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Application News

X-ray Analysis

Screening Analysis with EDX-7000 Navi Software

No.X256

When conducting elemental screening analysis of environmentally hazardous substances using a conventional, general-purpose X-ray fluorescence instrument, a great deal of time and effort are required for both generating a calibration curve as well as managing the instrument itself. The EDX-7000, a successor to such a general-purpose instrument, together with the Navi software, greatly facilitates screening analysis.
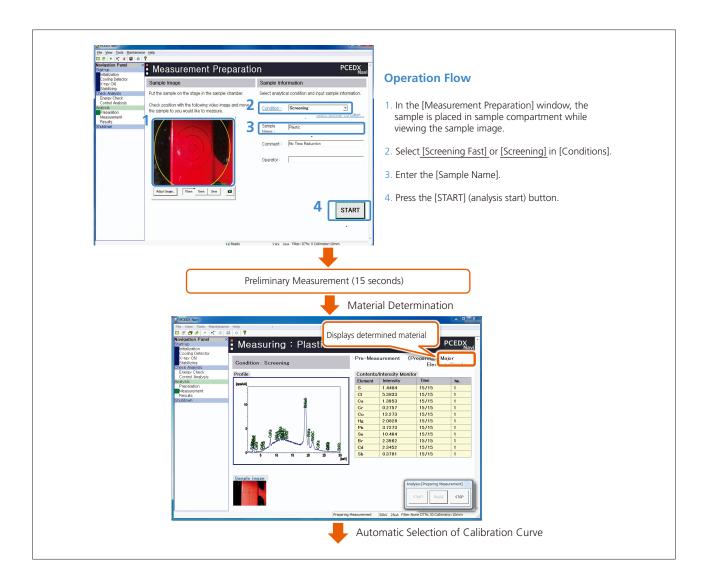
Specifically, liquid nitrogen is not required for cooling the detector with this system, and sensitivity has been improved by 1.5 to 5 times that possible with previous models. As a result, metal samples consisting of copper alloy or tin alloy, etc. can now be analyzed from 3 to 10 times more quickly than previously possible.

■ **Screening Analysis Using PCEDX-Navi Software**

Operations from standard assessment to report generation are easily conducted even by unexperienced analysts using PCEDX-Navi (RoHS, halogen, and antimony screening analysis kit). This ease of use is possible thanks to the following features.

1) All operations from instrument startup to analysis and report output are conducted on a single screen.
2) There are just 2 measurement conditions, [Screening Fast] (short time mode) or [Screening] (precision mode).
3) Automatic selection of internal calibration curves (for materials listed below)

Resin (PE, PVC), aluminum alloy, iron and steel, copper alloy, tin alloy operations, and overall analysis flow are shown in Fig. 1.

Operation Flow

1. In the [Measurement Preparation] window, the sample is placed in sample compartment while viewing the sample image.

2. Select [Screening Fast] or [Screening] in [Conditions].

3. Enter the [Sample Name].

4. Press the [START] (analysis start) button.

Preliminary Measurement (15 seconds)

