Teel

Detecting a Restricted Substance in Polypropylene and PVC

It is gratifying for an analytical lab when it can develop a custom test method that not only helps an individual client, but also continues to prove useful for subsequent projects, especially when that method can help improve products for regulatory compliance and the benefit of human health.

Teel Analytical Labs (TAL) was able to develop such a test for extracting and detecting bis(2-ethylhexyl) phthalate (DEHP), a common plasticizer used in the manufacture of many plastic products to make them more flexible. Some research has linked DEHP and other phthalates to negative health effects, including reproductive, neurodevelopment, and respiratory harm. Numerous regulatory bodies have acted on this research. The FDA restricts its use in food packaging, the EPA restricts its presence in public water systems, REACH has listed it as an SVHC (substance of very high concern), and California's Proposition 65 requires warnings on products about its potential harm.

Given these regulations, detecting, limiting, and removing DEHP has been a concern for manufacturers of plastic products. One such manufacturer approached TAL about developing a method to detect the material in polypropylene.

The Problem

The customer asked TAL to first add DEHP to virgin polypropylene they supplied. They wanted it added to different samples at two precise levels: 0.5% and 1% by weight. The customer then wanted to see if TAL could develop a test method to detect the DEHP in the samples at both percentages.

Mixing DEHP with Polypropylene

TAL set to work devising a way to add the DEHP to the polypropylene supplied by the customer. To mimic the extrusion process where plastic is melted and combined with plasticizers and other additives, TAL first attempted to melt the polypropylene and then mix in liquid DEHP. Using a balance and an aluminum pan, TAL first weighed the polypropylene resin pellets, calculating the appropriate weight and DEHP needed to achieve the customer's specifications.

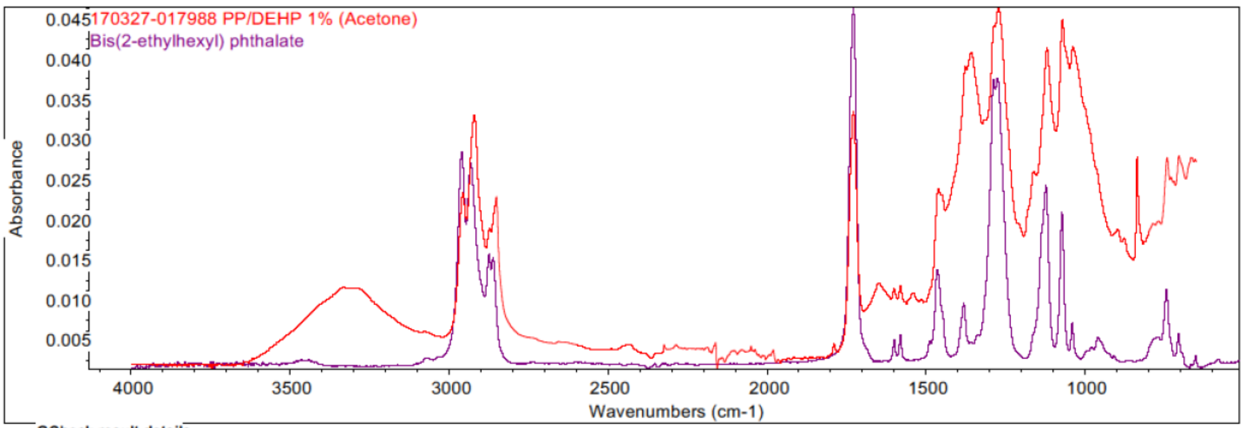
The first melting method they tried was heating the pellets on the pan using a hot plate. Unfortunately, this proved ineffective, as the hot plate applied insufficient heat to allow the DEHP to distribute the throughout the material. TAL then used a hydraulic lab press to make compression molds of the polypropylene and DEHP mixture. The top and bottom heating plates of the press helped the DEHP move through the polypropylene more easily and proved successful in creating samples with the specified percentages. Now having solid samples at the two percentages needed, TAL moved on to finding a way to detect the DEHP.

