CDS Analytical, Inc.

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Residual Monomer in Poly Methyl Methacrylate Analysis using the CDS Model 6500 Autosampler

The analysis of polymeric materials for residual small molecules, including solvents and monomers, is an important part of the quality control process. Although these analyses are frequently done by solvent extraction, thermal desorption offers some distinct advantages. First, there is no solvent to concentrate or dispose of after the analysis. Secondly, since the analysis does not include a large solvent peak, the technique is a sensitive way to identify traces of compounds which might be masked by solvent injection. Thirdly, the entire sample may be transferred to the GC for analysis, as opposed to an aliquot from a solvent extraction.

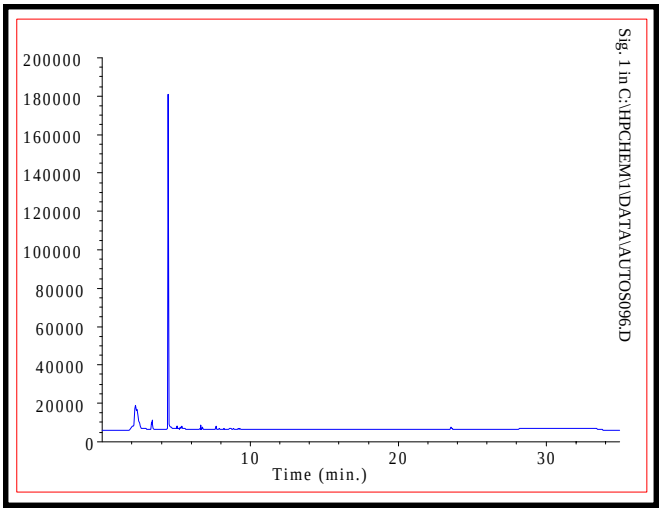

Figure 1. Residual MMA from 1 g of PMMA

Figure 1 shows the residual methyl methacrylate monomer (MMA) collected from a 1 gram sample of PMMA which was placed into a 36 mm diameter sampling chamber of the CDS Analytical 6500 thermal desorption autosampler. The polymer was heated to 100 °C and purged with Helium to a Tenax trap for 20 minutes, after which the trap was thermally desorbed to a gas chromatograph for analysis. To check for completeness, and to demonstrate that the MMA is not being produced by degradation of the PMMA, the same sample was processed again, producing the run shown in Figure 2. The GC was equipped with a 0.53 mm column, interfaced without split, so the entire sample was analyzed, for maximum sensitivity. Triplicate runs produced MMA peak areas which had an average difference of less than 6%.

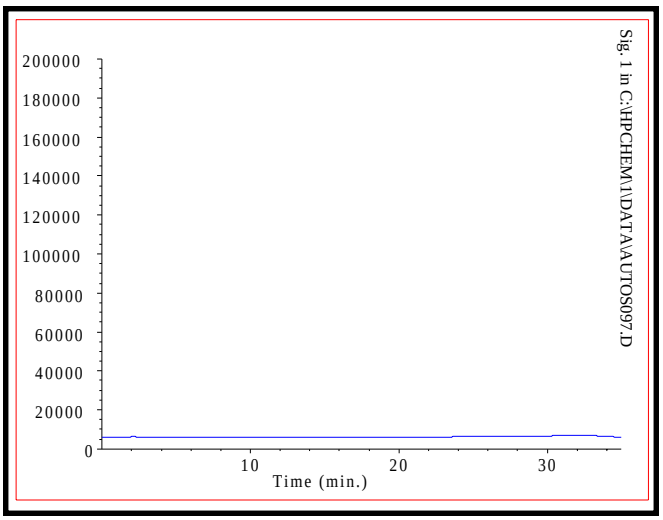

Figure 2. Second run of same sample material

Equipment

The samples were analyzed using a CDS Model 6500 Dynamic Headspace/Thermal Desorption Autosampler interfaced to a Hewlett-Packard 5890 gas chromatograph with a flame ionization detector.

Model 6500 Conditions

Valve oven:	300 °C
Transfer line:	300 °C
Temperature:	100 °C
Time:	20 min
Purge:	50 ml/min Helium
Trap:	Tenax
Trap desorb:	300 °C for 4 min

Gas Chromatograph Conditions

Carrier:	He
Column:	SE-54
	30 m x 0.53 mm
Detector	FID
Oven Initial temperature:	40 °C for 2 min
Ramp:	10 °C/min
Final temperature:	290 °C for 10 min

For more information concerning this application, we recommending the following reading:

T. P. Wampler, Thermal Desorption for GC Sample Preparation, LC GC, 16 (3) 812 (1998).

T. P. Wampler, Analysis of Food Volatiles using Headspace-GC Techniques, in R. Marsili (Ed.) *Techniques for Analyzing Food Aroma,* Marcel Dekker, New York, 1997.

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.

CDS Analytical, Inc. has been a leader in the design and manufacture of laboratory instruments for sample preparation and analysis since 1969. We are dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of pyrolysis, CDS manufactures the Pyroprobe® 1000 and 2000 for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of dynamic headspace instruments for the analysis of volatile organic compounds in environmental, pharmaceutical and food applications, as well as custom systems for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet your needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, or call us at 800-541-6593.

frm.c.65.1198

CDS Analytical, Inc.

465 Limestone Road, P.O. Box 277, Oxford, PA USA 19363-0277
610-932-3636 - Fax: 610-932-4158
e-mail: cds@dpnet.net - www.cdsanalytical.com