CDS olutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Residual Limonene (Hydrocarbon) Analysis in Recycled PET using Thermal Desorption

Plastic bottles that are recycled can contain adsorbed flavor compounds. Recycled PET (Polyethylene terphthlate) is used as packaging for many consumer products including soft drinks. The compound limonene is a very common impurity adsorbed by PET packaged soft drinks.

Limonene and other monoterpenes occur naturally in trees and plants. It occurs in a myriad of products ranging from a component in a metal degreaser to household cleaners and a paint solvent. It should be noted that food is the principle source of limonene exposure (citrus and spices). Although limonene has been found to be a skin and eye irritant, it is catagorized as a compound with low toxicity. However, limonene does have a distinct odor and taste that needs to be removed from the recycled plastic before reuse. In the recycling of PET, the removal of limonene can be verified by performing a thermal extraction of the original (flake, Figure 1) and processed (pellet, Figure 3) samples.

Thermal extraction of volatiles from solids can be accomplished using direct thermal desorption. This technique enables the analyst to characterize as well as quantitate trace compounds such as residual solvents, various additives, and product impurities. Samples for thermal treatment should be homogeneous and be able to fit into a small tube (4mm i.d.). Thermal analysis consists of heating the sample tube and its contents to a certain temperature and time. The volatiles are collected on a packed (Tenax, Carbosieve, etc) narrow bore trap (2mm i.d.) and thermally des-

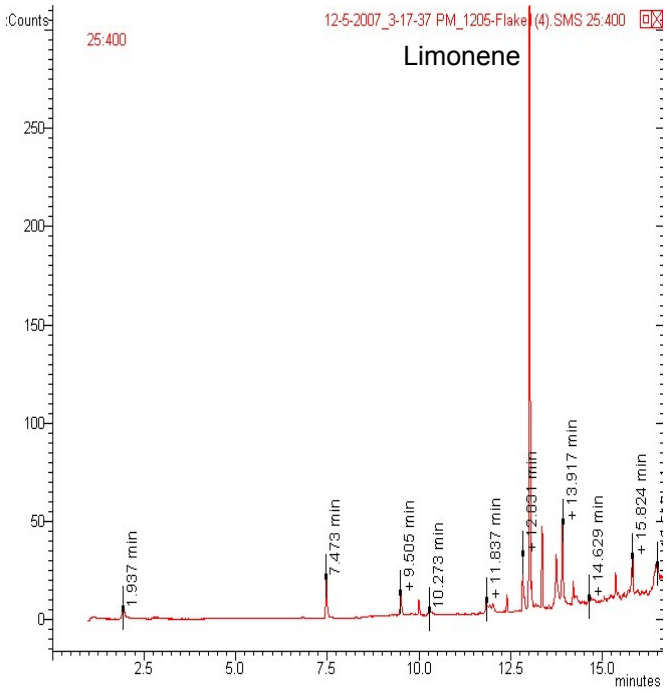

Figure 1. Flake PET

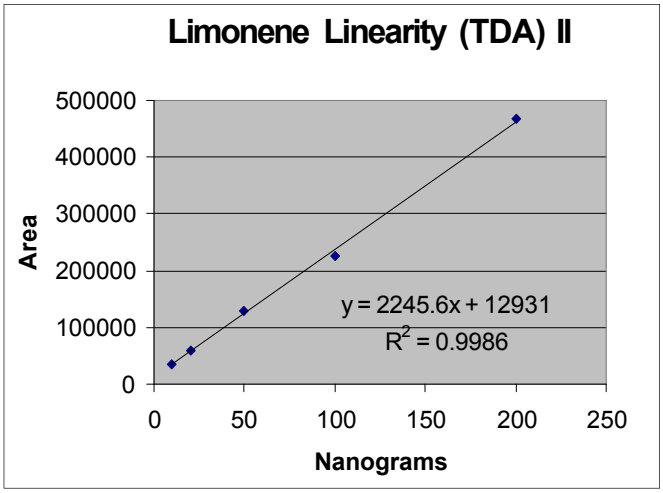

Figure 2.

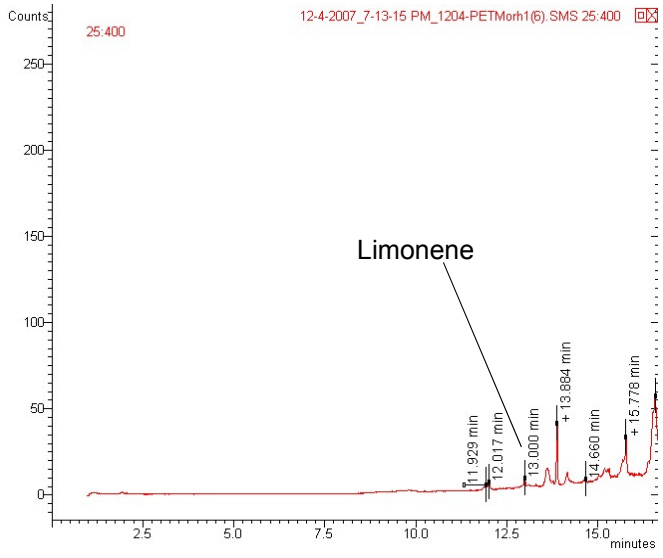

Figure 3. Pellet PET

orbed through a heated transfer line and into the inlet of the GC/MS. To aid in the quantitation of the limonene,a calibration curve was constructed spiking concentrations of 10, 20, 50, 100, 200 nanograms onto glass wool in empty tubes (Figure 2). Samples of recycled flake and pellet PET were thermally desorbed in an 8mm tube. Samples were done in duplicate. The samples were run on the CDS 9300 TDA using a Tenax/ Carboxen/Carbosieve focussing trap. The CDS 9300 was interfaced to a Gas Chromatograph/ Ion Trap. This duplicate analysis gave limonene values of 1.96µg/g and 1.88µg/g respectively for the flake and duplicate 0.003µg/g values for the pellet.

In summary, volatile hydrocarbon analysis in polymeric materials can be quantified using thermal extraction. Standard calibration curves are easily prepared via spiking into adsorbent tubes used in air monitoring. Trapping adsorbents are selected based on the adsorption and ease of thermal release of the compounds.

CDS TDA 9300 Conditions

Valve Oven: 250°C
Transfer Line: 250°C

Tube Idle: 30°C
Dry Tube: 30°C/0min
Tube Heat: 230°C/6.00min
Tube Cool: 0.5min

Trap Idle: 35°C
Trap Heat: 200°C

Interconnect Line: 250°C

GC Conditions

Carrier: Helium
Column: CP-Select 624
(30m x 0.25mm x 1.4µm)

GC Program: 30°C/4min, 15°C/ min to 220°C

Detector: Ion Trap

Additional literature on this and related applications may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

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