Product Introduction

Oil Content Analyzer OCMA-500 series

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Measuring and controlling the concentration of oils released into the environment is essential for aquatic conservation perspective. In this paper, our new oil content meter OCMA-500 series with 20% decrease in the usage of solvents and improved usability is introduced. A comparative measurement results between OCMA-505' NDIR method and n-hexane extract method are also reported, in addition to the measurement results of the comparison of OCMA's extract solvent. These results indicate that OCMA-500 series is applicable to the measurements of oils which are high volatile and low molecular weight.

Introduction

The world's population has exceeded seven billion and the load on the global environment caused by mankind's activities is increasing. Despite the growing population, only less than 1% of the water on earth is available for humans to drink. Conservation of water environments will be increasingly important in the future[1]. In particular, oil discharged to rivers and oceans is one of the causes of water environment pollution. In Japan, the Water Pollution Control Act and the Sewerage Act stipulate the standards for oil content in wastewater. Table 1 shows a list of methods for measuring oil content[2]. As a main method for oil content measurement, a gravimetric method (n-hexane extraction method) is described in the Japanese Industrial Standards (JIS). However, the method requires cumbersome laboratory manipulations such as extraction, separation, solvent evaporation, weight measurement, and so on. In addition, volatile oil will form an azeotrope with the solvent and boil during evaporation of the solution to dryness.

Meanwhile, so far we have been selling the OCMA-300 series, which enables easy determination of oil content by automatically performing the processes from oil extraction to measurement. The OCMA-300 has been used not only for the simple measurement of oil content in water, but also for measuring the residual oil in solids or on surfaces of products or goods-in-progress (mostly made of metal). Figure 1 summarizes the features of the

Table 1 Features of oil concentration measurement method[4]

		Gravimetric	Infrared ray			Fluorescence	Gas chromatograph	Turbidity	Quartz resonator	Orgastor
			Fourier transform Dispersive	Non-dispersive	Laser					
Solvent	dichloroethane	possible	possible	possible	unnecessary	unnecessary	possible	unnecessary	unnecessary	unnecessary
	trichloroethane	possible	possible	possible						
	S-316	impossible	possible	possible						
	H-997	possible	possible	possible						
	n-hexane	possible	impossible	impossible						
detection	volatile oil	impossible	possible	possible	possible	possible	possible	possible	possible	possible
	saturate oil	possible	possible	possible	possible	impossible	possible	possible	possible	possible
Standard measurement time		2 hour	3 min	3 min	real-time	continuous	30 min	15 min	real-time	real-time

Figure 1 Appearance of OCMA-500 series

OCMA-500 series, a successor product to the OCMA-300. The OCMA-500 series consists of the following models, which can be selected depending on the extraction solvent to be used: the OCMA-505 (Figure 1, for H-997) and the OCMA-500 (for S-316).

Measurement Principle

Oil content measurement in the OCMA-500 series is based on the non-dispersive infrared absorption method (non-dispersive type listed in Table 1), which has been used in the OCMA-300 series (see Figure 2). In comparison with other methods listed in Table 1, the non-dispersive infrared absorption method is marked by its capability of detecting both, volatile and saturated oils/fats, at a very short measurement time. A light beam from the infrared light source is directed to the cell filled with the solvent, which contains the extracted oil.. After passing through the cell, the beam is be modulated by a chopper. Then, through an interference filter, only the absorption wavelength band attributable to the stretching vibration of carbon-hydrogen bond (3.4-3.5 μm) is introduced to the detector (pyrosensor). The higher the oil content in the extraction solvent is, the more sharply the light passing through the cell attenuates, and the lower the

