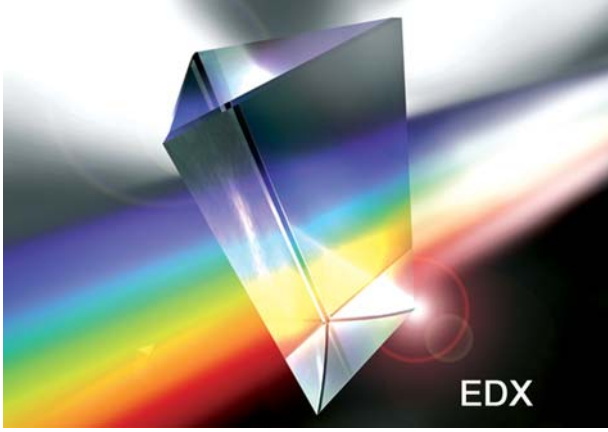
Application Note



EDXRF Analysis of Oil

When analyzing metallic elements within oil or gel-form substances the method of dropping and drying the sample onto filter is effective but difficult to handle and prepare. EDX is therefore the best and easiest method of analysis as it does not require the sample to be prepared beforehand. As liquids cannot be analyzed in a vacuum, the analysis is carried out in either air or helium. Whereas the X-ray fluorescence from a sample is only slightly absorbed in air in the case of heavy elements, with light elements this absorption greatens, so helium is used as it absorbs only a small amount.
Shown below is an example of qualitative analysis of a standard oil from Conostan Co.

Sample
Conostan S-21 Blended Standard
Hydrocarbon oil which includes 100 ppm of each element listed below.

Elements
Si, P , Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Mo, Ag, Cd, Sn, Pb

Sample Preparation
5 µm polypropylene was stuck onto the base of liquid sample receptacle into which approximately 5 ml of the sample was poured without any preparation.

Lower Limit of Detection by Qualitative Analysis
Taking this sample to be the standard sample, the lower limit of detection for each element was calculated from the results of the qualitative analysis, and this is shown in Table 1. From these results it can be seen that the ppm levels of heavy elements in the air can be detected.
In addition, a logarithmic graph of the X-ray fluorescence energy of this lower limit of detection is shown in Fig. 1.

Element	*Spectra*	*L.L.D.*	*Filter*
$_{14}Si$	Kα	77ppm	
$_{15}P$	Kα	59	
$_{20}Ca$	Kα	15	Al
$_{22}Ti$	Kα	12	Al
$_{23}V$	Kα	11	Al
$_{24}Cr$	Kα	9.6	Al
$_{25}Mn$	Kα	6.4	Ti
$_{26}Fe$	Kα	4.7	Ti
$_{28}Ni$	Kα	3.2	Zr
$_{29}Cu$	Kα	2.1	Zr
$_{30}Zn$	Kα	1.9	Zr
$_{42}Mo$	Kα	6.9	Ni
$_{47}Ag$	Kα1	14	Zr
$_{48}Cd$	Kα1	13	Zr
$_{50}Sn$	Kα1	17	Zr
$_{82}Pb$	Lα	5.3	Ni

