Introduction of the Quantum Yield Measurement Method of the Slight Quantity Phosphor Dilution by Aluminum Oxide Powder

INTRODUCTION

By using F-7000 fluorophotometer with the quantum yield measurement system, the quantum yield of a powder phosphor can be measured. Usually, about 1.5 cm^3 of a sample is required while in some cases, the sample cannot be obtained in a sufficient quantity. This time, to describe a know-how of the quantum yield measurement method for a slight quantity of a phosphor, sodium salicylic acid powder was used as the sample and the analysis method for a sample in a slight quantity by using a powder diluent is introduced.

It is desired that powder diluents absorb little light, are highly reflective, have a high chemical stability, and are low cost. This time, the powder diluents shown in Figure 1, PTFE powder, aluminum oxide, and barium sulfate, were studied.

For the selection of a powder diluent, the reflectances of highly reflective powders used for the dilution were measured by using U-4100 spectrophotometer (Figure 2). As a result of the reflectance measurements, it was found that the PTFE powder and aluminum oxide have a high reflectance and thus, they are suitable for the dilution. While the reflectance of PTFE powder is high, the cost is higher compared with the other materials. In addition, its static electricity causes a poor mixing performance, resulting in handling difficulty. After taking the reflectance property, safety, mixing performance, handling, and cost into consideration, aluminum oxide powder was selected as the powder diluent.

Evaluation of Powder Diluent Properties

Figure 1 Appearances of Powder Diluents Studied

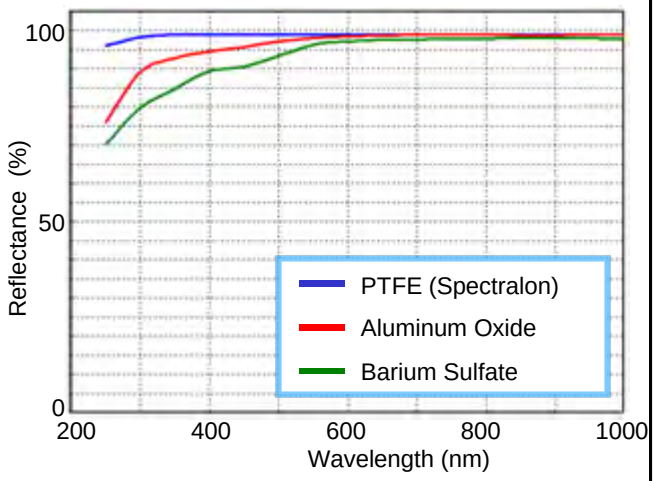


Figure 2 Reflectance Spectra of Various Reflective Materials

Table 1 Properties of Various Reflective Materials

	PTFE powder	Aluminum oxide	Barium sulfate
Reflectance property	○	○	×
Safety	○	○	△
Mixing performance・Handling	×	○	○
Cost	×	○	○

Hitachi U-4100 Spectrophotometer

* The content of this datasheet has already been introduced at the 58th Annual Meeting of JSAC.

KEY WORDS
Material/Processing Material Related, Pigment/Paint/Dye, Aluminum Oxide, Phosphor, Slight Quantity, Trace Amount, Dilution, Quantum Yield, Al_2O_3, Aluminum Oxide Powder, UV, U-4100

Fluorophotometer (FL)

Sheet No. FL110010-01A

Introduction of the Quantum Yield Measurement Method of the Slight Quantity Phosphor Dilution by Aluminum Oxide Powder

