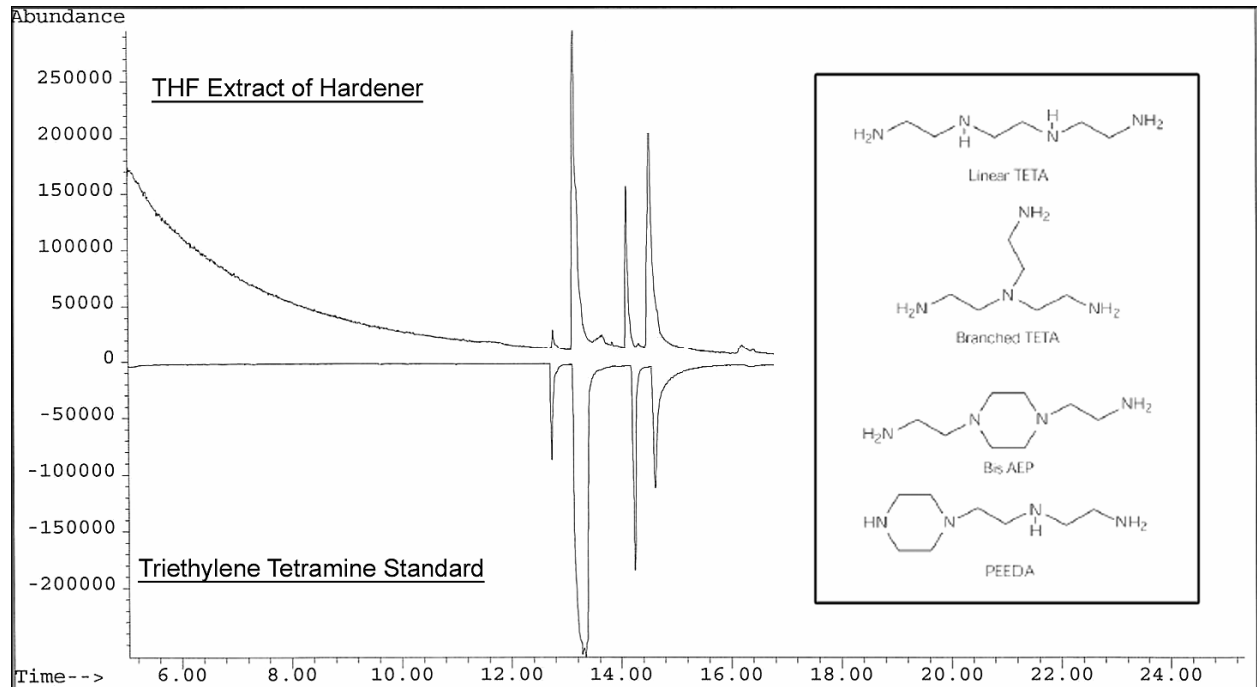
The following text highlights some of the key information obtained during this materials characterization project.

Fourier Transform Infrared Spectroscopy (FTIR) provides a very useful starting point for the identification of most organic and some inorganic compounds. The IR spectra shown in the following figure suggest that the hardener is mainly composed of a polyamide synthesized from the reaction of fatty acids and a polyamine.

