attempt to prepare the Diels-Alder adduct of compound III with tetracyanoethylene failed. Apparently, an unstable $\pi$-complex did form however, since the reaction mixture assumed a persistent coloration, first red then brownish yellow, but only starting materials could be isolated. Recrystallization from ethanol gave the purified adduct (V) as fine, white needles, m.p. 227-229 ${ }^{\circ}$ (uncorr.). Anal. Calcd. for $\mathrm{C}_{12} \mathrm{H}_{12} \mathrm{O}_{4}$ : C, 65.45; H, 5.49; O, 29.06. Found: C, 65.47; H, 5.36; O, 28.87.

The infrared spectrum (see paragraph on infrared spectrum in section above) (potassium bromide wafer, 4 mg . per 200 mg .) exhibited $\mathrm{C}=\mathrm{C}$ absorption at 6.08 (w), the cyclic ether band at 9.25 (s), as well as the components of the cyclic anhydride grouping characterized (2) by the broad, very strong absorptions of the carbonyl (doublet) at 5.40 to 5.65 , and the $\mathrm{C}-\mathrm{O}-\mathrm{C}$ linkage centered at 8.20 .

NMR data ( $10 \%$ in trifluoroacetic acid) showed these peaks: triplet at $3.47,3.56$, and 3.64 ( 2 vinyl protons); a multiplet between 5.74 and 6.43 (4 protons ortho to oxygen); a broad peak at 6.56 ( 4 protons at bridgehead and anhydride ring); and a multiplet at 7.18 ( 2 angular protons).

## cis-2-OXAHEXAHYDROINDANE (cis-HEXAHYDROPHTHALAN)

A. From the Hydrogenation of Diolefin III. One gram of III in 1 ml . of ethanol was shaken with 100 mg . of $5 \%$ palladized charcoal catalyst for 3 hours at room temperature under 25 p.s.i.g. hydrogen pressure. Two equivalents of hydrogen were taken up. The catalyst was removed by filtration, washed with ethanol, and the combined filtrate and washings were fractionated. One gram of compound IV was obtained, b.p. $51^{\circ}\left(8 \mathrm{~mm}\right.$. of Hg ) $, n_{\mathrm{D}}^{21} 1.4701, n_{\mathrm{D}}^{25} 1.4683$, $\mathrm{d}_{4}^{25} 0.9724$ [lit. $(4,5)$ b.p. $59^{\circ}(9 \mathrm{~mm}$. of Hg$), 179^{\circ}(740 \mathrm{~mm}$. of Hg$\left.), n_{\mathrm{D}}^{21} 1.4712, n_{\mathrm{D}}^{25} 1.4683, \mathrm{~d}_{4}^{25} 0.9727\right]$.
B. From the Hydrogenation of Monoolefin I. Ten grams of compound I, prepared by the method of Eliel and Pillar (3) b.p. $66.0-66.5^{\circ}\left(13.5 \mathrm{~mm}\right.$. of Hg ), $n_{\mathrm{D}}^{25} 1.4890$ [lit. (3) b.p. $63-64^{\circ}(13 \mathrm{~mm}$. of Hg$\left.), n_{\mathrm{D}}^{25} 1.4884\right]$, in 5 ml . of ethanol were hydrogenated in the same manner described for compound III; one equivalent of hydrogen was absorbed. Nine grams of IV were recovered, b.p. $51.5^{\circ}$ ( 9 mm . of Hg ), $n_{\mathrm{D}}^{26} 1.4680, \mathrm{~d}_{4}^{25} 0.9733$.

The infrared spectra (neat, 0.0212 mm .) of the product derived from method $A$ and method $B$ were superimposable and corresponded with that of a published spectrum (4). Also, GLC analysis ( $1 / 4 \times 120$ inch column packed with $15 \%$ terephthalic acid-terminated Carbowax 20 M supported on 60 to 80 -mesh Chromosorb W) of a mixture of the two products gave a single peak.

