

Tin in Resin Analysis Using EDX - Sn Screening Analysis Kit -

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User Benefits

- ◆ The screening analysis package provides everything necessary for analysis including control samples and software.
- ◆ EDX can simultaneously screen for tin (Sn), RoHS elements, antimony (Sb), and chlorine (Cl).
- ◆ By using the PCEDX-Navi software, even novices can operate from judgment to report output.

Introduction

Organotin compounds have been used in a wide range of applications, including stabilizers and catalysts for resin products, ship bottom paints, antifouling agents, and preservatives. On the other hand, the toxicity of some organotin compounds has led to restrictions on their use around the world, such as the European Union's REACH regulations.

Table 1 lists the organotin compounds specified as restrictions in the REACH regulations. The manufacture, import and use of articles containing these substances are restricted. In addition, six more organotin compounds and their reaction products have been designated as candidate substances for approval, which are required to be communicated to the European Chemicals Agency (ECHA) and business partners.

Energy dispersive X-ray fluorescence spectrometry (EDX) can not perform qualitative or quantitative analysis of organotin compounds, but it can analyze the concentration of tin (Sn) as an element. Therefore, EDX enables screening analysis aimed at controlling total Sn concentrations in regulated organotin compounds.

This application news introduces the flow of screening analysis of Sn using PCEDX-Navi software and examples of screening analysis of Sn in resins.

Table 1 Organotin Compounds Specified in the REACH Regulations.¹

Substance	Abbreviations	Control Value
Trisubstituted organotin compounds	—	1000 ppm as Sn element
Dibutyltin compound	DBT	
Dioctyltin compound	DOT	

Sn Screening Analysis Kit

Shimadzu has long offered screening analysis kits that support the analysis of RoHS 5 elements (Cd, Pb, Cr, Hg, Br), chlorine (Cl) and antimony (Sb). By using these EDX analysis kits, even novices can screen and analyze for harmful elements.

As an extension, the EDX-7200 adds a new Sn screening analysis kit to support screening analysis of organotin compounds in resins. EDX can, in one sample, simultaneously measure Sn, RoHS elements, Cl, and Sb.

Screening Setting Display

Fig. 1 shows the threshold settings for the screening analysis kit. From this display, you can set the threshold value for each material and element.

The screening analysis software is also equipped with a time reduction function that automatically shifts to the next measurement condition when it is determined that a managed element clearly exceeds, falls below, or enters a gray zone. The time reduction function therefore significantly increases sample throughput.

Threshold		Judgment Display		Report Template			
Settings of Threshold(ppm)							
		Material					
		Plastic	Al	Fe	Cu	Sn	
Element	Cd	<input checked="" type="checkbox"/> 70-130	<input checked="" type="checkbox"/> 70-130	<input checked="" type="checkbox"/> 70-130	<input checked="" type="checkbox"/> 70-130	<input checked="" type="checkbox"/> 70-130	
	Pb	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	
	Cr	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	
	Hg	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	<input checked="" type="checkbox"/> 700-1300	
	Br	<input checked="" type="checkbox"/> 300	<input checked="" type="checkbox"/> 300	<input checked="" type="checkbox"/> 300	<input checked="" type="checkbox"/> 300	<input checked="" type="checkbox"/> 300	
	Cl	<input type="checkbox"/> 700-1300					
	Sn	<input checked="" type="checkbox"/> 700-1300					
	P	<input type="checkbox"/> 700-1300					
	Meas. Condition		Precise filter				<input type="checkbox"/> Time Reduction
	Estimated Time		6 minutes	6 minutes	6 minutes	6 minutes	6 minutes

Fig. 1 Setting Display (Example of Threshold Setting)

Management Sample

For equipment management, the Sn containing polyethylene control sample is included in the analysis kit. Fig. 2 shows the appearance of the control sample.



Fig. 2 Check Sample of Sn Screening Analysis Kit

■ Sample

Moldings made of Sn containing polyvinyl chloride (PVC) resin were analyzed. Fig. 3 shows a photograph of the of the samples. When measuring, four molded articles were measured side by side.



Fig. 3 Sample appearance

■ Analysis Results

The samples were analyzed using the conventional screening analysis condition for RoHS and Sn elements. The judgment results are shown in Fig. 4. The quantitative values and the judgement with "OK / ?? / ND" is displayed. The profile of Sn is also shown in Fig. 5.

Sn was measured in replicate 10 times. The results are shown in Table 2.

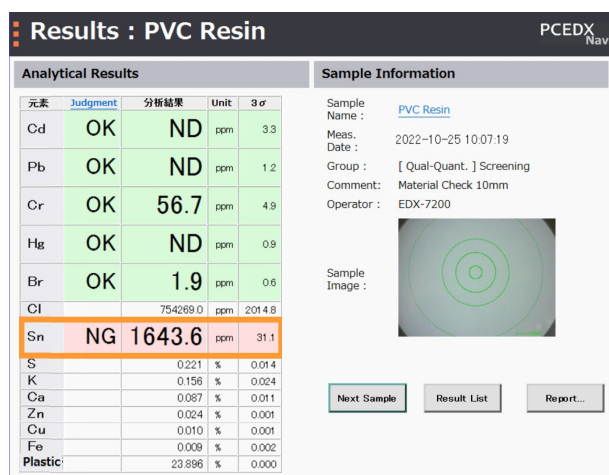


Fig. 4 Screening Analysis and Judgment Results Screen

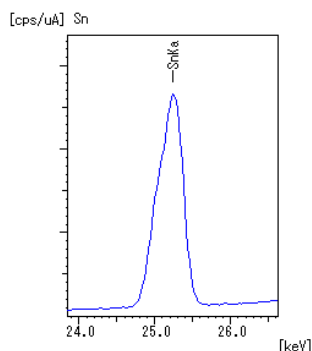


Fig. 5 Sn Kα Profile

Table 2 Replicate Results

Mean [ppm]	1641.7
Standard deviation [ppm]	20.3
CV value [%]	1.2

■ Limit of Detection

The limit of detection for Sn in PE and PVC resins under screening analysis conditions (air atmosphere, 100 seconds) are shown in Table 3.

Table 3 The Limit of Detection for Sn in Resin Materials

Material	PE resin	PVC resin
Lower limit of detection [ppm]	3.4	5.7

■ Conclusion

The EDX-7200 Sn screening analysis kit features:

- Everything necessary for analysis, control samples, and software.
- Screen tin (Sn) simultaneously with RoHS 5 elements, Cl and Sb.
- Intuitive and easy to use PCEDX-Navi software allowing even those unfamiliar with analysis to easily operate from measurement to judgement to report.

*See "Application News No. 01-00300-EN RoHS Screening Analysis by EDX" for the flow of analysis using the screening analysis kit.

*If Sn is detected, use other analytical equipment such as GC/MS to check whether it is a regulated compound.

■ Measurement conditions

Table 4 Quantitative Analysis Conditions

Equipment	: EDX-7200
Element	: ^{50}Sn
Analysis group	: Screening
Detector	: SDD
X-ray tube ball	: Rh target
Tube voltage	: 50 [kV]
Tube current	: Auto [μA]
Collimator	: 10 [mm ϕ]
Primary filter	: #1
Atmosphere	: Air
Integration time	: 100 sec
Deadtime	: Up to 30 (%)

<References>

- 1) List of ECHA REACH Regulation Restricted Substances (Annex XVII) URL: <https://echa.europa.eu/cs/substances-restricted-under-reach> (see 2023 - 3 -2)