g = conversion factor, 32.2 lb. mass-ft./lb. force-sec.^{2}
h = enthalpy, B.t.u./lb. mass
Δh = difference in enthalpy between two locations, B.t.u./lb. mass
J = symbol for parameter as defined in the text
k = orifice coefficient
L = length of heated passage, ft.
M = constant of proportionality
N = number of equal-size holes in each orifice plate
n = exponent on mass velocity term or on temperature
p = pressure, lb. force/sq. ft.
Δp = difference in pressure between two locations, lb. force/sq. ft.
Q = average heat flux along heated passage, B.t.u./(hr.)(sq. ft.)
q = local heat flux, B.t.u./(hr.)(sq. ft.)
R = ratio of control valve pressure drop to fluid passage pressure drop
r = fractional permanent pressure loss across an orifice
T = temperature, °F.
v = specific volume, cu. ft./lb. mass
x = distance from passage inlet, ft.
α = coefficient in Equation (22)
ρ = density, lb. mass/cu. ft.
τ = shear stress, lb. force/sq. ft.
σ = fraction of cut-off in sine distribution
μ = viscosity, lb. mass/(ft.)(hr.)

Subscripts

a = acceleration
b = bulk
c = heated passage
e = entrance, exit
f = friction
i = inlet
max = maximum
o = outlet
t = total
v = valve or orifice
w = wall

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Light Transmittance as a Measure of Interfacial Area in Liquid-liquid Dispersions

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Interfacial area in liquid-liquid systems has been measured photographically. Precision and accuracy of the method have been shown to be better than 10%. To avoid tedium of counting drops, a simple light probe of easily reproducible design has been developed to measure the light transmission through the dispersions formed. A correlation of light transmittance with interfacial area is presented and its usefulness and limitations are discussed.

Frequently workers concerned with dispersions of immiscible liquids desire knowledge of the interfacial area of these systems. Unfortunately it is generally difficult or impossible to determine the interfacial area of liquid-liquid dispersions. In the few cases where interfacial areas have been determined, most investigators have relied on photographic techniques.

In recent years the phenomenon of light scattering by small particles has been the subject of a number of investigations (*14*); however, very little work has been reported in the literature concerning light scattering as a measure of particle size in liquid-liquid dispersions. Undoubtedly a major deterrent to such investigations has been the problem of measuring the interfacial area of rapidly coalescing disperse systems by other than photographic methods.

In this paper a photographic technique for measuring interfacial area is described.

Also, an empirical correlation based on theoretical considerations is presented which gives the light transmittance of liquid-liquid dispersions as a function of the interfacial area. Experimental data are presented for several nonabsorbing (water-white) liquid pairs of differing relative refractive indexes.

PREVIOUS WORK

Most of the interest in light scattering has been restricted to very dilute monodisperse* systems of small particles generally less than 1 μ in diameter. Mathematical treatments of concentrated polydisperse† systems are not available. In addition, there are few quantitative data available relative to liquid-liquid or gas-liquid dispersions.

It is interesting to note that in 1925 Stamm and Svedberg (*19*) were interested in the relationship between the right-angle scattering of light and the size distribution of soap-stabilized benzene-water emulsions. They suggested a linear relationship between light transmittance and concentration for a given particle size. Clark and Blackman (*3*) investigated light transmittance as a measure of the interfacial area of foams. On the basis of admittedly qualitative data, a linear relationship between light transmittance and volumetric area was suggested.

Recently Langlois and coworkers (*11, 12*) published a correlation of light transmission with interfacial area for liquid-liquid systems. Their results were correlated empirically by a linear equation in the following form:

$$\frac{I_0}{I} = 1 + \beta A \qquad (1)$$

where

I_0/I = the ratio of incident to emergent light intensity
A = interfacial area per unit volume of total mixed phases (volumetric area)
β = a constant characterizing a specific liquid pair

This relationship was considered to be independent of the volume-fraction dispersed phase. The constant β was given as a function of the relative refractive index of the dispersed phase.

