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Application News

Shimadzu Energy Dispersive X-Ray Fluorescence Spectrometer EDX-7200

RoHS Screening Analysis by EDX

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User Benefits

- The EDX-7200 achieves up to 30 times higher count values than a previous model EDX-720. The performance improvements contribute to shorter analysis times.
- Equipped with internal calibration curves for a wide variety of materials, the software can automatically select the optimal calibration curve for the given sample.
- Using the PCEDX-Navi software makes it easy to perform all operations, from measuring samples to generating reports, even for operators without analytical experience.

■ Introduction

Shimadzu's previous model EDX-720 required the preparation of calibration curves using standard samples for RoHS screening analysis. Therefore, analysis and equipment management required long time and effort.

However, the new EDX-7200 includes PCEDX-Navi software that makes RoHS screening analysis easy even for users without analytical experience. Higher sensitivity (up to 30 times higher counts) and an improved analytical algorithm achieve significantly shorter measurement times (up to 1/30 shorter). For example, a previous model required 400 seconds to measure plastic materials, but the EDX-7200 can shorten that to 51 seconds (refer to Table 1).

This Application News describes the following.

1. Operation flow for RoHS screening analysis
2. Automatic time-reduction function
3. Lower limit of detection and repeatability

■ Operation Flow for RoHS Screening Analysis

With the PCEDX-Navi software, even users without analytical experience can easily perform all operations from measuring samples to creating reports. System features include the following.

(1) All operations from instrument startup to analysis and report creation can be performed from one window.

(2) Calibration curves no longer need to be created using standard samples.

(3) The system automatically selects the optimal calibration curve from those included in the system for the materials listed below.

Plastics (PE and PVC), aluminum alloys, steels, copper alloys, and tin alloys

Figs. 1 to 4 show the overall operation flow for using the RoHS, halogen, and antimony screening kit to simultaneously screen for the five RoHS elements, chlorine, and antimony.

[Measurement Preparation] Window

(1) Place the sample in the sample compartment while viewing an image of the sample.
(2) Select [Screening] or [Screening Plastic] in the [Condition] field.
(3) Enter the sample name in the [Sample Name] field.
(4) Click the [START] button.

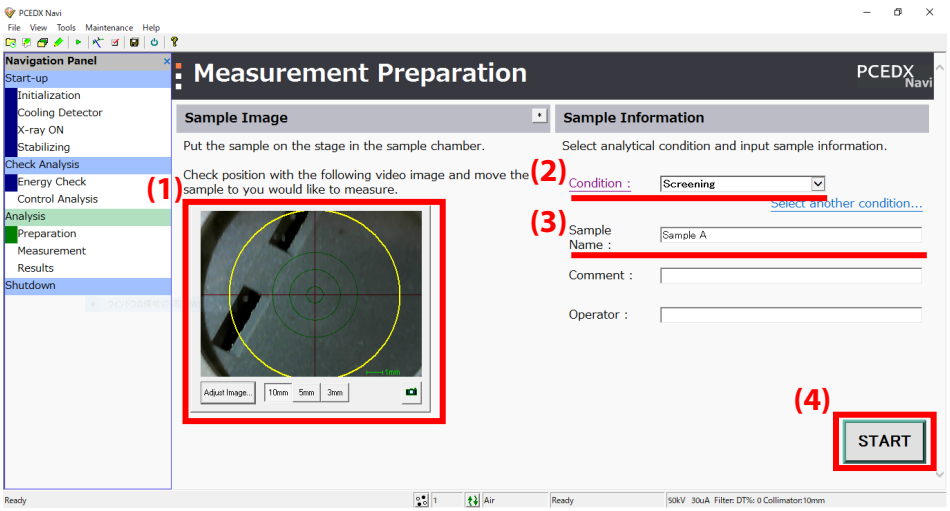

Fig. 1 [Measurement Preparation] Window

[Measuring] Window

First, the sample is preliminarily measured for about 15 seconds to determine the material type. Then the software automatically selects the calibration curve that is best suited for the given sample and starts RoHS screening (Fig. 2).

