

Application News

Spectrophotometric Analysis

No. A406

Analysis of Black Rubber Diaphragm by FTIR and EDX

Qualitative analysis of rubber can be conducted quickly and easily using ATR measurement. With this technique, an infrared spectrum is easily obtained for qualitative analysis by affixing a rubber sample to the surface of an ATR prism. However, the composition of elements in a rubber sample can also be analyzed easily and quickly, and further, non-

destructively by EDX (energy dispersive X-Ray fluorescence spectrometry). This Application News article introduces the qualitative analysis of a rubber diaphragm sample using both FTIR and EDX techniques.

■ The Rubber Diaphragm

One type of diaphragm is basically a membrane that is mounted in the space between a moving part and a stationary part to prevent the mixing of fluids. Rubber diaphragms are available in various shapes and compositions, but a black rubber diaphragm as shown in Fig.1 was used as the sample for these analyses. This type of diaphragm is typically susceptible to deterioration at the site where it folds back on itself, and here we conducted qualitative analysis by FTIR and EDX on a diaphragm that had not yet been used.



Fig.1 Black Rubber Diaphragm Test Sample

■ Analysis of Diaphragm by FTIR - ATR Spectroscopy

Since it is highly likely that the black rubber diaphragm sample contains carbon, measurement was conducted using the MIRacle single-reflection Ge crystal ATR accessory (PIKE Technologies). Fig. 2 shows the spectral results of the ATR measurement.

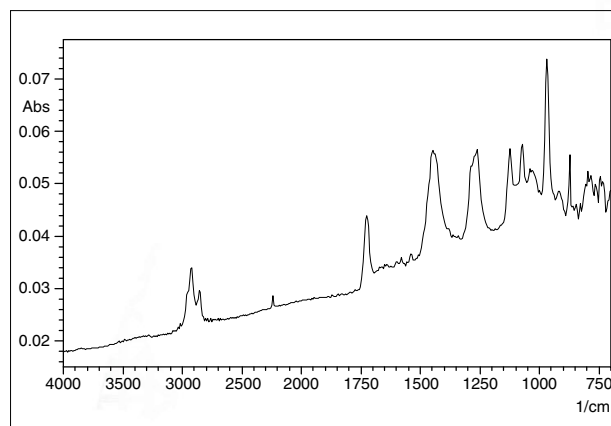
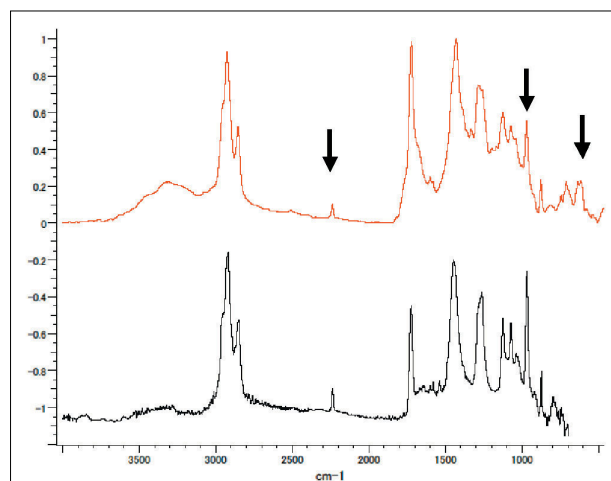


Fig.2 ATR Spectrum of Diaphragm



Name	ENSOLITE TYPE ML
Technique	FILM (CAST FROM TRICHLOROETHANOL)
Comments	Chemical Description= CLOSED-CELL FOAM; SPECIALLY MODIFIED PVC WITH NITRILE RUBBER TO STRENGTHEN CELL WALLS, PRODUCING A CROSS-LINKED POLYMER
Classification	Polymers= PLASTICIZED POLYVINYL CHLORIDES
Density	0.04 - 0.064
Source of Sample	UNIROYAL, INC.

Fig.3 ATR Spectrum Search Results (KnowItAll™ Infomatic System 7.0)

Table 1 FTIR Analytical Conditions

Resolution	: 4 cm ⁻¹
Accumulation	: 40
Detector	: DLATGS

Fig. 3 shows the results of the spectrum search with respect to the data of Fig. 2 after conducting baseline correction processing. The lower spectrum depicts the measurement results, while the upper one shows the spectrum corresponding to the database hits, a nitrile rubber spectrum containing PVC (polyvinyl chloride). In the infrared spectrum of Fig. 3, the arrows indicate a peak attributed to a nitrile group at about 2200 cm^{-1} and one attributed to the $=\text{C-H}$ out-of-plane deformation vibration peak at about 966 cm^{-1} , which confirm the existence of nitrile rubber. Another peak, attributed to a C-Cl stretching vibration at about 600 cm^{-1} confirms the existence of PVC. However, in the lower ATR spectrum of Fig. 3, the C-Cl peak is not confirmed because measurement cannot be conducted below 700 cm^{-1} due to the absorption of the Ge crystal itself.