The papers by Langlois are of particular interest because they represent the first successful attempt to establish a quantitative relationship between light transmission and volumetric area. However, no provisions were made for variation of the optical path length, and each light probe required individual calibration because the reproducibility of results with different probes of the same design was poor (*10, 15*). Duplication of this work would be difficult.

It is apparent from the literature that the theoretical equations relating particle size and concentration to light transmittance are limited and cannot be applied to concentrated polydisperse systems of large spherical particles. The available empirical relationships for polydisperse systems do not demonstrate satisfactory accuracy and precision. In addition, it is difficult to reproduce the equipment design, and each light-detector unit requires individual calibration.

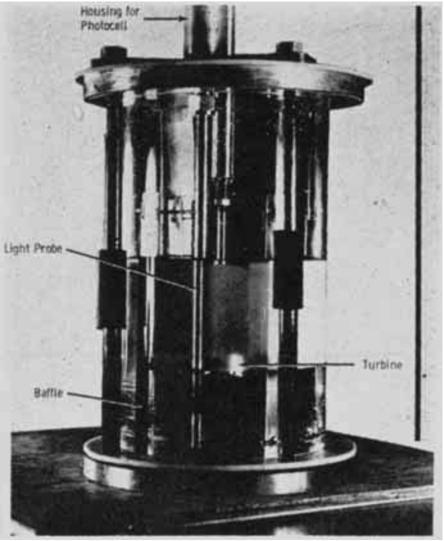

Fig. 1. Mixing vessel.

THEORY

Basic Theory

The light transmitted through a polydisperse system of transparent isotropic spherical particles provides a measure of the interfacial area if it can be demonstrated that light transmission is a function of the total projected area. This means that it must be shown that the light transmission is a function of the mean surface volume or sauter diameter d_{sv}. Conversely a correlation is not possible if the scattered light energy is sensitive to the particle-size distribution. For a polydisperse system consisting of N_i particles of diameter d_i in a given size interval

$$d_{sv} = \frac{\sum N_i d_i^{\,3}}{\sum N_i d_i^{\,2}} \qquad (2)$$

For a volume fraction of dispersed phase of ϕ the volumetric area is defined as follows:

$$A = \frac{6}{d_{sv}}\phi \qquad (3)$$

The factors which control light transmittance are embodied in the familiar extinction or Beer-Lambert relationship:

$$Sn\,dL = \frac{dI}{I} \qquad (4)$$

The scattering cross section S is the ratio of the light energy lost by scattering to the incident light energy per unit area and n is the number of particles per unit volume. Integrating over the distance between light source and detector L, as the incident light diminishes from I_0 to I, yields the conventional extinction equation

$$\ln\frac{I_0}{I} = SnL = K_s\frac{d^2\pi}{4}nL \qquad (5)$$

where

$$K_s = \frac{4S}{d^2\pi} \qquad (6)$$

d = particle diameter

Experimental data and calculations by several investigators (*2, 5* to *9, 18*) based on theory developed by Mie (*13*) show that the scattering area coefficient K_s is extremely sensitive to particle sizes near the wave length of the incident light. The effect of particle diameter, however, decreases with increasing particle size. Calculations by Boll and coworkers (*2*) show that for the limiting conditions of this investigation K_s is essentially constant for particles larger than 50 μ.

In this investigation the observed particle-size distribution has generally been between 50 and 2,000 μ and the scattering area coefficient may be considered as a constant with respect to particle diameter. Therefore, the drop diameter d may be defined in terms of the sauter diameter d_{sv}, and Equation (5) may be written in terms of the interfacial area per unit volume of total mixed phases A and a scattering factor α.

$$\ln\frac{I_0}{I} = \frac{K_s}{4}\pi d^2 nL = \frac{K_s}{4}\pi d^2\left(\frac{6N\phi}{N\pi d^3}\right)L = \alpha AL \qquad (7)$$

For a given incident-light wave length the scattering factor α should be a unique function of the relative refractive index of the liquid pair.