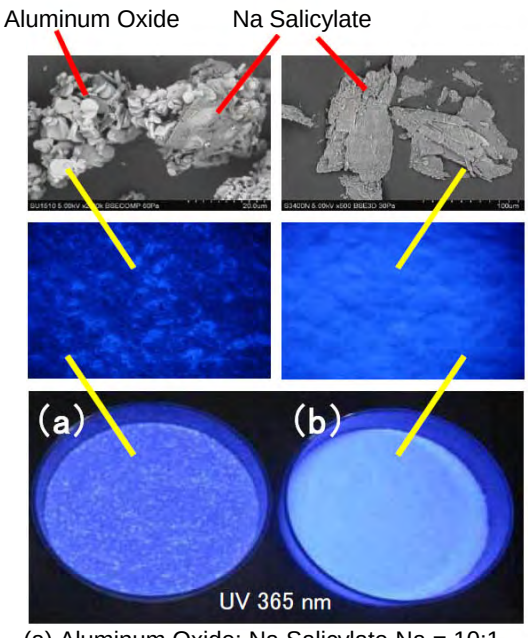
INTRODUCTION

A stereoscopic microscope and an electron microscope were used to confirm the mixing state of a powder diluent and a sample. Aluminum oxide (Al_2O_3) and Na salicylate ($C_6H_4(OH)COONa$) were mixed at a ratio of 10:1.
During the observation under the stereoscopic microscope, a photo was taken with the irradiation of ultraviolet ray at 365 nm from a black light lamp. During the observation under the electron microscope, in addition to the examination of the image by the electron microscope, the mixing state of aluminum oxide and Na salicylate was confirmed by energy dispersive X-ray spectrometry (EDX).
Based on the SEM image and element mapping image, it was found that the mixing was achieved by the adherence of the aggregated aluminum oxide particles to the plate-shaped particles of Na salicylate.

OBSERVATION CONDITIONS

Instrument	: Hitachi SU1510 scanning electron microscope	Instrument	: Hitachi S-3400N scanning electron microscope	Energy dispersive X-ray spectroscopy	Horiba, Ltd. EMAX ENERGY
Accelerating Voltage	: 5.0 kV	Accelerating Voltage	: 5.0 kV		
Observation Magnification	: × 2000	Observation Magnification	: × 500	Stereoscopic microscope	OLYMPUS SZ40
Detection mode	: BSECOMP (backscattered electron compositional image)	Detection mode	: BSE3D (backscattered electron 3D image)		
Vacuum degree	: 60 Pa	Vacuum degree	: 30 Pa		

Confirmation of Mixing State



(a) Aluminum Oxide: Na Salicylate Na = 10:1
(b) Only Na Salicylate

Figure 3 Mixing State of Aluminum Oxide and Na Salicylate

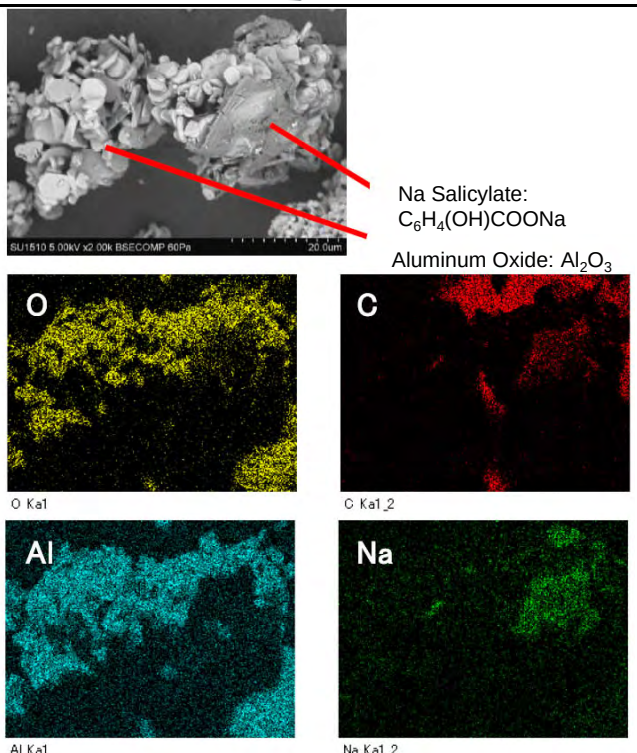


Figure 4 Element Mapping Image of Mixed Powder

KEY WORDS
Material/Processing Material Related, Pigment/Paint/Dye, Aluminum Oxide, Phosphor, Slight Quantity, Trace Amount, Dilution, Quantum Yield, Al_2O_3, Aluminum Oxide Powder, SU1510, S-3400N

Fluorophotometer (FL)

Sheet No. FL110010-02A

Introduction of the Quantum Yield Measurement Method of the Slight Quantity Phosphor Dilution by Aluminum Oxide Powder

INTRODUCTION

The relationship between the dilution rate and measured quantum yield value was obtained by gradually mixing a powder diluent and a sample.
The dilution rates were calculated by assigning the sample amount of 1.5 cm^3, which is the amount generally used, as a dilution rate of 1. For Na salicylate evaluated this time, a consistent quantum yield was obtained up to 80 times dilution. This dilution is equivalent to the sample amount of about 0.0019 cm^3 and thus, it was confirmed that the fluorescence quantum yield could be measured with the sample amount as small as this. F-7000 fluorophotometer, with the highest sensitivity for the instrument class and a dynamic range of 6 degrees of magnitude, allows highly accurate measurement of a weak fluorescence due to a high dilution rate.