## ACKNOWLEDGMENT

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# Refractive Indices of Some Ternary Systems Containing Naphthalene 

## Binary Solvent Mixtures Among Benzene, Tolvene, Ethylbenzene, Carbon Tetrachloride

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#### Abstract

Refractive indices have been determined at $25^{\circ} \mathrm{C}$. for the ternary systems of naphthalene dissolved in the six solvent pairs from among benzene, toluene,ethylbenzene, and carbon tetrachloride; and for the various binary subsystems. Equations are given relating refractive index to solution composition in mole fraction. The average deviations in the ternary systems, from predicted refractive indices by these equations, range from 2 to $13 \times 10^{-5}$ units among the systems. Only in the ethylbenzene-toluene-naphthalene and ethylbenzene-benzene-naphthalene ternary systems are deviations randomly distributed.


Analysis, when performed using physical properties dependent upon the concentrations of all system components, grows more complex as the number of components increases. Graphical procedures, commonly used in relating a property to composition in a binary system, are generally not applicable in multicomponent systems. Analytical functions, although often impractical in hand calculations, may be conveniently used in analysis when
computer facilities are available. The present work illustrates the latter approach in representing the refractive indices, $n$, of some ternary systems containing naphthalene.

## EXPERIMENTAL DATA

Equipment and procedure in determination of $n$, as well as the purity and $n$ of the purified reagents, have been
described in a previous study of naphthalene solubilities (3). The $n$ value of liquid components differs from literature values by from 4 to $14 \times 10^{-5}$ units. All $n$ values in this study refer to $25.00 \pm 0.01^{\circ} \mathrm{C}$. and to the sodium D-line, 5893 A.

Binary system data are given in Table I. In liquid-solid binaries, the data extend to within 0.03 mole fraction of saturation. Ternary system data are given in Table II. More $n$ were observed in a system (usually upward of 30 ) than the 20 that have been listed for each. Twenty points were considered adequate to furnish an indication of the behavior in each system. All data are from the region quite removed from the binaries. The points represent the extent of the concentration range covered, and they have been selected to include the values showing the greatest error in predicted $n$, as described below. Each system contains several points within a few hundredths mole fraction of naphthalene saturation.

## REPRESENTATION OF DATA BY ANALYTICAL FUNCTIONS

Scatchard and Ticknor (6) have shown that in the ternary methanol-carbon tetrachloride-benzene system, $n$ is expressible as a quadratic in component volume fractions. As one of the components in the present work is a solid, the volume fraction relation cannot be used because the
molal volume of supercooled naphthalene is not precisely known. Thus, mole fraction has been adopted despite $a$ priori evidence (6) that this approach is less suitable.

It is proposed here that $n$ of the binary systems be represented by

$$
\begin{equation*}
n=n_{1} x_{1}+n_{2} x_{2}+x_{1} x_{2} A_{12} \tag{1}
\end{equation*}
$$

where $n_{i}$ and $x_{i}$ are the refractive index and mole fraction, respectively, of pure component $i . A_{12}$ is a concentration dependent function defined by

$$
\begin{equation*}
A_{12}=B_{12}+C_{12}\left(x_{1}-x_{2}\right)+D_{12}\left(x_{1}-x_{2}\right)^{2} \tag{2}
\end{equation*}
$$

where $B_{12}, C_{12}, D_{12}$ are empirical constants evaluated from experimental $n$ data. The constants of Equation 2 for the liquid-liquid binaries are listed in Table III.

Measured values of supercooled naphthalene $n$ are not available. Estimation by atomic refractions is not possible because of the optical exaltation of naphthalene (2). Estimation by combining the accepted temperature coefficient of $n$ (1) with $n$ above the naphthalene melting point was rejected because of the work of Ward and Van Winkle (9). Their results indicate that this procedure may lead to considerable error.