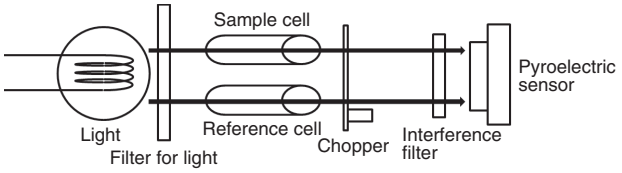

Figure 2 Diagram of light, cells and detector

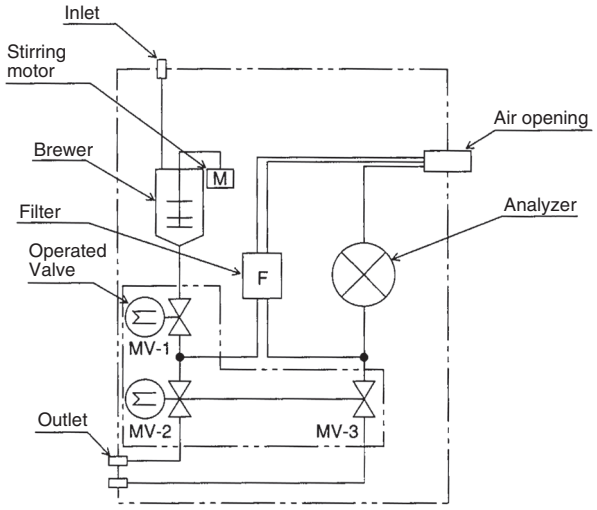

Figure 3 Flow diagram of OCMA-500 series

electric current generated by the pyroelectric effect becomes. This electric current value can be used to calculate the oil content. Meanwhile, fluctuations in light intensity of the light source is corrected in real time using the double-beam method in which the light is passed through a sample cell and a reference cell.

Basic Configuration

The OCMA-500 series can perform the processes from oil extraction to drainage in a fully automatic manner. Figure 3 shows the measurement flow. The instrument consists of an extraction chamber, an automatic switching valve for pumping the sample liquid, a water filter and an analyzing unit. The sample water is introduced to the instrument and is mixed with the oil extraction solvent in the extraction chamber, where the oil is extracted into the solvent. Next, the automatic switching valve opens to pump the extraction solvent by means of hydraulic head pressure. Water is removed by the water filter and only the extraction solvent is sent to the analyzing unit.

Specifications and Features

The OCMA-500 series can perform a set of processes, from extraction of oil from the sample to feeding, analysis and drainage of the solvent, in a fully automated manner. Here we describe the improvements from the OCMA-300 series.

The global warming potential of the extraction solvents H-997 and S-316 is 370 and 5000 (raw material of S-316), respectively, and the use of these solvents should be minimized. In the wake of growing awareness to environmental conservation, mitigation of the environmental load of the measuring instrument itself is

Table 2 Property of H-997

Chemical formula	$CF_3CF_2CHCl_2CClF_2CF_2CHClF$
Molecular weight	208
Boiling point	54°C
Melting point	-131°C
Density	1.55 g/mL (25°C)
Vapor pressure	0.0377 MPa (25°C)
Saturated solubility in water	0.033 g/100 g (25°C)
Acute oral toxicity (LD50)	5 g/kg or more

an issue that must be addressed in the product development stage. For this purpose, the OCMA-500 series has adopted an optimized structure with a slim-shaped extraction chamber, successfully achieving a 20% reduction of the amount of oil extraction solvent used. The series uses an aluminum gasket in the light source instead of a conventional lead gasket, and electronic parts that are compliant with RoHS (Restriction of Hazardous Substances) in the circuit board.

In addition, the operability has further improved by reflecting the feedback from customers that use the OCMA-300 series. An LED lamp is used to illuminate the boundary face in the extraction chamber to facilitate easy checking of the separation of the sample water and the extraction solvent in the chamber[3]. Also, a 3.5-inch color LCD has been adopted for the panel screen. Furthermore, the series provides a USB port to allow easy operation of the instrument and data management on a PC. As a result of these modifications, a reduced environmental load and improved usability have been achieved.

Extraction solvent

The OCMA-500 series uses the H-997 or S-316 solvent to extract oil from the sample. Of these, H-997 is supplied for use in Japan. Table 2 shows the physical properties of H-997, and Figure 4 illustrates the infrared absorption spectra of H-997 and a standard mixture of OCB (iso-octane, cetane, benzene). Having a low ratio of C-H bonds in its molecular structure, H-997 absorbs less infrared radiation at wavelengths of 3.4-3.5 μm. When an oil with C-H bonds is extracted in the solvent, the C-H bonds absorb additional infrared light. The OCMA uses the difference in absorption to determine the oil content. For reclaiming the used oil extraction solvent, the solvent reclaimer SR-305 is provided. The device has a double-layered structure with activated charcoal and activated aluminum. The activated charcoal layer removes oil and the activated aluminum removes water and low-molecular polar substances. The device enables recycling of the solvent and also helps reduce running costs.