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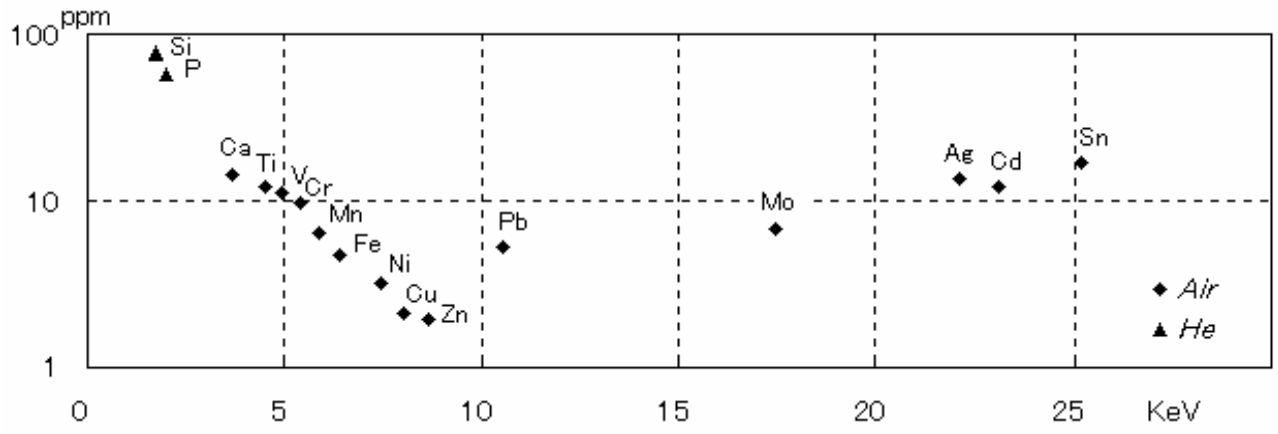
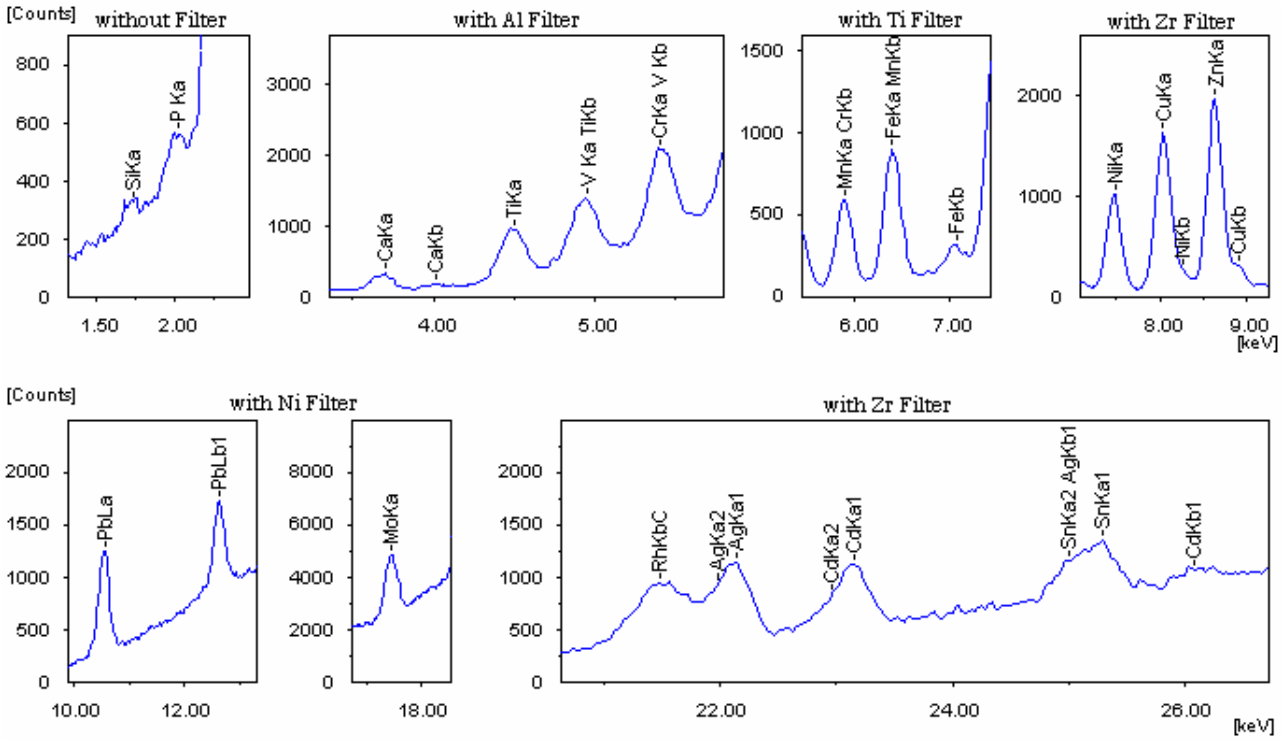


Fig.1 Lower Limit of Detection of Metal Elements in Standard Oil, S-21

Qualitative Profile

The result of the qualitative analysis is shown in Fig. 2.



Analytical Conditions

Instrument: EDX-700
X-ray Tube: Rh target
Filter: Al, Ti, Ni, Zr or without
Voltage - Current:
15 kV-301 µA(Auto), -352 µA(Auto)
50 kV-24 µA(Auto), -39 µA(Auto),
-286 µA(Auto)
Atmosphere: Air, He
Measurement Diameter: 10 mm
Measurement Time: 1000 sec
Dead Time: 24-25 %
*The equation for the Lower Limit of Detection (L.L.D.)

$$L.L.D. = 3 \times \frac{C}{Inet} \times \sqrt{Iback}$$

C = Standard Value(wt%)
Inet = Net Intensity(Counts)
Iback = Background Intensity (Counts)

The given specifications serve purely as technical information for the user.
No guarantee is given on technical specification of the described product and/or procedures.

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