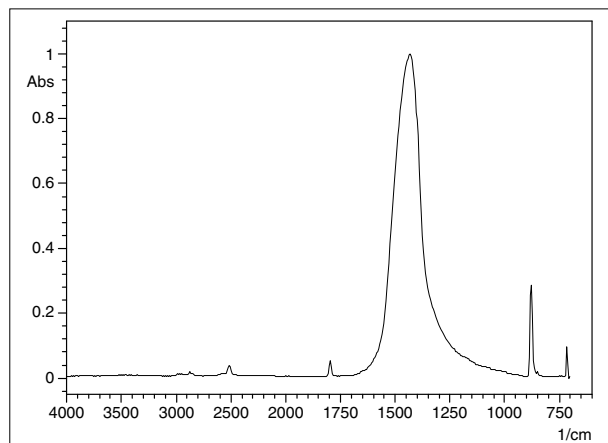


Fig.4 IR spectrum of Calcium carbonate

■ Analysis of Diaphragm by EDX

Fig. 5 shows the results of qualitative analysis of the same sample using EDX. The element range used for the analysis was Na - U. It is clear that the sample consists mainly of the elements S, Cl, Ca, and Zn.

Table 2 Analytical Conditions

Instrument	: EDX-720
X-ray Tube	: Rh target
Filter	: Filter #1 (for Cl)
Voltage	: 50 kV (for Ti - U), 15kV (for Na - Sc, Cl)
Current	: Auto
Atmosphere	: Air
Measurement Diameter	: 5 mm \varnothing
Measurement Time	: 60 sec
Dead Time	: 40 %

■ Analytical Results

It is presumed that the Cl detected by EDX as indicated in Fig. 5 is the same Cl included in the PVC shown in the search results of Fig. 3. On the other hand, the Ca is presumed to be that which is detected in the calcium carbonate. This assumption can be made because the distinct peaks (at 1430 cm^{-1} and 880 cm^{-1}) seen in the calcium carbonate infrared spectrum of Fig. 4 are also confirmed in the measurement results of Fig. 2.

From the above results, it can be assumed that the rubber diaphragm contains nitrile rubber and polyvinyl chloride, in addition calcium carbonate.

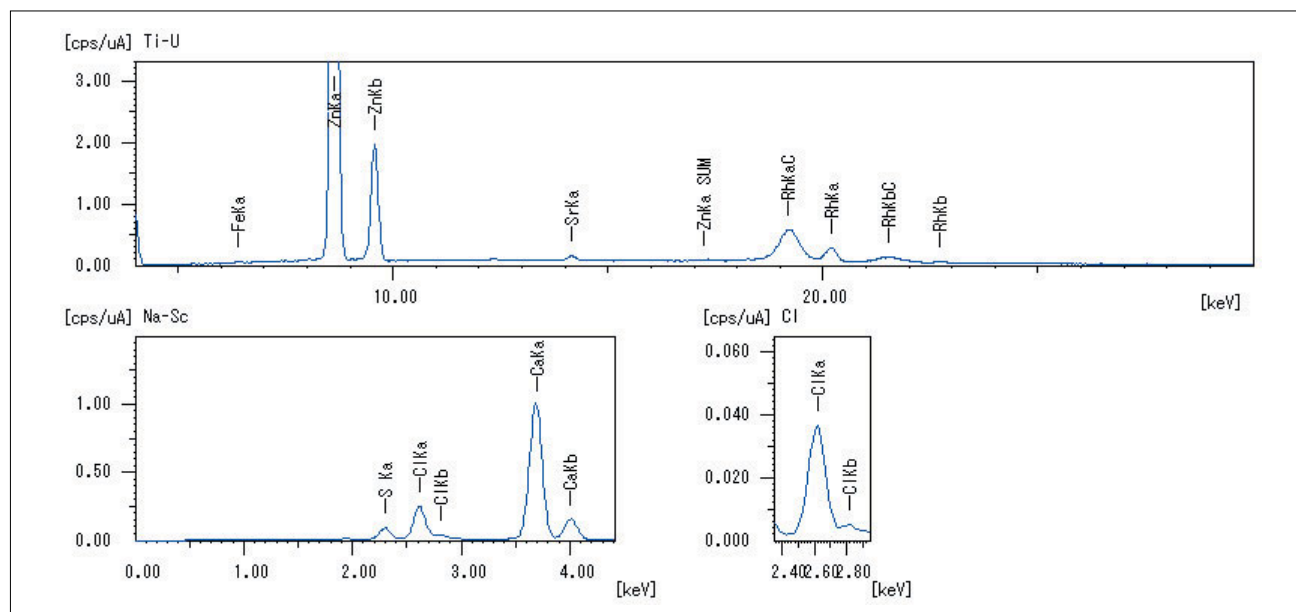


Fig.5 Results of Qualitative Analysis by EDX

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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