Product Introduction

Oil Extraction Solvent H-519

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Oil content analyzer (OCMA series) uses solvent extraction method. The oil extraction solvent H-997 has been used in Japan. However H-997 is going to be prohibited to produce from 2020 due to the Montreal Protocol. An oil extraction solvent H-519 was newly developed instead of H-997. H-519 has low volatility and is easy to handle. The measurement value of H-519 and H-997 are different, but shows consistent correlation. Therefore, H-997 can be replaced to H-519 by compensating the correlation analysis. In this article, the features of H-519, differences from H-997 and tips of solvent switching are introduced.

Key words

Oil concentration Infrared absorption Residual oil Oil extraction solvent

Introduction

Oil is widely used in daily life and in many industrial fields. Improper use and discharge of oil leads to environmental pollution because oil has poor biodegradability in many cases. Therefore, it is necessary to measure and control oil content for its effective use and environmental conservation. Devices and methods for quantitatively measuring have been used for many years.

HORIBA group offers a lineup of oil content analyzers (OCMA series). This series enable to measure oil content easily by extracting oil to solvent. With changes in environmental regulations, extraction solvent has been changed from carbon tetrachloride to safer fluorochemical solvent such as CFC (Chlorofluorocarbon) and HCFC (Hydrocholofluorocarbon). However, the regulations of chemical substances are further strengthened globally to prevent ozone depletion and global warming. Some fluorinated solvent have also increasingly been regulated for its purpose.

Production of oil extraction solvent H-997 (HCFC), which has been conventionally used in OCMA -505, 555, is also prohibited after 2020 (Table 1) in the Montreal Protocol^[1].

HORIBA Group has continuously researched oil extraction solvent that replaces H-997 for many years. As a result, we have newly developed H-519(Figure 1). H-519 is not regulated as ozone depleting substances. In this article, we introduce the features of H-519 and its compatibility with H-997.

Oil content analyzer OCMA series

In OCMA series, solvent extraction non-dispersive infrared absorption method is used. Oil content is calculated according to Lambert-Beer law, being measured



Figure 1 Oil extraction solvent H-519

able 1	Controlled substances	of the Montreal	Protocol (Annex	C)
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Group	Substance	Number of isomers	Ozone-Depleting Potential
Group I			
CF ₃ CF ₂ CHCl ₂	(HCFC-225ca)**	—	0.025
CF ₂ CICF ₂ CHCIF	(HCFC-225cb)**	—	0.033

Non-dispersive infrared absorption method can detect C-H bonds almost equal to the smallest unit of the molecular structure of oil. Therefore, this method can measure saturated, unsaturated or aromatic oils ^[2]. On the other hand, UV method and fluorescence method can detect only oil having unsaturated bond in molecular structure. Non-dispersive infrared absorption method is also possible to detect highly volatile oils which can not be detected by the gravimetric method (i.e. normal hexane extraction method)^[3].

Table 2 shows OCMA series in Japan. With the change of the oil extraction solvent, OCMA-505-H and 555-H (For H-519) are introduced instead of OCMA-505, 555. Calibration oil is also changed from OCB standard material (mixture of octane, cetane and benzene) to tridecane.

OCB standard substance contains benzene classified as hazardous substances according to GHS. On the other hand, tridecane is a safer substance because it is not listed in regulations about safety.

Oil extraction solvent H-519

Overview and features

Table 3 shows a comparison between H-997 and H-519. The main components of H-997 and H-519 are different; the former is dichloropentafluoropropane and the latter is mainly composed of chlorotrifluoroethylene trimer. Physical property such as boiling point, viscosity, oil solubility and safety are different from each other. Compared with H - 997, although the KB value which is an index of oil solubility falls from 31 to 25 in H-519, H-519 is not listed in safety regulations.

H-519 has not only a higher boiling point than H-997 but

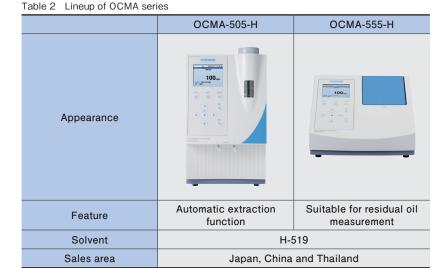


Table 3 Comparison of specification of H-997 and H-519

	H-997	H-519
Main component	Dichloropentafluoropropane	Chlorotrifluoroethylene (Trimer)
CAS No.	507-55-1 42-56-0	9002-83-9
Boiling point (°C)	54	> 135
Vapor pressure(kPa) @25℃	38	1.2
Density (g/cm ³)	1.6	1.8
Viscosity (mPa ⋅ S) @25℃	0.6	4.3
KB value ^{*1}	31	25
GHS ^{*2}	\Diamond	—
Montreal Protocol	Listed	Not listed
PRTR	Listed	Not listed

*1: Kauri butanol value

It is an index of grease saturation ability. The larger value, the more oil sample dissolves. *2: Globally Harmonized System of Classification and Labelling of Chemicals

also low volatility due to its low vapor pressure at 25 degrees Celsius. Therefore, H-519 hardly volatilize during extraction step for oil measurement, resulting in less measurement error due to solvent volatilization. In addition, low volatility of H-519 also increases solvent collection



Figure 2 Solvent reclaimer

absorption attributable to C-H stretches at 3.4 to 3.5 μ m (2941 to 2857 cm⁻¹). Therefore, it is reasonable that extraction solvent does not show absorption in this wavelength range. Since H-519 has no carbon-hydrogen bond (C-H bond) in its molecular structure and does not have specific absorption between 3.4 to 3.5 μ m, it is suitable for oil measurement in OCMA series.