Material Determination

Displays determined material

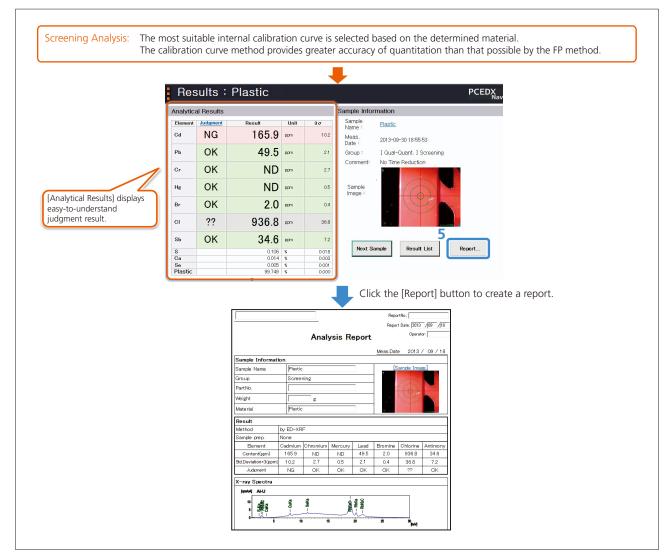Automatic Selection of Calibration Curve

Fig. 1 Flow of Screening Analysis

■ Automatic Time Reduction

Even before the set measurement time (100 sec) is reached, analysis can be suspended in advance, at the point that the judgment is output based on determination of the threshold value and quantitation value, and output of the standard deviation.
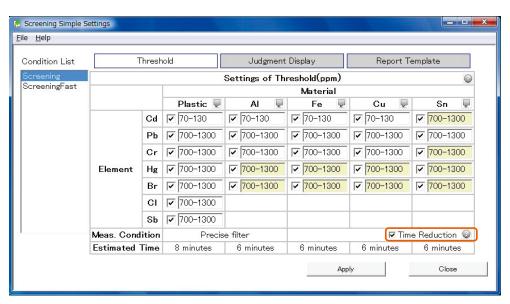

Fig. 2 Threshold, Time Reduction Conditions Setting Window

■ Example of Time Reduction

(1) Table 1 shows a comparison of the results obtained with Automatic Time Reduction turned ON and OFF, respectively, in analysis of a resin material (Fig. 3). The total analysis time took 400 sec when Time Reduction was set to OFF, and 133 sec when set to ON, clearly demonstrating the effect of Time Reduction.

Fig. 3 Resin Material

Table 1 Analytical Results and Measurement Times in Analysis of Resin Material (EDX-7000)

Automatic Time Reduction	ON				OFF			
Element	Quantitative Value [ppm]	3σ [ppm]	Judgment	Analysis Time*1 [sec]	Quantitative Value [ppm]	3σ [ppm]	Judgment	Analysis Time*1 [sec]
$_{48}$Cd	183.3	34.2	NG	11	165.9	10.2	NG	100
$_{51}$Sb	40.3	21.3	OK		34.6	7.2	OK	
$_{82}$Pb	49.7	6.2	OK	11	49.5	2.1	OK	100
$_{80}$Hg	ND	1.7	OK		ND	0.5	OK	
$_{35}$Br	2.6	1.4	OK		2.0	0.4	OK	
$_{24}$Cr	ND	7.7	OK	11	ND	2.7	OK	100
$_{17}$Cl	916.3	36.3	GRAY	100	936.8	36.8	GRAY	100
	Total			133	Total			400

*1 The thresholds settings were for Cd: 70 – 130 ppm, and for elements other than Cd: 700 – 1300 ppm.

(2) Similarly, the measurement results for copper alloy (Fig. 4) are shown in Table 2. Here, also, the effect of Automatic Time Reduction is clearly demonstrated.

Fig. 4 Copper Alloy

Table 2 Analytical Results and Measurement Times in Analysis of Copper Alloy (EDX-7000)

Automatic Time Reduction	ON				OFF			
Element	Quantitative Value [ppm]	3σ [ppm]	Judgment	Analysis Time*1 [sec]	Quantitative Value [ppm]	3σ [ppm]	Judgment	Analysis Time*1 [sec]
$_{48}$Cd	21.3	12.3	OK	36	20.8	7.2	OK	100
$_{82}$Pb	206.9	104.1	OK	20	207.7	48.0	OK	100
$_{80}$Hg	ND	78.3	OK		ND	40.6	OK	
$_{35}$Br	ND	27.3	OK		ND	13.5	OK	
$_{24}$Cr	445.3	110.4	OK	22	442.8	51.0	OK	100
	Total			78	Total			300

*1 The thresholds settings were for Cd: 70 – 130 ppm, and for elements other than Cd: 700 – 1300 ppm.

(3) For comparison, the results obtained using the EDX-LE, a dedicated screening instrument, are shown in Table 3.

Table 3 Analytical Results and Measurement Times in Analysis of Copper Alloy (EDX-LE)

Automatic Time Reduction	ON				OFF			
Element	Quantitative Value [ppm]	3σ [ppm]	Judgment	Analysis Time*1 [sec]	Quantitative Value [ppm]	3σ [ppm]	Judgment	Analysis Time*1 [sec]
$_{48}$Cd	20.7	13.3	OK	71	19.5	7.9	OK	300
$_{82}$Pb	206.3	122.8	OK	52	201.3	59.0	OK	300
$_{80}$Hg	ND	132.9	OK		ND	51.3	OK	
$_{35}$Br	ND	44.7	OK		ND	21.1	OK	
$_{24}$Cr	442.5	116.2	OK	26	447.0	48.2	OK	300
	Total			147	Total			900

*1 The thresholds settings were for Cd: 70 – 130 ppm, and for elements other than Cd: 700 – 1300 ppm.