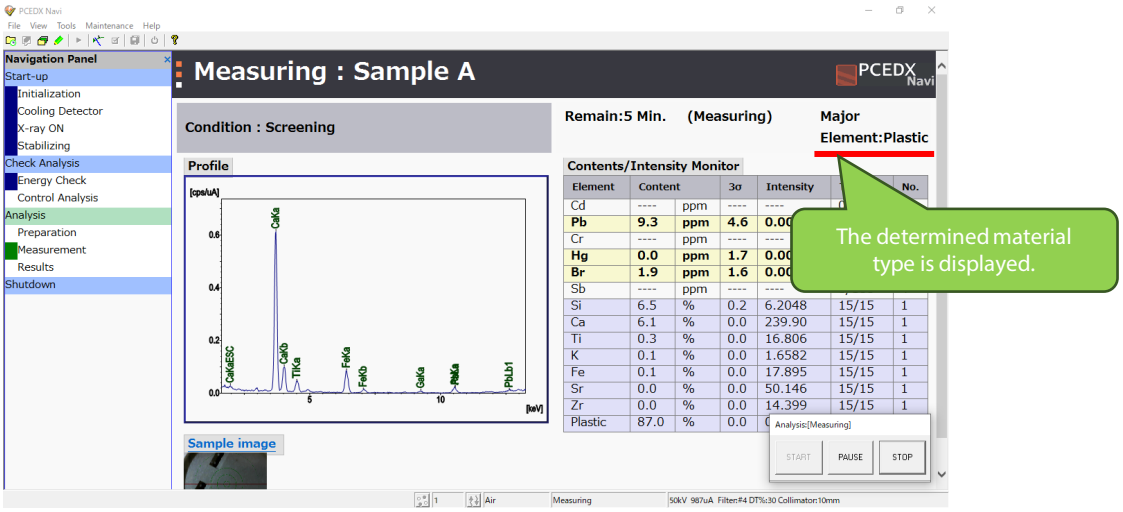

Fig. 2 [Measuring] Window

[Results] Window

In addition to analytical results, pass/fail results with respect to threshold values (“OK,” “??,” or “NG”) are also displayed. A report can be created by simply clicking the [Report] button (Fig. 3).

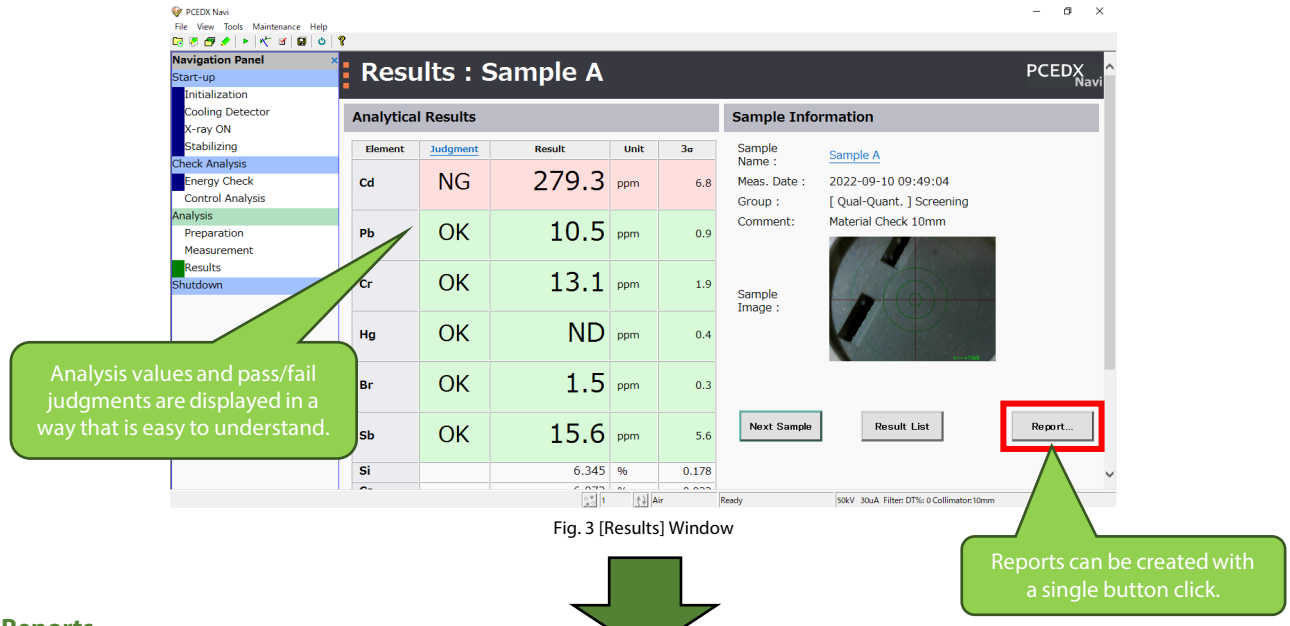

Fig. 3 [Results] Window

Reports

Reports can be created in two file formats, either Excel® or HTML.