For the ethylbenzene-naphthalene system, as interaction is small (3), $n$ should vary linearly with volume fraction (6).

| System | Table I. Refractive Indices of Binary Systems at $25^{\circ} \mathrm{C}$. |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
|  | Mole Fraction of First-named Exptl. Component Refr. Index | System | Mole Fraction of First-named Component | Exptl. <br> Refr. Index |
| Benzene-toluene | $0.7567 \quad 1.49634$ | Naphthalene-benzene | 0.2844 | 1.54312 |
|  | $0.6066 \quad 1.49570$ |  | 0.2638 | 1.54006 |
|  | $0.4457 \quad 1.49516$ |  | 0.2450 | 1.53736 |
|  | $0.2364 \quad 1.49454$ |  | 0.2377 | 1.53628 |
|  | By Eq 1:Std. Dev. $10^{5}=1$, |  | 0.2285 | 1.53502 |
|  | Av. Dev. $10^{5}=1$ |  | 0.2243 | 1.53446 |
|  |  |  | 0.2127 | 1.53279 |
| Benzene-ethylbenzene | $0.8273 \quad 1.49619$ |  | 0.1561 | 1.52409 |
|  | $0.5877 \quad 1.49487$ |  | 0.0985 | 1.51472 |
|  | $0.4501 \quad 1.49428$ |  | 0.0489 | 1.50629 |
|  | $0.1835 \quad 1.49349$ |  | By Eq. 1:Std. | v. $10^{5}=7$, |
|  | By Eq. 1:Std. Dev. $10^{5}=2$, <br> Av. Dev. $10^{5}=2$ |  | Av. Dev. $10^{5}=6$ |  |
|  |  | Naphthalene-toluene | 0.2884 | 1.53601 |
|  |  | Naphthalenetoluene | 0.2660 | 1.53294 |
| Benzene-carbon tetrachloride | 0.82971 .49086 |  | 0.2589 | 1.53197 |
|  | $0.6391 \quad 1.48323$ |  | 0.2511 | 1.53104 |
|  | $0.3684 \quad 1.47229$ |  | 0.2278 | 1.52767 |
|  | 0.28451 .46885 |  | 0.2129 | 1.52556 |
|  | By Eq. 1:Std. Dev. $10^{5}=0$, |  | 0.1408 | 1.51518 |
|  | Av. Dev. $10^{5}=0$ |  | 0.0978 | 1.50882 |
|  |  |  | 0.0474 | 1.50118 |
| Toluene-ethylbenzene | 0.8230 1.49384 <br> 0.6112 1.49364 |  | $\text { By Eq. } 1: \mathrm{Std} .$ | $\text { ev. } 10^{5}=5$ |
|  | 0.6165 1.49349 | Naphthalene-ethylbenzene | Av. Dev. $10^{\circ}=$ |  |
|  | $0.1801 \quad 1.49325$ |  | 0.2622 | 1.52806 |
|  | $\begin{aligned} & \text { By Eq. } 1: \text { Std. Dev. } 10^{5}=0, \\ & \text { Av. Dev. } 10^{5}=0 \end{aligned}$ |  | 0.2449 | 1.52579 |
|  |  |  | 0.2338 | 1.52430 |
|  |  |  | 0.2163 | 1.52198 |
| Toluene-carbon tetrachloride | $0.7735 \quad 1.48718$ |  | 0.1830 | 1.51752 |
|  | $0.5867 \quad 1.48105$ |  | 0.1237 | 1.50962 |
|  | $0.4297 \quad 1.47536$ |  | 0.0657 | 1.50197 |
|  | $0.2476 \quad 1.46816$ |  | By Eq. 1:Std. | ev. $10^{5}=3$, |
|  | By Eq. 1:Std. Dev. $10^{5}=1$, |  | Av. Dev. $10^{5}=$ |  |
|  | Av. Dev. $10^{5}=1$ | Naphthalene-carbon tetrachloride | 0.2395 | 1.50770 |
| Ethylbenzene-carbon tetrachloride |  |  | 0.2289 | 1.50538 |
|  | $0.8150 \quad 1.48825$ |  | 0.2231 | 1.50444 |
|  | 0.6358 1.48297 |  | 0.2165 | 1.50321 |
|  | $0.4112 \quad 1.47533$ |  | 0.2010 | 1.49992 |
|  | $0.2201 \quad 1.46769$ |  | 0.1879 | 1.49745 |
|  | $\begin{aligned} & \text { By Eq. } 1 \text { :Std. Dev. } 10^{5}=0 \text {, } \\ & \text { Av. Dev. } 10^{5}=0 \end{aligned}$ |  | 0.0791 | 1.47337 |
|  |  |  | By Eq. 1:Std. <br> Av. Dev. $10^{5}$ | $\operatorname{lev} \cdot 10^{5}=12$ |

Table II. Refractive Indices of Ternary Systems at $25^{\circ} \mathrm{C}$.