Comparing Table 2 and Table 3, when Automatic Time Reduction is set to ON, the measurement time using the EDX-7000 was about one half that using the EDX-LE, and when set to OFF, one third the time. Furthermore, the 3σ value (standard deviation × 3) is clearly smaller when using the EDX-7000.

■ **Lower Limits of Detection**

Table 4 shows the lower limit of detection for each material as calculated from the calibration curves. As a reference, the values obtained based on use of the EDX-LE[*2] are also presented. Comparing the results obtained using the EDX-7000 and EDX-LE, the sensitivity with the EDX-7000 is 2 to 6 times higher than that using the EDX-LE.

Table 4 Lower Limit of Detection Using Screening Conditions (ppm) (EDX-7000, EDX-LE)

Element / Material	EDX-7000						EDX-LE					
	PE	PVC	Al	Fe	Cu	Sn	PE	PVC	Al	Fe	Cu	Sn
$_{48}$Cd	2	2	1	5	7	(160)	5	5	5	9	18	(180)
$_{82}$Pb	1	2	2	16	32	15	6	12	10	46	130	79
$_{24}$Cr	3	8	5	43	38	(25)	6	18	10	120	60	(88)
$_{80}$Hg	0.6	2	(2)	(27)	(31)	(21)	4	8	8	(56)	(130)	(110)
$_{35}$Br	0.3	0.5	(1)	(9)	(14)	(7)	1	4	4	(21)	(44)	(35)
$_{17}$Cl	9						25					
$_{51}$Sb	5						10					

- The data within parentheses ( ) were calculated for the FP quantitation method based on the differences of the theoretical standard deviation with respect to non-included samples.
- This is the lower detection limit with respect to each 100 sec of analysis time.

*2 Regarding Fe, Cu and Sn using the EDX-LE, the actual analysis time was 300 seconds. (The values above were calculated based on 100 seconds.)

■ **Repeatability**

Table 5 shows the repeatability obtained based on 10 repeat measurements of a C-H-B-F-5-046H metal-containing sample (LDPE)[*3] using the Screening conditions. The coefficient of variation was within 1 % at 300 ppm, and just 2 % at 100 ppm, indicating excellent repeatability.

Table 5 Repeatability (EDX-7000)

[ppm]

Element	$_{48}$Cd	$_{82}$Pb	$_{24}$Cr	$_{80}$Hg	$_{35}$Br	$_{17}$Cl	$_{51}$Sb
No. of Repetitions / Standard Value	104	309	297	300	304	893	1029
1	100	309	298	299	305	875	1016
2	102	311	290	299	304	872	1020
3	100	308	298	299	303	882	1018
4	101	311	291	300	305	908	1034
5	100	309	296	300	307	902	1024
6	102	308	297	304	309	902	1021
7	103	312	291	304	306	901	1023
8	103	312	299	301	306	882	1044
9	103	308	297	303	307	892	1031
10	108	309	294	300	306	892	1040
Mean Value	102.1	309.7	295	300.8	305.8	890.7	1027.0
Measured Standard Deviation	2.2	1.4	3.2	1.9	1.6	11.7	9.6
Theoretical Standard Deviation	2.5	2.0	3.1	1.9	2.3	9.2	8.8
Coefficient of Variation [%]	2.1	0.4	1.1	0.6	0.5	1.3	0.9

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

Analytical Conditions

Instrument	: EDX-7000	Collimator [mmφ]	: 10
Elements	: Cd, Pb, Cr, Br, Hg, Cl, Sb	Primary Filter	: #1, #2, #3, #4
Analytical Group	: Screening	Atmosphere	: Air
X-ray Tube	: Rh target	Detector	: SDD
Tube Voltage [kV]	: 10, 30, 50	Integration Time [sec]	: Max100 (Real Time) /ch
Current [μA]	: Auto	Dead Time [%]	: Max30

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