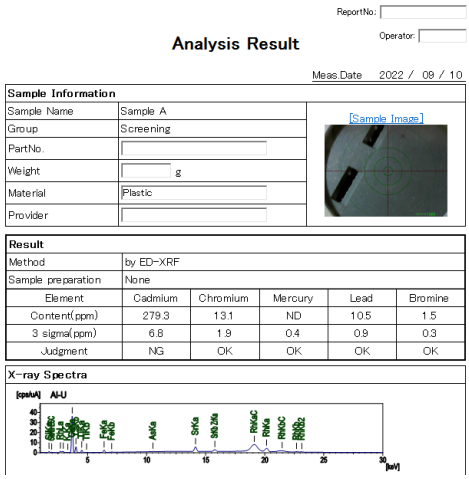

Fig. 4 Report

■ Automatic Time-Reduction Function

As soon as the system determines that the concentration of the element being controlled is clearly higher or lower than the corresponding threshold values, or is within a gray zone, this function automatically skips to the next measurement condition, which helps improve analysis throughput. The window for setting threshold values and enabling the automatic time-reduction function is shown in Fig. 5.

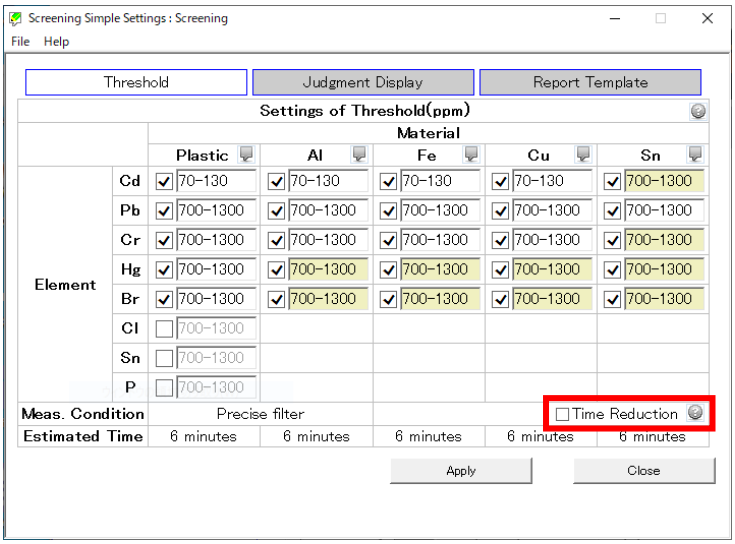

Fig. 5 Window for Setting Threshold Values and Enabling the Automatic Time-Reduction Function

■ Example of Analysis Using the Automatic Time-Reduction Function

This shows the results by analyzing a plastic material and a steel material with the automatic time-reduction function ON and OFF.

Threshold values were set to 70-130 ppm for Cd and 700-1300 ppm for elements other than Cd.

1. Plastic Material

Analysis results are shown in Table 1. It shows that the automatic time-reduction function shortened the measurement time from 400 seconds to 51 seconds.

Fig. 6 Plastic Material

Table 1 Plastic Material Analysis Results

Automatic Time-Reduction	ON				OFF			
Element	Quantitation Value [ppm]	3σ [ppm]	Pass/Fail Result	Analysis Time [sec]	Quantitation Value [ppm]	3σ [ppm]	Pass/Fail Result	Analysis Time [sec]
$_{46}$Cd	283.9	14.4	NG	14	283.3	6.6	NG	100
$_{51}$Sb	34.5	14.7	OK		26.6	5.4	OK	
$_{82}$Pb	9.1	2.8	OK	11	10.2	1.0	OK	100
$_{80}$Hg	N.D.*	1.2	OK		N.D.*	0.4	OK	
$_{35}$Br	1.8	1.0	OK		1.3	0.3	OK	
$_{24}$Cr	13.5	5.6	OK	11	10.5	1.9	OK	100
$_{17}$Cl	94.2	56.8	OK	15	98.2	22.1	OK	100
	Total			51	Total			400

* N.D.: Not Detected

2. Bolt (Steel Material)

Analysis results are shown in Table 2. They show similar time reductions using the automatic time-reduction function.

