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Application News

No.A499

Spectrophotometric Analysis

Confirmation of Raw Material Quality -Dealing with "Silent Change" Counterfeiting-

Introduction

Safe and good quality raw materials are essential to the consistent production of high-quality products. However, materials may be changed by suppliers without the knowledge of the manufacturer, either in order to reduce costs or because they are unable to accommodate changes in materials that have been dictated by regulations. This kind of change is known as a "silent change" in Japan. Use of a non-standard raw material in a product means not only quality can no longer be assured, but has been known to cause accidents and is becoming a problem for society.
Manufacturers can avoid these situations by checking at receipt whether the raw materials delivered are according to standard.
In this article, we use samples that may have been subject to a "silent change" (hereafter called "silent change" product) and introduce case analyses that confirm such changes using energy dispersive X-ray fluorescence spectrometry (EDX) (Fig. 1) and FTIR infrared spectrophotometry (Fig. 2).

Fig. 1 EDX-7000 Energy Dispersive X-Ray Fluorescence Spectrometer

Fig. 2 IRAffinity-1S with MIRacle 10 Single-Reflection ATR Accessory

Case of Replacement with a Cheaper Metal Material

Stainless steels are a special type of steel that are resistant to rust, that are created by adding various materials including chrome and nickel to iron. The stainless steel known as SUS316 has the composition 18Cr-12Ni-2.5Mo. It is created by adding molybdenum to SUS304 stainless steel, thereby improving its resistance to corrosion by sea water and other materials.
We used EDX to analyze the stainless steel in a "silent change" product and in a genuine product. Analytical conditions are shown in Table 1, and EDX profiles are shown in Fig. 3.
Fig. 3 shows that the molybdenum peak present in the genuine SUS316 is missing from the "silent change" product, which exhibits a profile identical to SUS304. This result shows the steel material in the "silent change" product has been replaced with a cheaper type.
Though it is impossible to tell by visual examination whether steel material has been changed, taking measurements by EDX provides confirmation of a "silent change."

Table 1 EDX Analytical Conditions

Instrument	: EDX-7000
X-Ray Tube	: Rh target
Voltage/Current	: 50 kV (Na-U) /Auto
Atmosphere	: Air
Measurement Diameter	: 10 mmφ
Integration Time	: 30 sec.

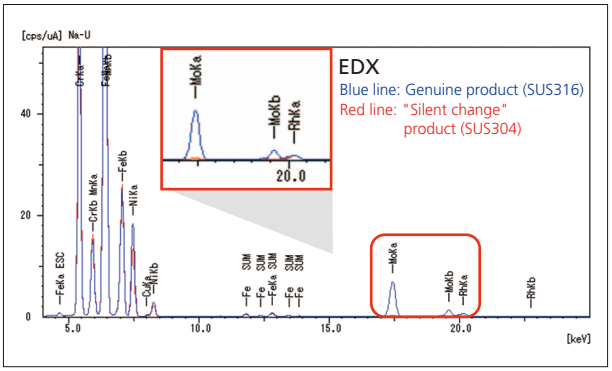

Fig. 3 Results of EDX Analysis of SUS316 Genuine Product and "Silent Change" Product (SUS304)

Case of Replacement of a Plastic Material

We used FTIR to analyze a genuine polypropylene (PP) product and a "silent change" product. Analytical conditions are shown in Table 2, and the spectra obtained are shown in Fig. 4. Fig. 4 shows a peak was detected in the "silent change" product originating from CH_2 rocking vibrations in the region of 718 cm^{-1} (marked with a star). Spectral search confirmed that the "silent change" product contains polyethylene (PE) mixed in with PP.
This result indicates that recycled plastic may have been mixed into the raw material.