Devising a DEHP Detection Method

TAL cut the 0.5% and 1% molds into approximately 2.5-gram pieces and ran Fourier Transform Infrared Spectroscopy (FT-IR) tests on the samples to see if the DEHP could be detected. FT-IR tests detect chemical bonds in a test sample and produce a profile comparing the substances in the sample against a catalog of materials, allowing lab techs to determine what materials are present in the sample. The FT-IR analysis of the samples failed to detect the DEHP. TAL then decided to apply solvents to the pieces to see if the DEHP could be drawn out of the polypropylene for easier FT-IR detection.

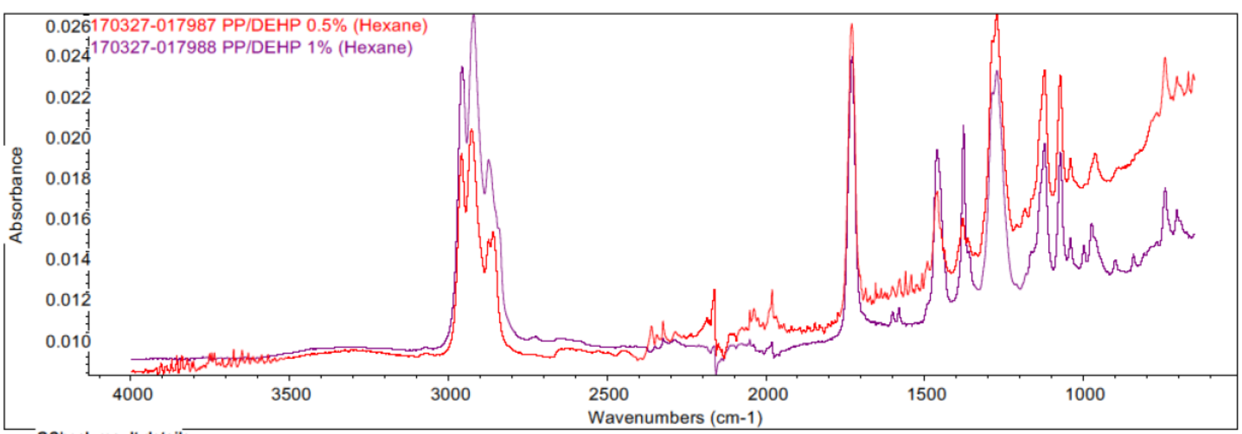
The first solvent applied to the pieces was acetone. TAL placed a piece at each of the two DEHP levels into a 2oz. glass jar with the acetone and then placed the jar into a shaker on low for 30 minutes to dissolve the polypropylene. The acetone containing the dissolved polypropylene sample was then decanted into a new 2oz. jar and allowed to evaporate in a chemical hood. Once the dissolved mixture had dried, TAL tested the residue with using FT-IR again. Yet again, the DEHP was not detected, leading TAL to conclude the acetone was not a sufficiently strong dissolution agent to extract the DEHP for detection.

FT-IR Overlay of Dissolved Acetone Residue and DEHP as a Reference: No Match to DEHP



After investigating other potentially more effective solvents, TAL tried the same method of dissolution and FT-IR analysis with hexane. TAL found research indicating hexane's general effectiveness in phthalate dissolution and decided to test its effectiveness on DEHP specifically. After analysis of the dissolved and dried sample using hexane, the DEHP was detected with a high degree of confidence.