Applied Theory

In the basic relationship, Equation (4), it is assumed that light once scattered from the path of the primary beam is lost. This is true only for very dilute dispersions. For the systems studied, however, light scattered from the primary beam may be returned to the original path by secondary scattering. Hence multiple scattering is an important factor which

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*Monodisperse in the sense that all particles in the dispersion are the same size.

†Polydisperse in the sense that a distribution of particle size is present.

must be considered. The effect of multiple scattering is a function of both the relative refractive index and the incident-light-beam diameter.

Other factors which may be considered to have a significant effect on the transmittance relationship are functions of the light source and detector geometry. As an example the problem of measuring the emergent light intensity I may be considered. The incident light I_0 is a collimated beam. The emergent light I is defined as that portion of I_0 which passes unimpaired through L cm. of dispersion. This means that the emergent light is that portion of the incident light scattered in the forward direction parallel to the path of the primary beam. Gumprecht and Sliepcevich (*4*) point out that it is technically impossible to obtain an exact measure of the emergent light. Any detector design will measure, in addition to light scattered in the forward direction, some of the light scattered at small angles from the path of the primary beam.

The transmission relationship [Equation (7)] may be modified to conform with these practical considerations:

$$\ln \frac{I_0}{I} = f(\alpha, A, L) \qquad (8)$$

Equation (8) shows that an exponential relationship exists between light transmittance and some function of α, A, and L. The exact nature of the function must be determined experimentally.

EXPERIMENTAL

Experimental techniques and equipment were developed to measure the interfacial area per unit volume of total mixed phases (volumetric area) and the light transmittance of the dispersion at a specified optical path. The volumetric area was determined photographically, and the light transmittance by photoelectric measurements.

Mixing Equipment

Dispersions were created in 5 9/16-in.-diam. Pyrex mixing vessel shown in Figure 1. The vessel was equipped with four baffles, each one tenth of the diameter of the mixing vessel in width, and a 3-in., six-blade flat-blade turbine. The baffles and agitator were constructed to conform with the design suggestions of Rushton *et al.* (*17*). All metallic components were of 304 stainless steel; gaskets were of Teflon.

Light Source and Detector

The photoelectric equipment required for light-transmittance measurements consisted of three major components: a light source to provide a uniform, collimated beam; a sensitive light-detector unit; and an electronic circuit to measure the amplified output of the detector unit. In the design of the equipment ruggedness and reproducibility were of prime importance. Of secondary concern was the desire to mount as much of the equipment as possible external to the mixing vessel to minimize contamination of the liquid and interference of the normal flow pattern.

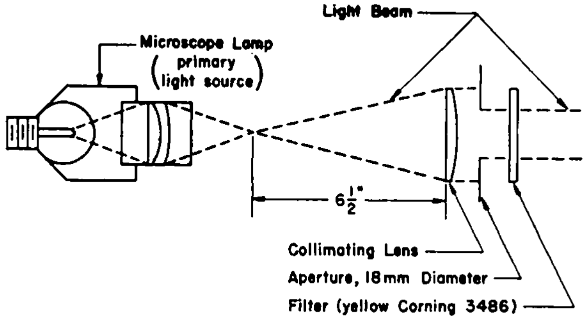

Fig. 2. Schematic diagram of light source.

Both the light source and detector were externally located. The light source was located below and the detector above the mixing vessel. A light pipe or probe extended down from the detector into the dispersion. The distance from the bottom of the vessel to the end of the light pipe determined the optical path.

The light source was a Universal Microscope Illuminator, No. 353, made by the American Optical Company and fitted with a 6-volt automobile bulb. Light from the microscope lamp converged at the focal point of a condensing lens. The collimated beam leaving the lens was restricted with an 18-mm. aperture and filtered with a Corning 3486 yellow filter. The light source is shown schematically in Figure 2.

The emergent light was transmitted to a photocell by a glass sensing probe extending into the dispersion. The probe was made adjustable to allow light paths of variable lengths through the dispersion. A 30-cm. ⅜-in.-diam. Pyrex 7740 glass rod polished on both ends served as the probe. The detector unit is shown in Figure 3.