Std. Dev. 105: 11 (Eq. 3a), 24(Eq. 3b)
Av. Dev. 10 ${ }^{5}$ : 9 (Eq. 3a), 19(Eq. 3b)

Std. Dev. $10^{5}: 10$ (Eq. 3a), 30(Eq. 3b)
Av. Dev. $10^{5}: 8$ (Eq. 3a), 20 (Eq. 3b)

Moreover the molar volume of dissolved naphthalene is 123 ml . at $20^{\circ} \mathrm{C}$. (4), and that of ethylbenzene is 123.06 ml . at $25^{\circ} \mathrm{C}$. (5). Linearity of $n$ with mole fraction should therefore also be obtained. A plot of $n$ vs mole fraction is precisely linear from 0 to 0.2662 mole fraction naphthalene in this system, the latter concentration being near saturation. Accordingly, the value of supercooled naphthalene $n$ at $25^{\circ} \mathrm{C}$. assigned here is that obtained by extrapolation of this plot to pure supercooled naphthalene, 1.62615. On this
basis, $B_{12}, C_{12}, D_{12}$ values for the naphthalene-solvent systems are also included in Table III. The standard and average deviations of the experimental $n$ from those predicted with Equation 1 are listed in Table I.

As the first approximation to ternary behavior, the equation

$$
\begin{equation*}
n=n_{1} x_{1}+n_{2} x_{2}+n_{3} x_{3}+x_{1} x_{2} A_{12}+x_{1} x_{3} A_{13}+x_{2} x_{3} A_{23} \tag{3}
\end{equation*}
$$

is proposed. Differences between experimental $n$ and those

Table III. Binary System Constants

| System | Constants of Equation 2 ( $x_{1}$ is First-named Component) |  |  |
| :---: | :---: | :---: | :---: |
|  | $B_{12}$ | $C_{12}$ | $D_{12}$ |
| Benzene-toluene | -0.00260 | -0.00029 | -0.00112 |
| Benzene-ethylbenzene | -0.00397 | -0.00131 | -0.00137 |
| Benzene-carbon tetrachloride | 0.00029 | 0.00013 | -0.00124 |
| Toluene-ethylbenzene | -0.00056 | -0.00032 | -0.00123 |
| Toluene-carbon tetrachloride | 0.00877 | -0.00125 | -0.00165 |
| Ethylbenzene-carbon tetrachloride | 0.01291 | -0.00256 | -0.00039 |
| Naphthalene-benzene | 0.03063 | -0.03571 | -0.01649 |
| Naphthalene-toluene | 0.00669 | -0.04113 | -0.03218 |
| Naphthalene-ethylbenzene | 0 | 0 | 0 |
| Naphthalene-carbon tetrachloride | -0.03188 | -0.29878 | -0.25816 |

calculated with Equation 3 are considerable. The system showing the smallest differences is given in Figure 1. The differences range to a maximum of $50 \times 10^{-4}$ units in the systems containing carbon tetrachloride. In the latter, the differences show considerable liquid-liquid asymmetry. To fit the differences, Equation 3 has been expanded to an Equation 3a, which includes a ternary deviation function, $x_{1} x_{2} x_{3} A_{123}$, where

$$
\begin{equation*}
A_{123}=B_{123}+C_{123} x_{1}+D_{123} x_{2}+E_{123} x_{1}^{2}+F_{123} x_{2}^{2}+G_{123} x_{1} x_{2} \tag{4}
\end{equation*}
$$

In addition, an Equation 3 b has been considered, in which the fifth order term coefficients of Equation 4 ( $E_{123}, F_{123}$, $\left.G_{123}\right)$ are assigned a zero value. Table II lists the standard and average deviations of the experimental $n$ from those predicted with Equations 3a and 3b. Constants used in these equations are given in Table IV.