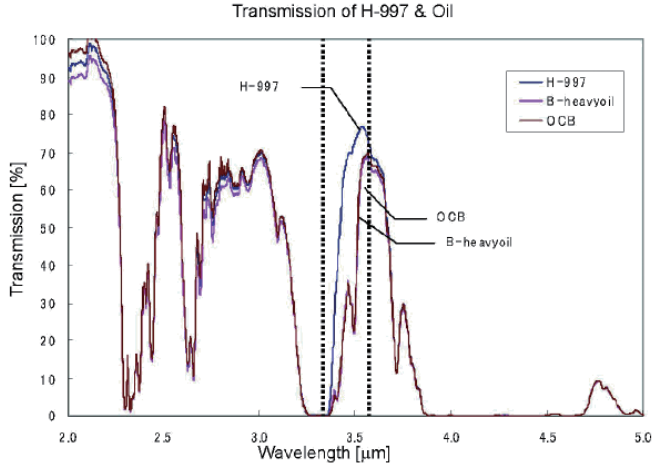

Figure 4 Infrared absorption spectrum of H-997, B-heavy oil and OCB standard solution.

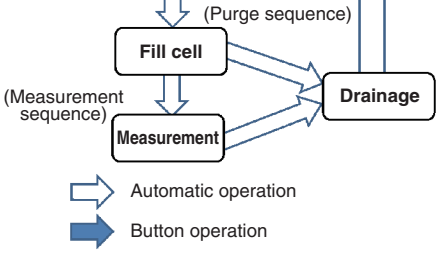

Figure 5 Operation flow of automatic measurement

Measurement Procedures

The measurement flow of the OCMA-500 is shown in Figure 5. Compared with the OCMA-300 series, the OCMA-500 allows easy operation without instruction manuals because the instrument displays operation guides (selection of the next step) as shown in Figure 6.

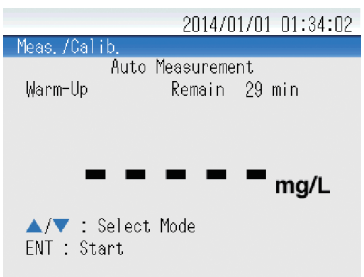

Figure 6 Start of automatic measurement mode

For an ordinary measurement, the instrument should be pre-rinsed at least twice with the sample water to be analyzed to eliminate any impact of the one previously examined. Particularly, if the difference of oil content between the new sample and the previous one is 100 mg/L or larger, pre-rinse must be performed five times or more.

Typical operating procedures are as follows: Use the MEAS button or up/down buttons to activate the "Automatic" measurement mode. Then inject 8 mL of the solvent from the inlet using a measuring cylinder or syringe for solvent. Use a dropper to add a drop of hydrochloric acid from the inlet. Inject 16 mL of the sample water from the inlet using a measuring cylinder or syringe for sample. These liquids should be fed in the order stated above to prevent the hydrochloric acid from remaining on the inner surface. Finally, mixing can be started by pressing the ENT button on the membrane key pad. If a pre-rinse process is performed, extraction, layer separation, liquid conveyance, and drainage are automatically carried out as shown in the flow in Figure 5. If a measurement process is performed, extraction, layer separation, conveyance, measurement, and drainage are carried out in an automated manner. The result is displayed after measurement, which can be taken out using a USB flash drive.

Properties of Oil Content Analyzer

Relative sensitivity for various oil types

For controlling oil content in wastewater, normal hexane (n-hexane) extracts specified in JIS are mainly used as an indicator for management. n-hexane extracts refer to the residual that remains after hexane extraction and subsequent evaporation of hexane at 80°C[4]. In this way, however, a highly-volatile oil cannot be detected because it forms an azeotrope with n-hexane and boils. Table 3 shows the change in weight of various types of oil; the data is obtained by sampling a prescribed amount for each oil type, adding 1 mL of n-hexane and then heating for 30 minutes at 80°C. Machine oil and clear kerosene, which tend to form an azeotropic mixture, have a residual ratio of 38% and 23%, respectively, indicating that these oil types are hard to measure by using n-hexane extraction. On the other hand, the OCMA series does not require evaporation of the solvent and can directly measure the content of volatile oil carried in the solvent. Figure 7 shows the results of analyses in which oily water with an oil content of 20 mg/L is measured using the OCMA-505 (with H-997) and the n-hexane method, respectively. Measured values for each oil type obtained by the n-hexane method were as low as about 20% compared to the oil content. Particularly, the recovery rate of clear kerosene was no more than 1%. In contrast, the OCMA obtained successful measurement results with a recovery rate of about 80% in many oil types except for vegetable oil. Meanwhile, the reason why the measurement values do not reach 100% of the oil content and the recovery rate differs by oil type is the difference in oil extraction ratios.