It was found that more reproducible readings were obtained if the sides of the probe were protected from stray light by a platinum mirror finish. The mirror finish was obtained by painting the cleaned rod with a thin coat of Liquid Bright Platinum No. 05, Hanovia Chemical and Manufacturing Company, then heating to dull cherry red to produce the bright finish. The several probes made in this manner gave reproducible results. Overheating resulted in a dull finish which gave inconsistent readings.

Light was transmitted from the probe to the photocell through a seal of Dow Corning 200 silicone oil, which also lubricated the joint and prevented chipping. Black ⅜-in. rubber tubing covered the junction to prevent stray light from reaching the photocell.

An end-type photoelectric cell, RCA 1P42, was used to give a better optical seal than that obtainable from side-entry types. A vacuum tube was used in preference to a gas type, as the linearity of response was better. The tube was sensitive in the visible range, allowing liquids with interfering colors to be detected. The 1P42 tube, together with the yellow filter, responded in the light band of 560 ± 50 mμ.

The output of the photocell was amplified and measured by balancing against a constant voltage with a calibrated helipot resistance. As the photocell had a linear response, the resistance required was a measure of the light energy reaching the photocell and was used in calculations without conversion as ratios of light energy are involved.

Volumetric Area

The volumetric area which exists in a liquid-liquid system during agitation was determined from photographs of the dispersion. Photographs were taken through the base of the mixing vessel with a 35-mm. Leica camera fitted with an Ektar 5-cm. lens, reflex prism, and close-range bellows using Kodak Plus-X film. Illumination was provided by a 0.0002-sec. Comet repeating flash lamp mounted beside the vessel. Light from the droplets reached the camera by reflection. Pictures with good drop resolution were obtained by overexposing the film by one stop and double-developing the negative. Resolution was improved by photographing through a G filter. A typical photograph is shown in Figure 4.

Preliminary runs were made to establish techniques for area measurements in a rectangular–cross-section mixing vessel specially designed to facilitate photographic study. An assortment of glass beads dispersed in a mixture of carbon tetrachloride and tetrabromoethane was photographed. These beads ranged in diameter from 210 to 710 μ. Several observers working independently determined the interfacial area from photographs by tracing the droplets in the plane of focus and classifying the diameters.

The results of this test are presented in Table 1. They show both the accuracy and precision of the photographic method, based on measurements by several independent observers, to be better than ±5%.

PROCEDURE

The following procedure was used to obtain experimental data. Equal-volume portions of each phase were placed in the mixing vessel and equilibrated for from 2 to 4 hr. to saturate the liquid pair mutually. Organic liquids were pretreated by distillation. The aqueous phase was distilled-water or glycerin-water mixtures. Data were obtained at 25°C. Refractive indexes were measured at the sodium D line.

For each system at selected impeller speeds transmittance measurements were obtained at optical path lengths of 1, 1.5, and 2.0 cm. Photographs were taken to provide a measure of the volumetric area at the selected impeller speeds. The range of impeller speeds that could be employed was

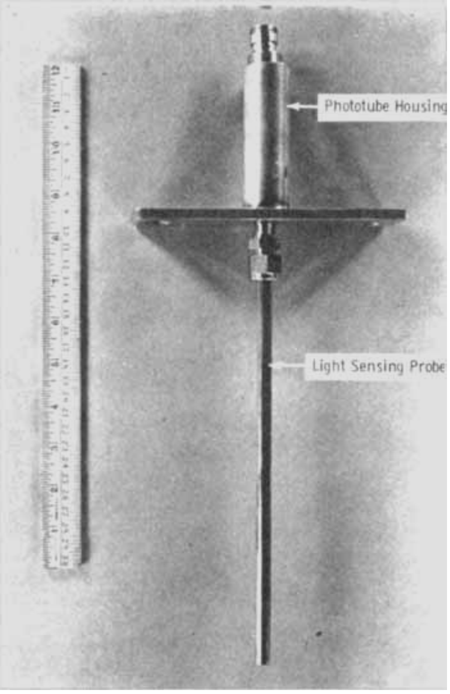

Fig. 3. Light-detector unit.