As there is greater interaction between carbon tetrachloride and naphthalene than between the latter and the other solvents (3), Equation 3 was modified for the systems containing carbon tetrachloride by an approach described by Scatchard and coworkers (7). This amounts to replacement in the ternary system of the quantity $\left(x_{i}-x_{j}\right)$ by $\left(2 x_{i}-1\right)$ where $x_{i}$ refers to the component whose property is in considerable contrast to those of the other two components. Results by this approach were in greater error than those obtained using Equation 3 itself.

Computations in the present work were programmed for a digital computer. The most complex aspect, evaluation of constants in the $n$ vs. composition equations, involved obtaining solution matrices through inversion of those matrices formed in least-squares treatment of the experimental $n$ and composition data pairs.


Figure 1. $10^{4}$ (exptl. $n-n$ by Equation 3 ) in the system ethylbenzene-benzene-naphthalene

## DISCUSSION

Differences between experimental and calculated $n$ are uniformly small in the binary liquid-liquid systems, approximating the inherent instrument limitation, about $\pm 3 \times 10^{-5}$ units. In the binary systems containing naphthalene, the differences are somewhat greater. The greatest difference in any single experimental binary $n$ is $19 \times 10^{-5}$ units.
An apparent difference in behavior between the liquidliquid and solid-liquid binaries-indicated by the contrast in Table III of $C_{12} / B_{12}$ that are small for the former and large for the latter-should be disregarded. This is the result of fitting the data over a restricted concentration range in the latter systems.
In the ternary systems, $n$ calculated with Equation 3 are systematically below the experimental values in all present systems. The extent of the disagreement reflects the difference in $n_{i}$ of the pure liquids.
In the present systems, Equations $3 a$ and $3 b$ appear to lead to significant results in representing the data. Errors in applying these equations in a given system show a comparable systematic trend, however, except in the benzene- and toluene-ethylbenzene mixed solvent systems, where the errors appear to be random. Both the average and extreme differences in the other systems are greater by Equation 3b than by 3a. With the former and latter, respectively, the greatest differences in any single ternary system are 60 and $40 \times 10^{-5}$ units.

No consideration to equations other than those proposed above has been given in the present work. Others may be more suitable. Thus it appears probable that an empirical expression for $n$ in terms of a power series in $x_{i}$ and $x_{j}$ might be obtainable. In fact, Equation 1 for binary systems reduces to such a form, with no cross terms in $x_{1} x_{2}$. Power series have been used for this purpose in binary systems. A quadratic expression in solute concentration is sufficient over a considerable concentration range even for some systems of electrolytes in water (8). However, combination of the pertinent quadratics for the binaries into a suitable form

Table IV. Ternary System Constants ${ }^{a}$

| System | Equation 3a |  |  |  |  |  | Equation 3b |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | $B_{123}$ | $C_{123}$ | $D_{123}$ | $E_{123}$ | $F_{123}$ | $G_{123}$ | $B_{12}$ | $\mathrm{C}_{123}$ | $D_{123}$ |
| Benzene-toluene-naphthalene | 0.8075 | -1.7337 | -1.6898 | 0.9412 | 0.8877 | 1.9111 | 0.1586 | -0.1528 | -0.1481 |
| Benzene-ethylbenzene-naphthalene | 0.2778 | -0.6442 | -0.5718 | 0.3956 | 0.3209 | 0.6672 | 0.0374 | -0.0323 | -0.0208 |
| Benzene-carbon tetrachloridenaphthalene | 2.0742 | -3.7785 | -4.4542 | 1.8343 | 2.3967 | 4.1429 | 0.7044 | -0.5899 | -0.8406 |
| Toluene-ethylbenzene-naphthalene | 0.2067 | -0.4174 | -0.3710 | 0.2138 | 0.1940 | 0.3879 | 0.0672 | -0.0735 | -0.0435 |
| Toluene-carbon tetrachloridenaphthalene | 1.8879 | -3.3782 | -4.1546 | 1.5647 | 2.2498 | 3.8422 | 0.5987 | -0.5015 | -0.7303 |
| Ethylbenzene-carbon tetra-chloride-naphthalene | 2.2320 | -4.2015 | -5.0901 | 2.0583 | 2.8616 | 4.8559 | 0.5799 | -0.4939 | -0.7346 |

${ }^{a}$ In applying these constants in Equations 3a or $3 b$ to a system, subscripts $1,2,3$ refer to the components in the order listed in Column 1 of this table.
for the resultant ternaries was not possible. The comparative magnitudes of $B_{123}, C_{123}$, etc. in Table IV, where only the deviations from Equation 3 were to be fitted, indicate that a rather large number of terms might be needed in an effective power series for these ternary systems.