Table 3 Weight change after adding 1ml of n-hexane to each oil and heating at 80 degree for 30 min.

	B-heavy oil	machine oil	ARB crude oil	heating oil
initial weight (mg)	55.4	60.6	54.6	60.1
weight after heating at 80 degree for 30min (mg)	35.3	23.2	41.6	14.1
residual ratio (%)	64	38	76	23

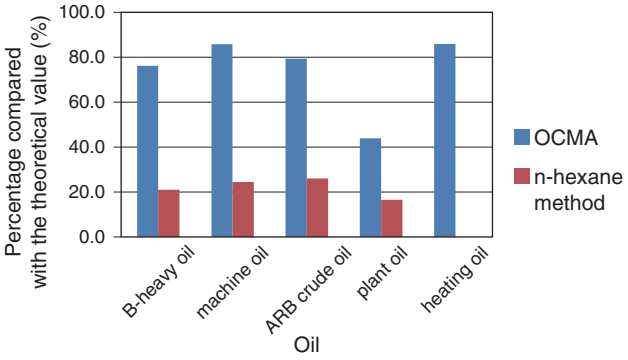

Figure 7 Measurement results of 20 mg/l oily water using OCMA-505 and n-hexane method

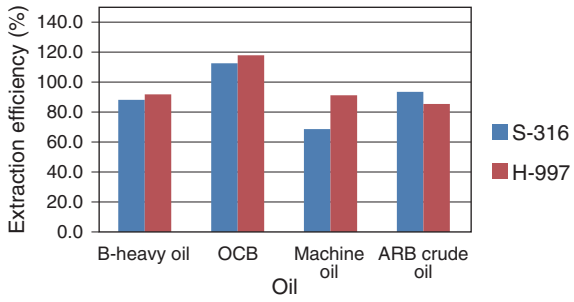

Figure 8 Extraction efficiency of each oil measured by using H-997 and S-316

The results indicates that the OCMA-500 series can be used for measuring low-volatile, low-molecular oil types. The series will especially help in measuring residual oil (e.g. machine oil) on machined metal parts, where it is necessary to prevent degradation of performance due to contamination, as well as emission of bad odor and smoke caused by heating.

H-997 and S-316: Comparison of extraction efficiency for each oil type

Figure 8 shows the extract efficiency for various oil types marked by the OCMA-500 series with use of solvents

H-997 and S-316, where water with oil content of 20 mg/L was analyzed for each oil type. The extraction efficiency η is expressed as follows:

$$\eta = \frac{B}{A} \times 100$$

where A is the measured value of the liquid extracted by shaking the solvent and sample in a separating funnel for three minutes, and B is the measured value of the liquid extracted in the extraction chamber attached to the OCMA-500 series. Meanwhile, the OCMA-500 series was calibrated using a standard mixture solution of OCB. The difference in extraction efficiency between these solvents were almost constant with any oil types. However, the extraction efficiency itself varies depending on the oil type; so if the type of the oil to be measured is known in advance, it is desirable that the oil type be preferably used for calibration of the instrument.

Conclusion

As stated above, the OCMA-500 series can perform measurement using 20% less solvent and sample than former products, and provides various features such as backlight for improved usability. This is a "user- and eco-friendly" oil content analyzer capable of measuring at a single touch of a button. Based on the non-dispersive infrared absorption method, the OCMA-500 series is a simple and sophisticated oil content analyzer that can quickly analyze water with low oil content or sample that contains highly-volatile oil. The OCMA series have been widely used, not only for examining environmental water, but also for indicator monitoring by measuring residual oil on the parts for industrial products, as well as for analyzing oil content in soils. These series are becoming the de facto standard of these measurements. They will also be utilized in food industries and for indicator monitoring in the management of cleanliness (oil content).

References

[1] I.A. Shiklomanov, John C. Rodda, World Water Resources at the Beginning of the Twenty-First Century, Cambridge University Press, 13 (, 2004)

[2] Electric Measuring Instruments Manufacturers' Association, edited by environment instrument guidebook

[3] Already applied the patent on the backlight

[4] Testing method for industrial wastewater, JIS K0102, Japanese Industrial Standard.

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