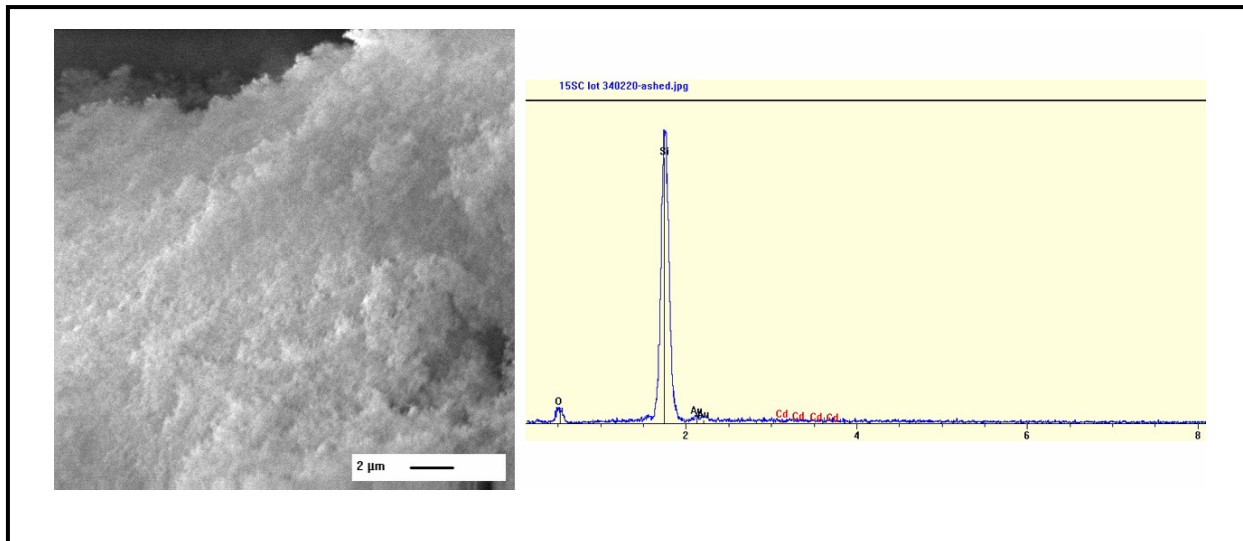


The organic fraction (polyamide and small organic compounds) can be separated from the inorganic compounds by solubility and density. The small organic compounds can be separated from the larger polyamide by a variety of chromatographic methods. Based on the volatility criterion, gas chromatography is the most direct method. Different small molecules can be further separated from each other by their temperature dependent volatility and specific interactions with the stationary phases coating the inner wall surface of the chromatography column. The following two chromatograms show that the technical

admixtures of triethylene tetramine and partially hydrogenated terphenyls are the main two small organic molecule components of the hardener. The specific chemical structures of the components shown in the boxes were determined by comparison of the unknown mass spectra to known reference library spectra.



The inorganic compounds were separated from the organic compounds based on thermal stability by thermogravimetric analyses and solubility/density by their insolubility in organic solvents and centrifugation. The amounts were determined by gravimetric analysis. Identification of the inorganic filler as amorphous silica was done by FTIR, elemental analyses and microscopic inspection of the TGA residue and the residue remaining after centrifugation. For example, only silicon and oxygen were detected in the energy dispersive spectrum of the residue remaining after the hardener was ashed in air.



The presence of carbon black was inferred by the hardener's black color, its disappearance when exposed to an oxygen plasma or heated in an open crucible.

### **Conclusion and Benefit To The Client**

The hardener contains five main ingredients and a small amount of water. Based on comparison of the separated hardener components to a number of independently sourced raw materials used as standards, the best quantitative determination for the hardener formulation approaches 100% mass balance.

<b>Component ID</b>	<b>Found Wt-%</b>	<b>±1 S.D.</b>
<b>Polyamide Resin</b>	<b>78.7</b>	
<b>Triethylene Tetramine</b>	<b>5.13</b>	<b>0.15</b>
<b>Hydrogenated Terphenyls</b>	<b>11.0</b>	<b>0.6</b>
<b>Amorphous Fumed Silica</b>	<b>3.0</b>	<b>0.3</b>
<b>Carbon Black</b>	<b>1.3</b>	
<b>Water</b>	<b>0.30</b>	<b>0.04</b>
<b>Total Wt-%</b>	<b>99.4</b>	

The client used these results to verify the information provided by the vendor, establish incoming raw material acceptance tests and establish QC methodology to assure accurate mix ratios with the epoxy resin. The implementation of these procedures enabled the client to manufacture their products more consistently with a much reduced scrap rate.