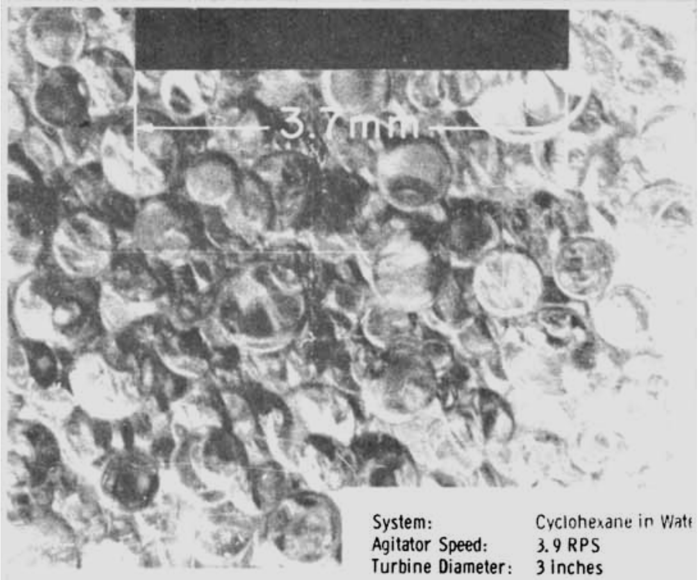

Fig. 4. Typical photograph of dispersion.

limited by the physical properties of the liquid pair. It was necessary to maintain sufficient agitation to ensure complete emulsification but not enough to beat air into the system thereby changing the optical properties. The volumetric area under a given set of conditions was found to be a reproducible function of agitator speed if surface-active contamination were absent. The presence of surface-active contamination was qualitatively indicated by the presence of surface films and by a marked change in the dispersion settling time. To minimize contamination, the equipment was thoroughly cleaned before each run.

The incident light I_0 was defined as the light energy per unit area measured through the clear-field phase before emulsification, and the emergent light I was defined as that portion of the incident light which reached the detector probe after emulsification.

The reading of the incident light was arbitrarily controlled by the voltage applied to the light source. Agitation was begun and emergent light readings were taken when steady state conditions were reached (from 2 to 45 min.), as indicated by a constant transmittance reading. Immediately after each emergent-light reading a measurement was taken to establish the reading for zero transmittance, as small changes in this quantity had considerable effect on the value obtained for the emergent light.

CORRELATION OF TRANSMITTANCE DATA

The experimental data were correlated empirically by means of a theoretically based exponential relationship:

$$\ln \frac{I_0}{I} = f(\alpha A L^{0.80}) \qquad (9)$$

The experimental data for several liquid pairs are presented in Table 2.

The experimentally determined scattering factor α is shown as a function of the relative specific refraction in Figure 5. The relative specific refraction M is defined by the following equation:

$$M = \left| \frac{m^2 - 1}{m^2 + 2} \right| \qquad (10)$$

where

m = refractive index of drop/refractive index of field phase

The scattering factor α is the optical parameter. It is primarily a measure of the effect of multiple scattering on the light transmittance as measured with the equipment employed in this investigation. For the specified light source and detector geometry, the scattering factor was found to be a complex function of the relative refractive index.

TABLE 1. GLASS-BEAD TEST FOR ACCURACY AND PRECISION OF PHOTOGRAPHIC METHOD FOR MEASURING INTERFACIAL AREA PER UNIT OF VOLUME DISPERSION

Photographically determined interfacial area

Observer	Sq. cm./cc.
A	123
B	121
C	125
D	122
E	122

Interfacial area determined by classification of glass beads

	Sq. cm./cc.
Maximum value	129
Minimum value	107
Arithmetic average value	117
Average value determined photographically	123

Size distribution of beads used in test

Diameter, μ	Wt. %
210–297	4.1
420–500	35.1
500–590	29.6
590–710	31.2

Bailey (*1*) and Rose and French (*16*), working with small solid particles less than 10 μ in diameter, obtained a satisfactory correlation by presenting their optical parameter as a function of the relative specific refraction or Lorenz-Lorentz coefficient M. In the present work the relative specific refraction proved to be a satisfactory correlating tool for polydisperse systems of large spherical particles ranging from 50 to 2,000 μ, as evidenced by the correlation.