Fig. 7 Bolt (Steel Material)

Table 2 Bolt (Steel Material) Analysis Results

Automatic Time-Reduction	ON				OFF			
Element	Quantitation Value (ppm)	3σ [ppm]	Pass/Fail Result	Analysis Time [sec]	Quantitation Value [ppm]	3σ [ppm]	Pass/Fail Result	Analysis Time [sec]
$_{46}$Cd	N.D.*	21.7	OK	100	N.D.*	17.3	OK	100
$_{82}$Pb	554.9	102.5	OK	21	551.3	46.9	OK	100
$_{80}$Hg	N.D.*	110.5	OK		N.D.*	49.9	OK	
$_{35}$Br	N.D.*	19.5	OK		N.D.*	9.0	OK	
$_{24}$Cr	3036.9	174.7	NG	12	2981.1	64.1	NG	100
	Total			133	Total			300

* N.D.: Not Detected

■ Lower Limit of Detection

The lower limits of detection for the respective materials are shown in Table 3. For reference, the lower limit of detection values for the EDX-LE Plus model are also shown.

Table 3 Lower Limit of Detection for RoHS Screening Conditions (ppm)

Element	EDX-7200						EDX-LE Plus					
Material / Element	PE	PVC	Al	Fe	Cu	Sn	PE	PVC	Al	Fe	Cu	Sn
$_{46}$Cd	2	2	1	4	5	(89)	2	2	1	5	6	(170)
$_{82}$Pb	0.5	0.8	0.9	8	13	8	1	2	2	14	31	14
$_{24}$Cr	2	4	2	21	18	(16)	3	8	4	43	41	(35)
$_{80}$Hg	0.2	0.8	(1)	(12)	(16)	(12)	0.4	1.4	(2)	(29)	(37)	(26)
$_{35}$Br	0.1	0.2	(1)	(4)	(7)	(4)	0.2	0.4	(1)	(8)	(12)	(8)
$_{17}$Cl	9	—	—	—	—	—	16	—	—	—	—	—
$_{51}$Sb	5	5	—	—	—	—	6	6	—	—	—	—

- Due to the quantitative FP method used, values shown in parentheses are calculated based on the theoretical standard deviation for a sample that does not contain the element.
- Lower limit of detection values are for an analysis time of 100 seconds.

—: Indicates elements not regulated by the RoHS directive.

■ Repeatability

Table 4 shows repeatability test results from measuring C-H-B-F-5-124HJ metal-containing LDPE sample* using screening condition. The results indicate good coefficient of variation values of less than 1.5 %.

*LDPE standard containing Cd, Pb, Cr, Hg, Br, Cl, and Sb: Sumika Chemical Analysis Service, Ltd.

Table 4 Repeatability (ppm)

Element	$_{46}$Cd	$_{82}$Pb	$_{24}$Cr	$_{80}$Hg	$_{35}$Br	$_{17}$Cl	$_{51}$Sb
Standard Value / Number of Repetitions	110	310	310	280	310	890	1100
1	109	309	313	279	311	900	1101
2	108	310	313	279	312	903	1100
3	109	309	313	278	310	901	1107
4	109	314	310	280	313	904	1111
5	110	311	314	279	311	911	1090
6	110	314	315	281	312	891	1102
7	109	309	314	276	309	897	1101
8	108	309	314	278	310	912	1098
9	112	310	313	279	311	881	1110
10	113	310	314	280	312	894	1113
Mean Value	109.7	310.6	313.1	278.8	311.1	899.4	1103.0
Std. Dev.	1.5	1.9	1.4	1.2	1.2	8.7	6.7
Coef. of Variation (%)	1.4	0.6	0.4	0.4	0.4	1.0	0.6

■ Conclusion

The characteristics of RoHS screening analysis using the EDX-7200 are described below.

1. Operability
All operations, from measuring samples to generating reports, are easy to perform, even for users not used to analyzing samples.

2. Lower Limit of Detection and Repeatability
Compared to a previous model, the lower limit of detection is significantly improved due to the higher sensitivity.

3. Analysis Results Window
In addition to analysis results, pass/fail results ("OK," "??," or "NG") with respect to threshold values are also displayed. Threshold values can be specified by the user.

4. Improved Analysis Throughput
Using the automatic time-reduction function is expected to shorten measurement times and improve the efficiency of analysis.

■ Analysis Conditions

Table 5 Analysis Conditions

Instrument:	EDX-7200
Elements:	$_{46}$Cd, $_{82}$Pb, $_{24}$Cr, $_{80}$Hg, $_{35}$Br, $_{17}$Cl, $_{51}$Sb
Analysis Group:	Screening
Detector:	SDD
X-Ray Tube:	Rh target
Tube Voltage-Tube Current:	10, 30, or 50 kV — Auto μA
Collimator:	10 mm dia.
Primary Filters:	#1, #2, #3, and #4
Atmosphere:	Atmospheric air
Integration Time:	100 sec
Dead Time:	Max 30 %

■ References

For information about RoHS screening analysis using the EDX-720, refer to Application News Nos. X224, X225, X226, and X227.

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