Table 2 FTIR Analytical Conditions

Instrument	: IRAffinity-1S MIRacle 10 (Diamond/ZnSe)
Resolution	: 4.0 cm^{-1}
Accumulation	: 20
Apodization	: Happ-Genzel
Detector	: DLATGS

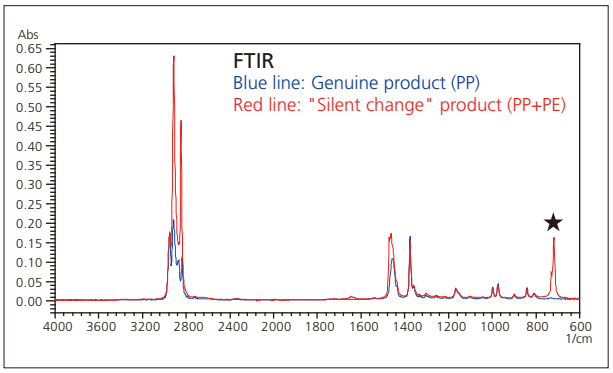

Fig. 4 Results of FTIR Analysis of Genuine PP Product and "Silent Change" Product (PP+PE)

Case of Toxic Element and Different Material Mixed into a Plastic Material

We used EDX and FTIR to analyze a genuine polyvinyl chloride (PVC) product, and a "silent change" plastic product. The EDX analytical conditions are shown in Table 1 with corresponding profiles shown in Fig. 5, while FTIR analytical conditions are shown in Table 2 with corresponding spectra shown in Fig. 6.
Fig. 5 shows that lead (marked with a star) was detected in the "silent change" product that was not detected in the genuine product. The plastic material analyzed in this experiment is subject to regulation under the Restriction on Hazardous Substances Directive (RoHS) and must not contain any lead. The results show that the "silent change" product does not meet the RoHS regulations.
Fig. 6 also shows that apart from peaks originating from PVC, peaks originating from acrylic were detected in the region of 2900 cm^{-1} and 1700 cm^{-1} (marked with a star). This result shows the presence of a different material mixed in with the material in the "silent change" product.

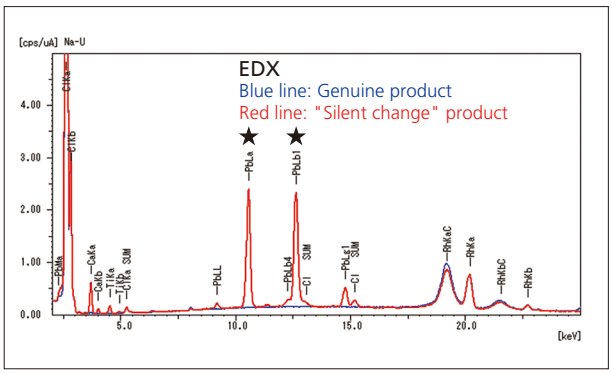

Fig. 5 Results of EDX Analysis of Genuine Plastic Product and "Silent Change" Plastic Product

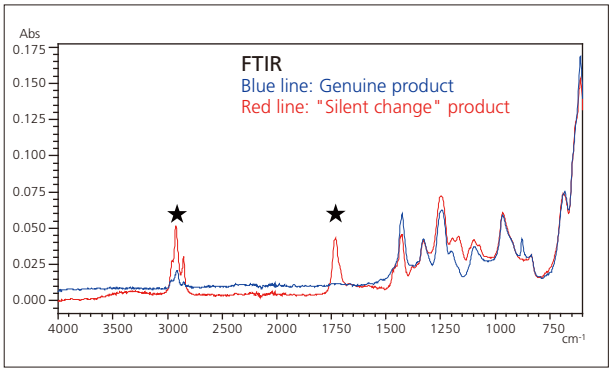

Fig. 6 Results of FTIR Analysis of Genuine Plastic Product and "Silent Change" Plastic Product

Conclusion

Using EDX and FTIR to check inorganic materials and organic materials allows for a more robust response to the problem of "silent change" counterfeiting.

First Edition: Dec. 2015

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