SHIMADZU
Excellence in Science

High performance Liquid Chromatography

Application News

Analysis of Cyclic Oligomers in Recycled PET Using Integrated LC System

Miki Mukai, Miho Akagi

User Benefits

- Evaluation of recycled PET can be performed following "Guidelines for Ensuring the Reliability of Indications of Specified Procured Goods, etc." from the Ministry of the Environment (Japan).
- Analytical throughput can be improved by high speed analysis using Integrated LC System "i-Series LC-2070" and Shim-pack Scepter™ C18-120 column.

■ Introduction

In recent years, the utilization of renewable resources has increased substantially. Among these, recycled polyethylene terephthalate (PET), primarily sourced from PET bottles, is recognized for its relatively low environmental impact and is extensively employed in the manufacture of clothing, miscellaneous goods, and various other products. However, the reliability of environmentally conscious products must be assured. To address this, the Ministry of the Environment in Japan has established the "Guidelines for Ensuring the Reliability of Indications of Specified Procured Goods, etc." [1] (hereafter referred to as The Guidelines). For the purpose of distinguishing between recycled and virgin PET, the quantification of cyclic oligomers serves as a key indicator. Due to inherent differences in the polymerization processes, recycled PET typically exhibits a lower concentration of cyclic oligomers compared to virgin PET. The Guidelines prescribe a method for this evaluation, which involves quantifying the cyclic oligomer content using high-performance liquid chromatography (HPLC).

LC-2070 i-series High Performance Liquid Chromatograph (Fig. 1) is a compactly designed integrated LC System, providing easy maintenance and applicability for general-purpose HPLC analysis. Additionally, Shim-pack Scepter C18-120 columns are packed with a fully porous organosilica hybrid particles, providing excellent durability and performance under a wide range of conditions.

This article employs Integrated LC System "i-Series LC-2070" and Shim-pack Scepter C18-120 column to quantify cyclic oligomers in recycled PET following The Guidelines and to do ultrahigh speed analysis for the same purpose.

Fig. 1 Integrated LC System i-Series (LC-2070)

■ Analysis of Cyclic Oligomers

According to the established Guidelines, recycled PET was analyzed using HPLC. The pretreatment protocol is illustrated in Fig. 2. A potential issue is that certain strongly hydrophobic cyclic oligomers may not be eluted and could accumulate within the chromatographic column, thereby extending the required analysis time. The application of gradient elution, which involves increasing the proportion of organic solvent to 100%, enables a reduction in the total analysis time, (Fig. 3 and Table 1).

Sample 10 mg
↓ Dissolve in 1 mL of HFIP
Dropwise to approx. 7.5 mL of acetonitrile while stirring
↓
Dilute to 10 mL with acetonitrile in a volumetric flask
↓
Filtration (0.22 µm)

Fig. 2 Pretreatment procedures of recycled PET

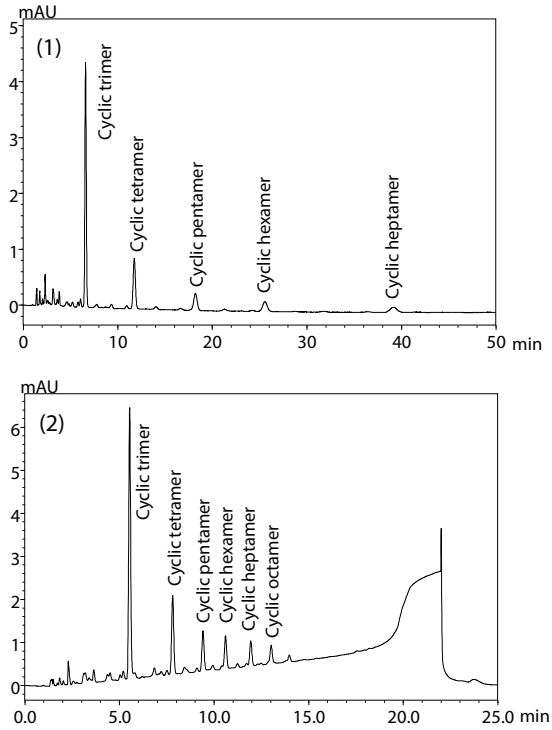


Fig. 2 Chromatograms of recycled PET: (1) isocratic elution, (2) gradient elution

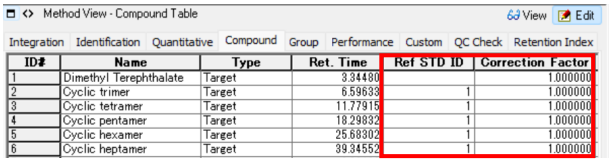
Table 1 Analytical conditions

System	:	i-Series LC-2070
Column	:	Shim-pack Scepter C18-120*1 (250 mm × 4.6 mm I.D., 5 µm)
Flow rate	:	1.5 mL/min
Mobile phase (Isocratic elution)	:	Water/Acetonitrile=30:70 (v/v)
Mobile phase (Gradient elution)	:	A) Water, B) Acetonitrile B.Conc 70% (0 min) → 100% (17.5-20 min) → 70% (20.01-25 min)
Injection vol.	:	20 µL
Vial	:	Shim-vial™ H glass*2
Detection	:	242 nm