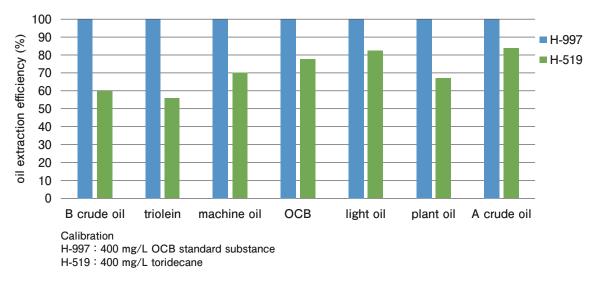


Figure 3 Measurement results of 100 mg/L oil in water using H-997 and H-519 (HCI was added as a acidification regent)

rate in reclamation. The recovery rate of solvent after reclamation has improved to about 90% in H-519 (about 60% in H-997). Figure 2 shows a solvent declaimer for reclaiming used solvent. The solvent reclaimer consists of two layers of activated carbon and activated alumina. Oil is removed by activated carbon, and activated alumina removes moisture and polar substances. Difference in measured value between H-997 and H-519.

Difference in measured value between H-997 and H-519

Difference in oil extraction efficiency between H-997 and H-519 leads to different measurement values in OCMA. Oil extraction efficiency is calculated by following equation.

oil extraction efficiency (%) = measured value (mg/L) / actual sample conc. (mg/L) ×100. Figure 3 shows results of measurement of 100 mg/L various oil in water with OCMA-505 (For H-997) and OCMA-505-H (For H-519), and measured value by H-997 is set to 100%. For all oil types, the measured value at H-519 was lower by about 20% to 40%. This difference in extraction efficiency is

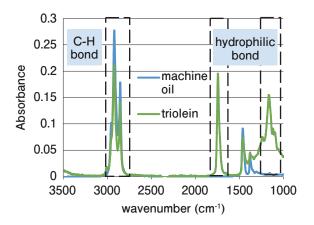
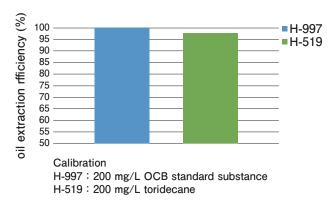


Figure 4 IR Spectrum of machine oil and triolein

thought to be mainly due to the difference in KB value between both solvents. It is indicated that the difference in extraction efficiency depending on the oil type was due to the affinity between solvents and oil. For example, **Figure 4** shows IR spectrum of machine oil and triolein. Triolein has a hydrophilic carbonyl group (C=O) in its molecular structure. It is expected that affinity will increase due to the fact that hydrogen molecule contained in the carbonyl group makes hydrogen bond with hydrogen molecule of H-997 by C-H...O interaction. However, H-519 does not have hydrogen molecule in its molecular structure and does not form hydrogen bonds with triolein. For this reason, it is expected that extraction efficiency will increase with H-997 rather than H-519 for triolein.

Since machine oil has almost no hydrophilic group, it is expected that it is difficult to form hydrogen bond with H-997. Therefore, the difference in the extraction efficiency of both solvents is smaller than triolein. From the above results, the extraction efficiency depends on the affinity between both solvent and oil.



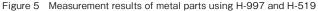


Figure 5 shows comparison of measured values using H-997 and H-519 about residual oil on metal parts. In the measurement of residual oil of parts, extraction efficiency of H-519 was about 5% lower than that of H-997, but difference in extraction efficiency was small compared with that of oil in water. However, H-519 has lower oil dissolving power (lower KB value) than H-997, and it is hard to infiltrate the microstructure of the part due to its high viscosity. When extracting oil, it is recommended to use ultrasonic wave or shaking for the purpose to enhance a penetration of solvent to oil.

Value correlation for switching to H-519

As described above, the measured values are different between H-997 and H-519. Therefore, when switching from H-997 to H-519, it is necessary to check correlation data between measured values using both solvents. In most cases, correlation expressed by a linear function is obtained between two solvent. Consequently, the same management and operation as before based on correlation data is possible.

For correlation confirmation, the measurement values using H-997 and H-519 need to be measured. Then, the values of H-997 and H-519 are plotted to X and Y axis on a graph, respectively. Figure 6 shows an example. In this case, a correlation formula was y = 0.96 x + 0.03. From this equation, the measured value of H-519 is 0.96 times the measured value of H-997. There are two ways to apply this result to a standard value. (1)Change the standard value using the coefficient obtained by correlation confirmation, (2)Adjust measured value at H-519 to the existing standard value by using concentration correction function. The concentration correction function is newly added to OCMA-505-H and 555-H. In the case of Figure 6, by inputting 1.1 (1 / 0.96) to the measurement setting of OCMA, the measured value of H-519 can be adjusted to the standard value used in H-997.

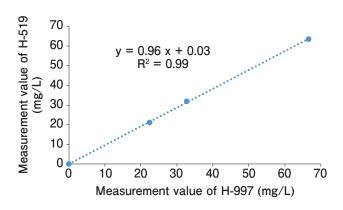


Figure 6 Correlation of measurement value between H-997 and H-519

Conclusion

The oil extraction solvent H-519 introduced in this article is environmentally friendly solvent because it is not listed to regulations and has low volatility. This low volatility makes it possible to perform a stable extraction operation than the conventional H-997, and it seems that the type of application for oil concentration measurement in OCMA will also be expanded. We believe we can contribute to the global environment protection, though it is in small portion, by developing applications of oil concentration measurement using H-519.

* This content is based on our investigation at this publish unless otherwise stated.

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