Application Note

Analysis of Foreign Matter Using CCD



The type of foreign matter adhered or mixed into food products, pharmaceuticals, and products can be easily determined in a short period of time using the EDX. If the observation capability of the CCD (optional) is utilized, foreign matter of less than 1mm can be easily measured. In addition, by using an X-ray irradiation collimator, the neighboring foreign matter can be easily separated and analyzed. 5 varieties of foreign matter (metal fragments) of about 0.3mm to 1mm were adhered or buried in a chocolate bar and used as the sample. The result of qualitative and quantitative analysis for each substance is shown below.

Sample

Unknown foreign matter A Unknown foreign matter B Unknown foreign matter C Known foreign matter D (a scrap of chocolate wrapping) Unknown foreign matter E (Refer to Fig.1)

Sample Preparation

Place the sample on the sample stage (see Fig. 2) and start the CCD image viewing software in Windows. An image like that shown in Fig. 1 will be displayed on the CRT. Fix the position by moving the sample so that the object of measurement is under the point where the crosshairs meet (Fig. 1 shows B as the object being measured). The concentric circles from the center indicate the diameter of the X-ray irradiation collimator (optional), and hence indicates the measurement range.

Results of Qualitative Analysis Qualitative analysis was carried out in air (vacuum for D only) for each foreign matter A, B, C, D & E using a 1 mm ϕ collimator. The overlaid qualitative analysis results are shown in Fig. 3. The elements detected are shown in Table 1.

Table 1 Detected Elements

Chocolate	K, Ca, Fe	
A	K, Ca, Cr, Mn, Fe, Ni, Mo	
В	K, Ca, Cu	
С	K, Ca, Fe, Cu, Pb, Sn	
D	AI, K, Ca, Fe	
E	K, Ca, Fe, Zn	

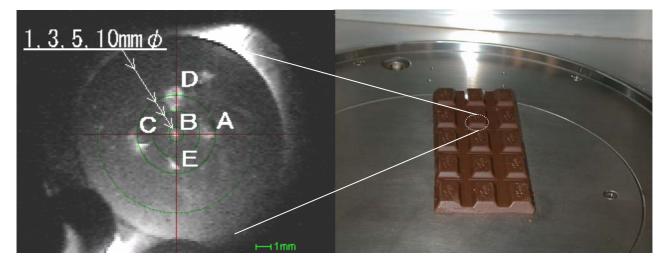
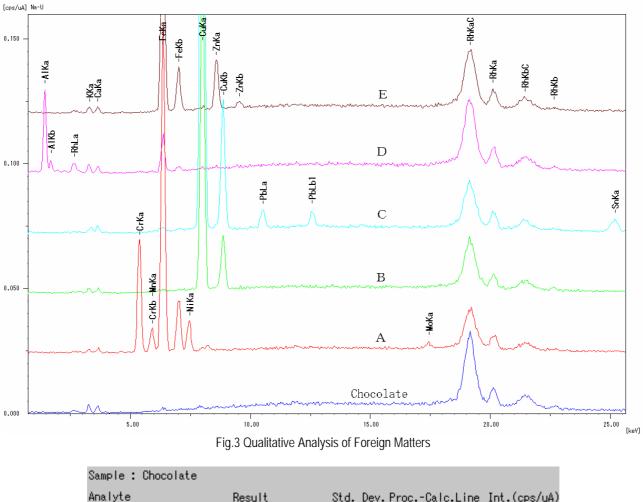


Fig.1 Picture of Foreign Matters by CCD

Fig. 2 sample stage

www.shimadzu.de





Analyte	Result	Std. Dev. ProcCalc.	Line Int.(cps/uA)
Fe Cr Ni Mn Mo	70.47 % 17.13 % 9.98 % 1.89 % 0.53 %	0.22 Quan-FP 0.24 Quan-FP 0.07 Quan-FP	FeKa 1.250 CrKa 0.409 NiKa 0.115 MnKa 0.043 MoKa 0.033

Fig.4 Quantitative Result of Foreign Matter A by FP Method

Results of Quantitative Analysis

As an example of the result of a quantitative analysis, the result of quantifying foreign matter A by the FP method is shown in Fig. 4. K and Ca are included in chocolate and were thus omitted from the quantitative calculations.

Investigation

It is inferred that the identity of each foreign matter is as follows.

- A: Stainless steel
- B: Copper wire
- C: Lead bronze
- E: Zinc plated steel wire

Shimadzu Europa GmbH

Albert-Hahn-Str.6-10, 47269 Duisburg

Analytical Conditions

Instrument:	EDX-700
X-ray Tube:	Rh target
Filter:	without
Voltage - Current:	50 kV-(Auto)
Atmosphere:	Air, Vacuum
Measurement Diameter:	1 mm
Measurement Time:	30 sec
Dead Time:	25 %



