

## msFineAnalysis iQ Ver.2 Target Analysis Example II Rapid Analysis of Polymer additives

Product used : Mass Spectrometer (MS)

### 1. Introduction

**Target Analysis** is a method for fast and accurate search and evaluation of only the target compounds based on integrated qualitative analysis<sup>1)</sup> with Electron Ionization (EI) and Soft Ionization (SI) data. In this method, it is necessary to register the CAS RN<sup>®</sup>, molecular formula (or molecular weight), fragment composition formula of target compounds in the "Target List" in advance. Based on the registered information, each extracted ion chromatogram (EIC) is plotted for EI and SI measurement data, and peaks are detected. The molecular ion peak in the mass spectrum, its isotopic pattern, and the similarity and retention index with the NIST database are confirmed on the detected peak. Based on these results, it is determined whether it is the target compound or not. This time, we report the results of analyzing optional polymer additives from the pyrolysis measurement data of polyvinyl chloride (PVC) using this method.

### 2. Experiment

A commercial PVC product weighing 1.0 mg was used as the sample. The measurement was performed using a GC-MS (JMS-Q1600GC, JEOL Ltd.) equipped with a multi-shot pyrolyzer (EGA/PY- 3030D, Frontier Lab Ltd.) as a pretreatment device. EI and Photoionization (PI) as SI were used as ionization methods. The obtained data were analyzed using the "Target Analysis" function in the integrated qualitative analysis software called "msFineAnalysis iQ". The measurement conditions are shown in Table 1.



EGA/PY-3030D-  
JMS-Q1600GC UltraQuad™ SQ-Zeta



Ion source  
(Share use of EI/PI)

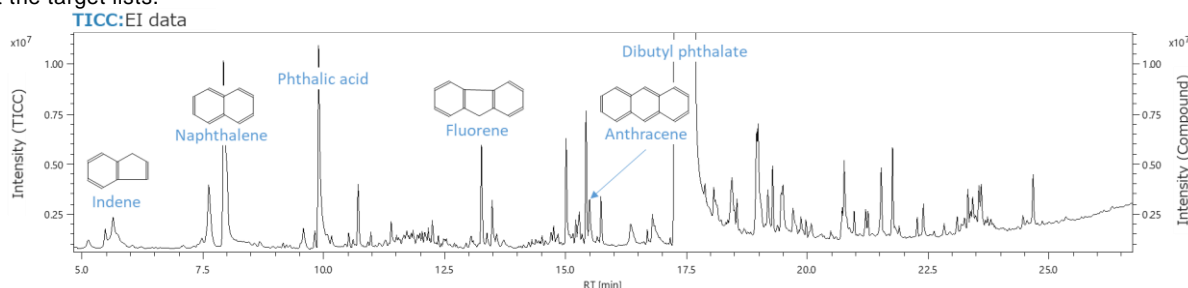
**Table 1 Measurement Conditions**

PY	Furnace temp.	50 °C-40 °C/min-400 °C
	Interface temp.	320 °C
GC	Column	ZB-5MSi (Phenomenex), 30 m length× 0.25 mm id, 0.25 µm film thickness
	Column flow	1.0 mL/min (He)
	Oven temp.	40 °C(0 min)-10 °C/min-320 °C(10 min)
	Inlet temp.	320 °C
	Injection mode	split (1/50)
MS	Interface temp.	250 °C
	Ion source temp.	250 °C
	Ionization	EI (70 eV, 50 µA) PI (approx.10 eV, Fil.Off)
	Scan range	m/z 29-700

### 3. Results

#### 3.1 TICC

The GC/EI TICC by pyrolyzer GC-MS is shown in Fig.1. Major pyrolysis products such as Naphthalene, Anthracene, Fluorene and Indene were detected. From the target analysis, only polymer additive components were identified from the measurement results . A preset list of compounds commonly used as polymer additives (Table 2) was used as the target list. In addition, it's possible to create and edit the target lists.