FT-IR Overlay of the Dissolved Hexane Samples: Match to DEHP

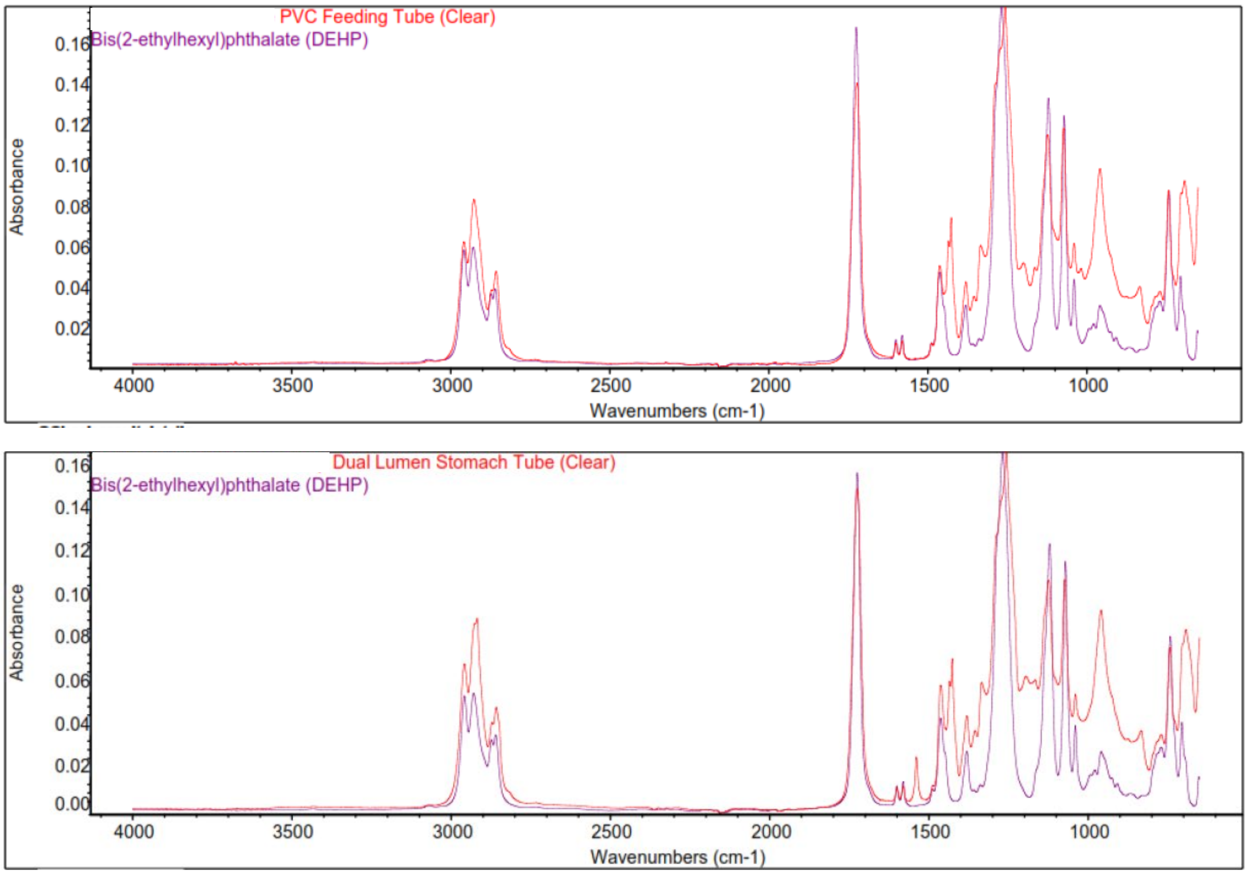


With this success, TAL was able to deliver a method to the customer for detecting DEHP at the specified levels.

Continued Use of the Method

After developing the method, TAL was able to successfully use for reverse engineering work internally at Teel. In multiple projects, the method helped Teel detect DEHP in flex PVC medical products.

FT-IR Analysis of PVC Tubing After TAL's Test Method: Both Samples Show Matches for DEHP



Conclusion

In this instance, TAL was able to not only meet the needs of an individual customer, but also develop an analytical tool that has shown continued usefulness. TAL used their combined troubleshooting expertise and knowledge of solvents to detect a key substance, the presence of which is important in determining a product's regulatory compliance and consumer safety.