The present approach to an analytical function for the ternary data would be quite unwieldy without the use of a computer. For example, the determination of an unknown composition by locating the intersection of two functions of the form of Equation 3a or 3b for two different physical properties would require extensive iterative procedures.

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## NOMENCLATURE

$A_{1}=$ function defined in Equation 2
$A_{t: k}=$ function defined in Equation 4
$B_{1}, C_{n}, D_{0}=$ constants in Equation 2
$B_{i k}, C_{i k}, D_{i k}, E_{i j}, F_{i, j} G_{i, k}=$ constants in Equation 4 $n=$ refractive index $n_{i}=$ refractive index of pure component $i$ $x_{i}=$ mole fraction of component $i$

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# Derivatives of 1-Benzoyl-1,2-dihydroquinaldic Acid (Reissert Acid) 

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$\mathrm{R}_{\mathrm{B}}$REISSERT ACID (1-benzoyl-1,2-dihydroquinaldic acid) was prepared by the method of Collins and Henshall (1), during an investigation of the chemistry of Reissert compounds. Two derivatives of this acid were preparednamely, methyl 1-benzoyl-1,2-dihydroquinaldate (I) and $d$ - $\alpha$-methylbenzylammonium 1 -benzoyl-1,2-dihydroquinaldate (II).


The methyl ester, I, was obtained as a crystalline solid by treating a methanol solution of 1-benzoyl-1,2-dihydroquinaldic acid with an excess of diazomethane in ether. The crystalline ammonium salt, II, was obtained by the reaction of 1 -benzoyl-1,2-dihydroquinaldic acid with $d$ - $\alpha$ phenylethylamine in anhydrous acetone.

## EXPERIMENTAL

Elemental analyses were carried out by Schwarzkopf Microanalytical Laboratory, Woodside, N.Y. All melting points are corrected.

Methyl 1-Benzoyl-1,2-dihydroquinaldate. To a solution of 3.0 grams ( 0.01 mole) of 1 -benzoyl-1,2-dihydroquinaldic acid in 25 ml . of absolute methanol cooled in an ice bath,
an excess, as noted by the yellow color of the solution, of an ice cold ether solution of freshly prepared diazomethane was added slowly (2). When the evolution of nitrogen had ceased, the solution was warmed to room temperature and the solvents distilled, the latter portion under reduced pressure. This gave 2.1 grams $(67.1 \%)$ of a light yellow solid; m.p. $115-118^{\circ} \mathrm{C}$. Recrystallization from methanol gave a white crystalline solid, m.p. $118-119^{\circ} \mathrm{C}$.
Anal. Calcd. for $\mathrm{C}_{18} \mathrm{H}_{15} \mathrm{NO}_{8}: \mathrm{C}, 73.72 ; \mathrm{H}, 5.15 ; \mathrm{N}, 4.77$. Found: C, 73.82; H, 5.33; N, 5.00.
d- $\alpha$-Methylbenzylammonium 1-Benzyol-1,2-dihydroquinaldate. A solution of $0.5 \mathrm{gram}(0.0017 \mathrm{~mole})$ of 1 -benzoyl1,2 -dihydroquinaldic acid in 5 ml . of absolute acetone was mixed with 0.21 gram ( 0.0017 mole ) of $d-\alpha$-phenylethylamine (3). The solution was warmed on a steam bath for one to two minutes and cooled. In approximately one-half hour, a white solid precipitated from solution. This was filtered and washed with acetone, m.p. 168-169.6 ${ }^{\circ} \mathrm{C}$.

Anal. Calcd. for $\mathrm{C}_{25} \mathrm{H}_{24} \mathrm{~N}_{3} \mathrm{O}_{3}$ : C, $75.03 ; \mathrm{H}, 6.04 ; \mathrm{N}, 7.00$. Found: C, 75.15 ; H, 6.06; N, 7.24 .

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