Equally satisfactory correlations for the scattering factor may be obtained with the relative refractive index or the refractive-index difference. However, the Lorenz-Lorentz coefficient is preferable because a unique curve is obtained which should hold for values of the relative-refractive index less than 1. The relative-refractive index for water in oil dispersions is less than unity. Transmittance data for 40, 20, and 10% dispersions which included some water-in-oil data were presented in a report by Rea and Vermeulen (*15*). For purposes of comparison, these data for each volume fraction were replotted in terms of an exponential relationship. Not only were improved correlations obtained, but it was also found that the correlation of scattering factor with relative specific refraction M held for values of the relative-refractive index m both greater than and less than unity.

Unfortunately, it was not possible in this investigation to obtain data for water-in-oil systems because they proved to be unstable. Photographs of 50% dispersions of water in oil showed that they consisted of drops of water in a field of oil with small drops of oil in the larger water drops.

The generalized correlation showing light transmittance as an exponential function of a scattering factor α, the volumetric area A, and the optical path L is presented in Figure 6. The average deviation in area based on transmittance is 4.7%. The optical path length L, it will be noted, appears as a reduced power function. An exponent less than unity is required because the attenuation of the light beam in passing through the dispersion was less than would be predicted from basic light-extinction theory, which ignores the effect of multiple light scattering. This correlation presents data for nine liquid pairs. The range of relative-refractive index was 1.004 to 1.168. The range in interfacial area was from 20 to 90 sq. cm./cc.

DISCUSSION

General Observations

Perhaps the most surprising fact was the limited range of particle sizes present in any given dispersion. It was evident from the photographs that the particle size usually varied by not more than a factor of 5. In rare cases the particle size was found to vary by as much as a factor of 15. Usually the drops appeared to be perfect spheres except in cases where the drops were relatively large. It was interesting to note that systems characterized by large drops, about 1.5 mm., did not correlate quite so well as others, possibly because of deformation of the large drops.

Investigators have occasionally reported difficulty in deciding which phase is dispersed and which is continuous. It should be borne in mind that a phase present in a volume fraction greater than 0.50 may still be the dispersed phase. The dispersed phase may be detected by simply observing the settling pattern of the dispersion to determine the direction of travel of the droplets toward the interface.

The continuous phase is usually that phase which best wets the mixing vessel. For that reason the dispersions studied in this work were oil in water with the exception of the methyl isobutyl ketone–glycerin-water system where methyl isobutyl ketone was the continuous phase.

Reproducibility of Data

Consecutive transmittance readings were reproducible to within 3% and in most cases 1%. In the case of duplicate runs, with the same supply of the liquid pair, the introduction of additional variables such as substitution of probes and readjustment of the optical path length caused some loss in reproducibility. However, transmittance measurements for these duplicate runs were usually within 3% and rarely varied by as much as 5%.

