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Application Data Sheet

No.135

GC-MS
Gas Chromatograph Mass Spectrometer

Screening Analysis of Phthalate Esters and Qualitative Analysis of Other Additives in Medical Supplies Using the Py-GC/MS Screening System

Although phthalate esters are used as plasticizing agents in plastics, there are concerns over their reproductive toxicity. This has led to regulatory restrictions on their use, including a Restriction of Hazardous Substances (RoHS) Directive that added phthalate esters to the list of restricted substances. The phthalate ester DEHP is used widely in blood transfusion bags, blood transfusion tubing, and other medical supplies, and the elution and transfer of DEHP into the contents of medical supplies is viewed as a problem. The use of medical supplies that are free of phthalate esters is recommended in notifications issued by various countries (Japan's Ministry of Health, Labour and Welfare in 2002, USA's FDA in 2002, Europe's EFSA in 2005, and Germany's BfR in 2013).
For some time, Soxhlet extraction-GC/MS and soaking extraction-GC/MS have been used to analyze phthalate esters in medical supplies, but these methods require a series of operations that can take several hours. Py-GC/MS is a new method of screening for phthalate esters that is adopted as part of International Standard IEC 62321-8 in 2017. The Py-GC/MS screening method requires no organic solvents and allows for rapid sample preparation. Shimadzu offers this analysis method in the form of the Py-Screener system.
This Application Data Sheet uses Py-Screener to analyze various medical supplies. A simultaneous Scan/SIM analysis method was paired with Py-Screener's high-speed scanning mode to perform a highly sensitive screening analysis for phthalate esters using SIM and, simultaneously, perform a qualitative analysis of other additives based on Scan information.

Analytical Conditions

Py-GC/MS was performed using the analytical conditions included with Py-Screener. Refer to GC/MS Application Data Sheet No. 110, "Analysis of Phthalate Ester Using the Py-Screener (1)“, for details on these analytical conditions.
Seven different medical supplies, including blood transfusion bags and blood transfusion tubing, were used as samples and data was gathered using simultaneous Scan/SIM analysis. SIM can perform highly sensitive analysis and was used to screen for phthalate esters. Simultaneously, other additives were identified by obtaining total ion current chromatograms (TICs) with Scan, then performing a similarity search for mass spectral data from unknown peaks with the NIST Mass Spectral Library (2014).
Calibration curves were prepared using a 1000 mg/kg standard sample of phthalate esters (P/N: 225-31003-91, Shimadzu) and used to quantify the phthalate esters content of medical supplies.

Analytical Results

SIM chromatograms of phthalate esters and TICs of Scan data obtained from analysis of each medical supply are shown in Figs. 1, 2 and 3. Results for each sample are shown in Table 1. A high concentration of DEHP (approx. 8%) was detected in the blood transfusion bag, blood transfusion tubing, and intravenous tubing 1. Additives detected other than phthalate esters were the phthalic acid alternative bis(2-ethylhexyl) adipate, the plasticizing agent tri(2-ethylhexyl) trimellitate (TOTM), the antioxidant agents tris(2,4-di-tert-butylphenyl) phosphate, and the lubricants palmitic acid, palmitic acid methyl ester, and stearic acid butyl ester.

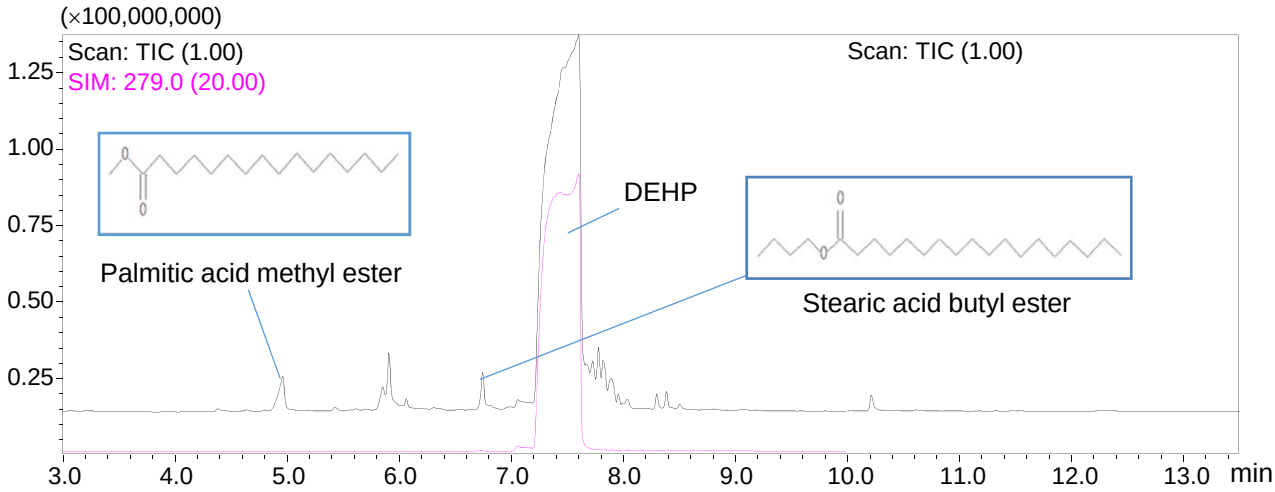

Fig. 1: SIM Chromatogram of Phthalate Ester and TIC of Scan Data Obtained by Analysis of a Blood Transfusion Bag