*1 P/N : 227-31020-06
*2 P/N : 227-34500-01

■ Quantitative Analysis Using Dimethyl Terephthalate

In The Guidelines, dimethyl terephthalate is used as the standard for all the cyclic oligomers because standard compounds of respective cyclic oligomers are difficult to obtain. This section describes how to configure the LabSolutions™ software when quantifying cyclic oligomers using a calibration curve of dimethyl terephthalate. At first, create a calibration curve using the concentrations and peak areas of dimethyl terephthalate standard solutions. Next, in the compound table, enter the ID of dimethyl terephthalate in the "Ref STD ID" field for each cyclic oligomer. Enter the "Correction Factor" as 1 assuming that detector responses of dimethyl terephthalate and respective cyclic oligomers are identical (Fig. 4). These procedures enable convenient quantitative calculations in LabSolutions using the calibration curve of another compound. The content of cyclic oligomers in recycled PET was calculated based on the quantitative results for cyclic oligomers obtained from the dimethyl terephthalate calibration curve (Formula 1, Table 2)



ID#	Name	Type	Ret. Time	Ref STD ID	Correction Factor
1	Dimethyl Terephthalate	Target	3.34480		1.000000
2	Cyclic trimer	Target	6.59633	1	1.000000
3	Cyclic tetramer	Target	11.77915	1	1.000000
4	Cyclic pentamer	Target	18.29832	1	1.000000
5	Cyclic hexamer	Target	25.68302	1	1.000000
6	Cyclic heptamer	Target	39.34552	1	1.000000

Fig. 4 Compound table in LabSolutions

【Calculation for content of cyclic oligomer contained in recycled PET】

$$\text{Content of Cyclic Oligomer[wt\%]} = \frac{C \times V \times 10^{-3}}{m} \times 100 \quad \text{…(Formula 1)}$$

C : Concentration of cyclic oligomer determined by HPLC[mg/L]
V : Specified volume[mL]
m : Measured weight of recycled PET sample[mg]

Table 2 Calculated content of cyclic oligomer in recycled PET

	Concentration[wt%]
Cyclic trimer	5.2×10^{-2}
Cyclic tetramer	1.7×10^{-2}
Cyclic pentamer	8.3×10^{-3}
Cyclic hexamer	5.9×10^{-3}
Cyclic heptamer	4.4×10^{-3}

■ Ultrahigh Speed Analysis

Scepter C18-120 columns of the Shim-pack Scepter series offers a wide lineup from HPLC to UHPLC and prep LC, providing seamless method transfer among analytical conditions of different column dimensions. Fig. 5 shows the chromatogram obtained using a column from this series with a length of 100 mm, an inner diameter of 3.0 mm and a particle size of 1.9 µm. Table 3 shows the analytical conditions for that column. Analysis time was reduced to approximately one-third, solvent consumption to approximately one-eighth and sample volume to one-fourth. LC-2070 with pressure tolerance of 50 MPa accommodates conventional to ultrahigh speed analysis. Strongly retained long-chain cyclic oligomers, including cyclic octamers, can be eluted within 7 min.

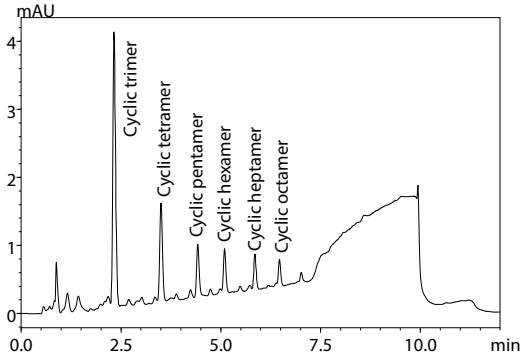


Fig. 5 Chromatogram of recycled PET (UHPLC analysis)

Table 3 UHPLC conditions

System	:	i Series LC-2070
Column	:	Shim-pack Scepter C18-120*1 (100 mm × 3.0 mm I.D., 1.9 µm)
Flow rate	:	0.8 mL/min
Mobile phase	:	A) Water, B) Acetonitrile B.Conc 70% (0 min) → 90% (6 min) → 100% (6.01-8.5 min) → 70% (8.51-12 min)
Column temp.	:	40 ℃
Injection vol.	:	5 µL
Vial	:	Shim-vial H glass
Detection	:	242 nm

*1 P/N : 227-31013-03

■ Conclusion

Using Integrated LC System "i-Series LC-2070" and Shim-pack Scepter C18-120 columns, Cyclic oligomer analysis of recycled PET was performed following The Guidelines from Ministry of the environment (Japan). The method for quantitative determination using calibration curves for other than target compounds on LabSolutions was introduced. Additionally, the use of UHPLC column provided reductions of mobile phase consumption to approximately one-eighth and sample volume to one- fourth.

<References>

1) Ministry of the Environment (Japan), Guidelines for Ensuring the Reliability of Indications of Specified Procured Goods, etc., Appendix 3, "4.8. Explanation of Survey Methods for Products Containing Recycled Plastic (by Resin Type)" (as of December 2025)
https://www.env.go.jp/policy/hozen/green/g-law/net/trust.html

01-01102-EN First Edition: Dec. 2025

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

Please fill out the survey

Related Products Some products may be updated to newer models.

i-Series
High Performance Liquid Chromatograph

Shim-pack Scepter LC Columns
HPLC Column

Related Solutions

- Chemicals
- Plastics
- Environment
- Waste-Others

- Price Inquiry
- Product Inquiry
- Technical Service / Support Inquiry
- Other Inquiry