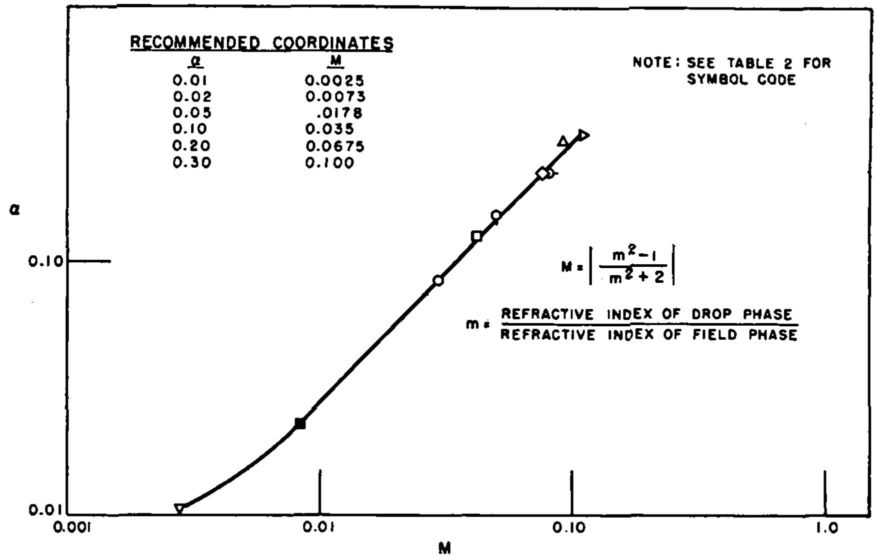

Fig. 5. Scattering factor α as a function of the relative specific refraction M for yellow-light spectrum.

Both area and transmittance data used in the correlation were obtained from measurements at the bottom of the mixing vessel. For that reason, there can be little doubt that the photographically determined areas represented the area at the point where the transmittance measurements were obtained. However, even considering the extreme degree of turbulence in these agitated systems, there might be some question concerning the homogeneity of the bulk of the dispersion, particularly near the agitator. To resolve this question a simple test was devised

TABLE 2. SUMMARY OF EXPERIMENTAL DATA

System	Symbol	Refractive index* Drop phase	Refractive index* Field phase	Volumetric area, sq.cm./cc.	ln I_0/I at specified optical path length 1.0 cm.	1.5 cm.	2.0 cm.
Bromobenzene in water	▷	1.5575	1.3335	33.2	4.17	5.02	
Monochlorobenzene in water	△	1.5221	1.3330	22.5	3.28	3.95	
				27.7	3.62	4.34	
Benzene in water	o-	1.4975	1.3332	22.2	2.73	3.33	3.82
				28.5	3.20	3.84	4.32
				35.6	3.56	4.20	4.65
Xylene in water	◇	1.4877	1.3330	28.8	3.04	3.72	4.25
				34.0	3.57	4.21	4.72
				40.2	3.98	4.59	5.19
Kerosene in water	♀	1.4338	1.3330	49.6	3.49	4.17	4.82
				56.7	3.81	4.60	5.03
				71.5	4.33	5.01	5.62
Cyclohexane in water	□	1.4181	1.3331	48.3	2.95	3.71	4.27
				52.6	3.28	3.93	4.53
				64.0	3.64	4.26	4.95
				73.3	3.88	4.52	5.35
Methyl isobutyl ketone in water	○	1.3942	1.3349	39.1	2.19	2.66	3.04
				50.6	2.53	3.02	3.45
				62.1	2.87	3.40	3.83
60% Glycerin water in methyl isobutyl ketone	■	1.4118	1.3942	87.1	1.49	1.84	2.22
				91.2	1.68	2.03	2.43
Cyclohexan in 60% glycerin water	▽	1.4182	1.4122	48.1	0.482	0.698	0.815

I_0/I = ratio of incident- to emergent-light intensities.
Experimental data were obtained at 25°C.
*Refractive indexes of mutually saturated liquids measured at sodium D line.

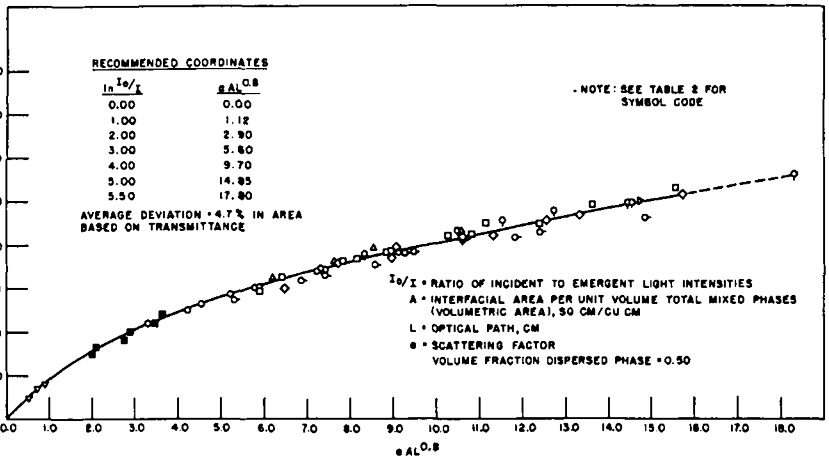

Fig. 6. General correlation of light transmittance with volumetric area for yellow-light spectrum.