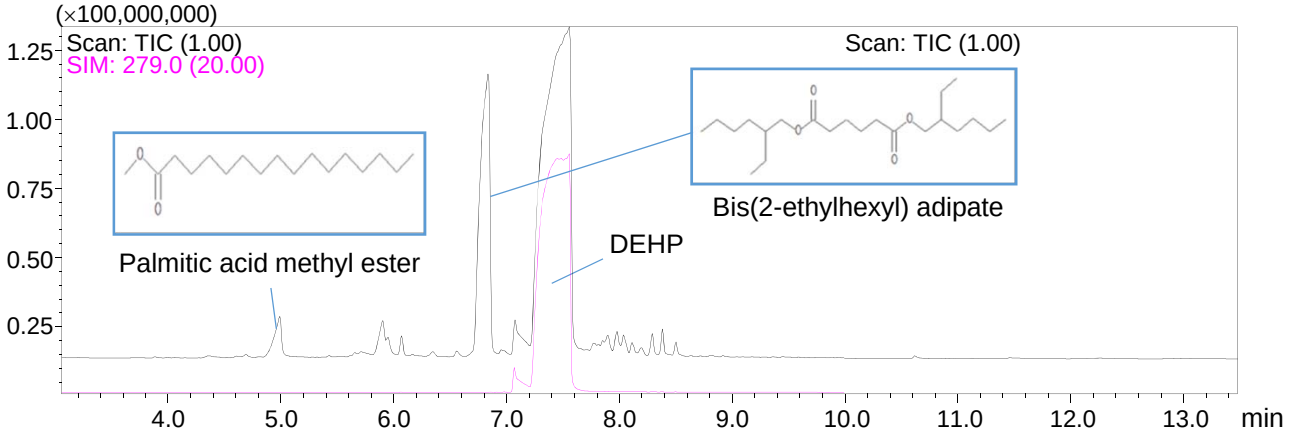

Fig. 2: SIM Chromatogram of Phthalate Ester and TIC of Scan Data Obtained by Analysis of Blood Transfusion Tubing

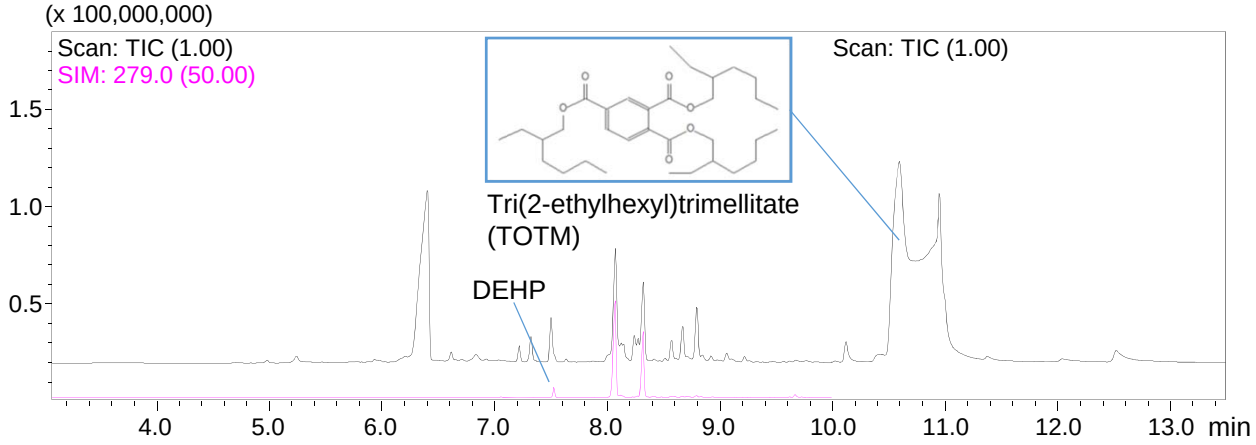

Fig. 3: SIM Chromatogram of Phthalate Ester and TIC of Scan Data Obtained by Analysis of Intravenous Tubing 2

Table 1: Phthalate Esters Screening Results and Qualitative Analysis Results for Other Additives by Medical Supply

Sample	Blood Transfusion Bag		Blood Transfusion Tubing		Intravenous Bag 1		Intravenous Bag 2		Intravenous Tubing 1		Intravenous Tubing 2		Syringe	
Compound	Concentration (mg/kg)	%RSD	Concentration (mg/kg)	%RSD	Concentration (mg/kg)	%RSD	Concentration (mg/kg)	%RSD	Concentration (mg/kg)	%RSD	Concentration (mg/kg)	%RSD	Concentration (mg/kg)	%RSD
DIBP	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-
DBP	N.D.	-	152	11.2	N.D.	-	144	7.1	N.D.	-	N.D.	-	N.D.	-
BBP	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-
DEHP	87,737	6.2	75,537	5.1	N.D.	-	107	9.3	81,199	3.4	206	2.4	N.D.	-
DNOP	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-
DINP	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-
DIDP	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-	N.D.	-

Mean concentration and %RSD calculated from results of n = 3 consecutive analyses. N.D.: <30 mg/kg

Other Additives	Palmitic acid (lubricant)	Bis(2-ethylhexyl) adipate (plasticizer)	Tris(2,4-di-tert-butylphenyl) phosphate (antioxidant)	Palmitic acid (lubricant)	Palmitic acid (lubricant)	Tri(2-ethylhexyl) trimellitate (TOTM) (plasticizer)	Tris(2,4-di-tert-butylphenyl) phosphate (antioxidant)
	Stearic acid butyl ester (lubricant)	Palmitic acid methyl ester (lubricant)					

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