**Fig.1 GC/EI TIC chromatogram**

**Table 2 Target list of Polymer Additives**

No.	Name	CAS#	NIST Library	EIC Creation	Molecular Formula	Fragment Formula	m/z (EIC)	RT [min]	RI [i.u] Non-Polar	RI [i.u] Semi Non-Polar	RI [i.u] Polar	Description	Spectrum
<input type="checkbox"/> 001	2,6-di-tert-butylphenol	128-39-2	✓	Molecular Ion	C14H22O		206			1442	2327	Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 002	2,6-di-tert-butyl-4-methylphenol	128-37-0	✓	Molecular Ion	C15H24O		220		1494	1513	1906	Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 003	2,6-di-tert-butyl-4-ethylphenol	4130-42-1	✓	Molecular Ion	C16H26O		234			1754		Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 004	2,4,6-tri-tert-butylphenol	732-26-3	✓	Molecular Ion	C18H30O		262			1627		Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 005	4-butan-2-yl-2,6-di-tert-butylphenol	17540-75-9	✓	Molecular Ion	C18H30O		262			1660		Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 006	2,6-di-tert-butyl-4-(hydroxymethyl)phenol	88-26-6	✓	Molecular Ion	C15H24O2		236			1774		Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 007	octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propanoate	2082-79-3	✓	Molecular Ion	C35H62O3		530			3603		Antioxidant	<a href="#">View</a>
<input type="checkbox"/> 008	2,6-di-tert-butyl-4-[(dimethylamino)methyl]phenol	88-27-7	✓	Molecular Ion	C17H29NO		263					Antioxidant	<a href="#">View</a>

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## 3.2 Result of target analysis

Fig.2 shows the compounds from the target list of additives that were used to evaluate these data. As a result, 6 types of polymer additives such as antioxidants, stabilizers, lubricants and plasticizers were confirmed in PVC products.

Background color : white...no peak detected, yellow...peak detected, blue... peak detected and clear target judgment

Target Information								
ID	Name	CAS#	Molecular Formula	m/z (EIC)	RI [iu]	Description	Num. of Detected	Num. of Passed
T002	2,6-di-tert-butyl-4-methylphenol	128-37-0	C <sub>15</sub> H <sub>24</sub> O	220	1513	Antioxidant	11	1
T016	2-tert-butyl-4,6-dimethylphenol	1879-09-0	C <sub>12</sub> H <sub>18</sub> O	178	-	Antioxidant	9	1
T022	2-tert-butyl-4-[1-(5-tert-butyl-4-hydroxy-2-methylphenyl)butyl]-5-methylphenol	85-60-9	C <sub>26</sub> H <sub>38</sub> O <sub>2</sub>	382	2673	Antioxidant	1	1
T075	1,3-diphenylpropane-1,3-dione	120-46-7	C <sub>15</sub> H <sub>12</sub> O <sub>2</sub>	224	2090	Stabilizer	2	1
T079	octadecanoic acid	57-11-4	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>	284	2172	Glidant	1	1
T081	butyl octadecanoate	123-95-5	C <sub>22</sub> H <sub>44</sub> O <sub>2</sub>	340	2388	Glidant/Plasticizer	2	1

Fig.2 Target information

## 3.3 Details of target analysis results for [ID:T022]

As an example, details of the target analysis results for compound ID:T022 are described. The EIC (m/z 382) specified in T022 is plotted from the EI and PI measurement data. A peak at RT23.40 minutes in the PI data plotted by EIC(m/z 382) is shown in Fig.3. Although it was difficult to confirm this peak by "Non-Target Analysis" because the intensity ratio was less than 1%, it could be quickly analyzed by "Target Analysis". Figure 4 shows the target judgment result for the RT23.40 minute peak in Fig.3. The result shows that the spectral similarity is 813 (Max:999), the measurement error of RI ( $\Delta$ RI) is 35 [iu]. In addition, the molecular ion peak is confirmed on the PI mass spectrum, the isotope ratio match score is 0.91 (Max:1.00), thus each evaluation point showed good results, so the peak at RT 23.40min was judged as 2-tert-Butyl-4-[1-(5-tert-butyl-4-hydroxy-2-methylphenyl)butyl]-5-methylphenol.