* It is expected that the maximum dilution differs depending on the fluorescence properties of the sample and wavelength range.
* It is recommended that a preliminary study be conducted with a sample having similar properties when applying this method to an actual sample.

ANALYSIS CONDITIONS

Instrument	: F-7000		
Excitation wavelength	: 350 nm	Scan speed	: 240 nm/min
Fluorescence wavelength	: 330 - 600 nm	Response	: Automatic
Slit on excitation side	: 5 nm	Detector	: R928F
Slit on fluorescence side	: 5 nm	Photomultiplier Vol.	: 350V

Calculation conditions

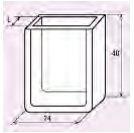
Wavelength range for absorbance calculation : 340 - 360 nm

Wavelength range for fluorescence calculation : 365 - 600 nm

ACCESORY

Quantum yield measurement system (P/N : 5J0-0148)

S20 Standard Cells with U-Shape Bottom Products of GL Sciences Inc. (GL Sciences Cat. No. 6210-21203)



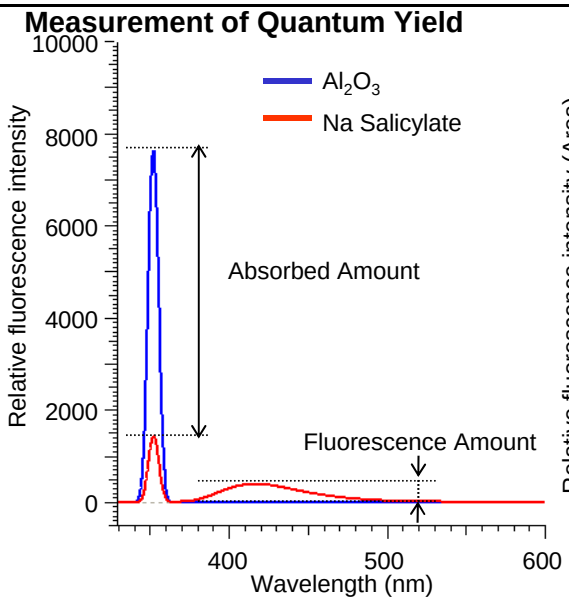


Figure 5 Quantum Yield Measurement of Na Salicylate

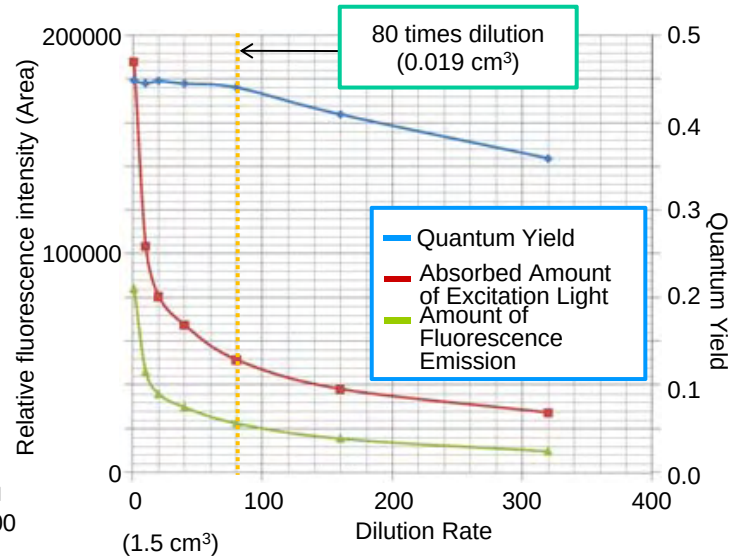


Figure 6 Study of Na Salicylate Dilution with Standard Cell

Table 2 Quantum Yield Measurement Result for Na Salicylate

Dilution rate	1	10	20	40	80	160	320
Internal quantum yield	0.449	0.445	0.448	0.445	0.441	0.410	0.360
Sample amount (cm^3)	1.5	0.15	0.075	0.038	0.019	0.009	0.005

Hitachi F-7000 Fluorophotometer

KEY WORDS

Material/Processing Material Related, Pigment/Paint/Dye, Aluminum Oxide, Phosphor, Slight Quantity, Trace Amount, Dilution, Quantum Yield, Al_2O_3, Aluminum Oxide Powder, FL, F-7000

Fluorophotometer (FL)

Sheet No. FL110010-03A