which showed the dispersions to be essentially homogeneous. A portable one-piece detector-source unit consisting of phototube and flash-light bulb was placed in several different locations in the mixing vessel. Within the limits of experimental precision, no variation in light transmittance was observed, indicating essentially complete homogeneity of the dispersion.

SUMMARY

Satisfactory equipment and techniques were developed to measure the interfacial area and light transmittance of liquid-liquid dispersions. Both the accuracy and precision of the photographic technique for measuring interfacial area were shown to be better than 10%. A light detector and source were designed which featured readily available component parts and permitted variation of the optical path length.

The over-all reproducibility of transmittance measurements, with substitution of components, readjustment of optical path length, and realignment of the light source and detector taken into account, was always within 5% and in most cases 3%.

The present work was designed to cover a useful range of relative-refractive index and to provide a sufficient quantity of experimental data to test adequately various methods of interpreting these data. The experimental data were correlated by means of an exponential relationship based on a combination of theoretical and applied light-scattering considerations. Attempts to develop a linear correlation were not successful.

The general correlation is valid only for the conditions of this investigation. The physical conditions are the specified light source, light detector, and incident-light spectrum. Other conditions, as defined by the experimental data and the restrictions imposed by the underlying theoretical and practical considerations, are

1. Liquids which are transparent in the incident-light spectrum.
2. Dispersions consisting of 50% by volume dispersed phase.
3. Systems where the relative specific refraction M is between 0.01 and 0.11.
4. Dispersions in which the smallest particle size present is greater than 50 μ.
5. Dispersions through which the percentage of light transmittance ($I/I_0 \times 100$) is greater than 0.65% for optical path lengths from 1.0 to 2.0 cm.

The results of this investigation show the combined effects of multiple scattering and equipment geometry on light transmittance. First considerably more light reached the detector than may be predicted from light-extinction equations, which do not take into account multiple scattering. Second, the attenuation of the incident light passing through these dispersions was less than may be predicted, as evidenced by the fact that optical path length entered the correlation as a reduced power function.

The correlation with an average deviation of less than 5% shows that the ability of nonabsorbing liquid-liquid dispersions to transmit light is of value as a method of estimating the amount of interfacial area present. Additional work is necessary, however, before this technique can be regarded as a standard service tool. Some criteria of absorbency, other than simply nonabsorbing liquids, must be established. Work in progress with 25 and 10% dispersions and with a light spectrum in the near infrared region indicates that similar correlations may be developed for these conditions.

ACKNOWLEDGMENT

The authors wish to acknowledge with gratitude the contributions of L. F. Dorsey, who obtained most of the experimental data, and to thank D. K. Fleming and J. B. West for their valuable criticism of the contents of this paper, G. Lindholm of the Graphic Arts Section for the development of photographic techniques, and various members of the Chemical Engineering Division of Argonne National Laboratory for technical assistance in the design of the experimental apparatus.

NOTATION

A = interfacial area per unit volume of total mixed phases (volumetric area), sq. cm./cc.
d = drop diameter, cm.
d_i = drop diameter in interval i, cm.
d_{sv} = mean surface-volume or sauter drop diameter, cm.
I_0 = incident-light intensity
I = emergent-light intensity
K_s = scattering-area coefficient
L = optical path length, cm.
M = relative specific refraction of drop (Lorenz-Lorentz coefficient)
m = relative-refractive index of drop
N_i = number of drops in interval i
n = number of drops per unit volume of total mixed phases, cc.$^{-1}$
S = scattering cross section, sq. cm.
α = scattering factor (optical parameter)
β = optical parameter
ϕ = volume fraction of dispersed phase

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Presented at A.I.Ch.E. Detroit Meeting