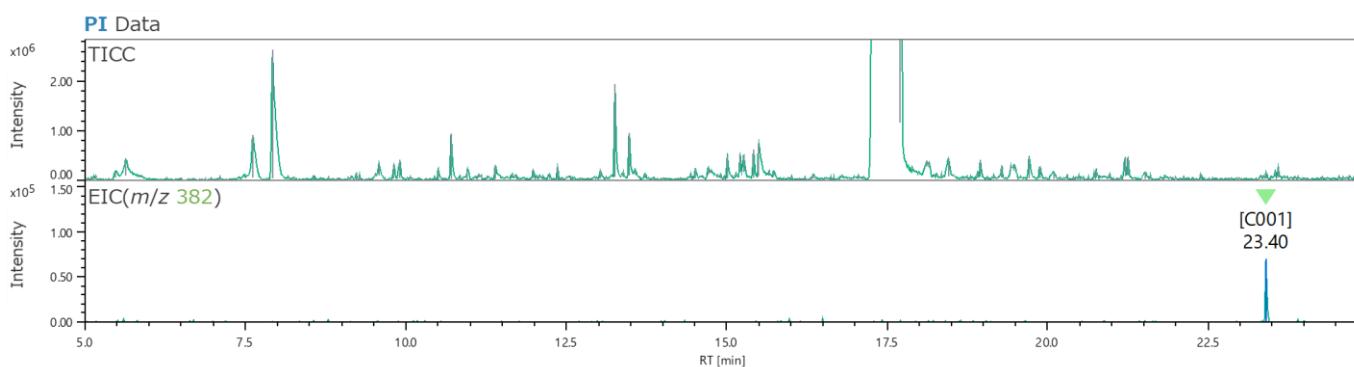


Fig.3 GC/PI Chromatograms [Upper: TIC, Lower: EIC(m/z 382)]

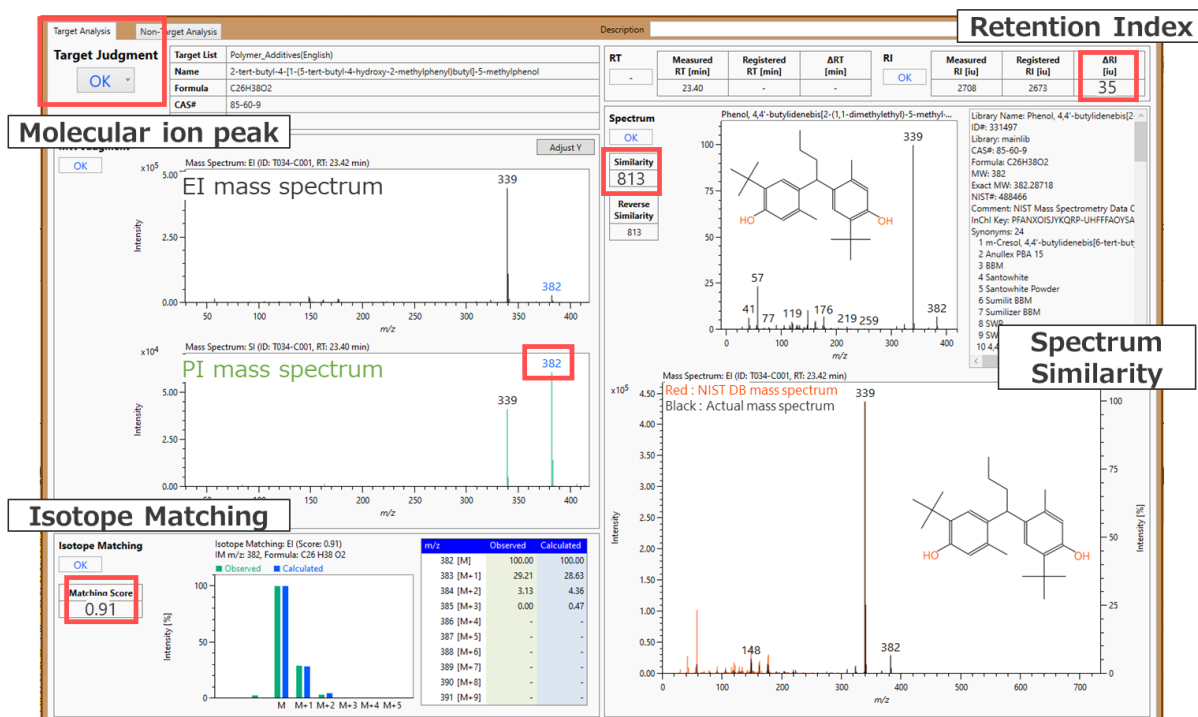


Fig.4 Result window of Target Analysis

## Conclusion

We analyzed the optional polymer additives in PVC product using Target Analysis. As a result, 6 types of polymer additives were confirmed and it was shown that it is possible to analyze the target compounds quickly and accurately by "Target Analysis".

1) M. Ubukata et al, Rapid Commun Mass Spectrom., 34 (2020). DOI: 10.1002/rcm.8820

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