

SOLVENTS AND SOLUTES FOR THE PREPARATION OF IMMERSION LIQUIDS OF HIGH INDEX OF REFRACTION*

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ABSTRACT

The types of compounds that should be suitable as solvents and solutes for the preparation of immersion liquids of high index of refraction are covalent inorganic, organic, and metal-organic compounds containing the nonmetallic elements of the carbon, nitrogen, oxygen, and fluorine groups of the periodic table, and mercury and thallium. Many of these compounds have already been used to make immersion liquids of high index of refraction. Other compounds of these types that might be suitable are suggested.

Most of the developmental work on immersion liquids of high index of refraction has generally been either one of trial and error or an extension of an earlier experimenter's work. The purpose of this paper is to define the types of inorganic and organic compounds that are most likely to be suitable as solvents and solutes in the preparation of liquids of high index of refraction. Practical considerations such as solubility, stability, and toxicity will limit the use of individual compounds and small groups of compounds although their indices of refraction may be high. Déverin (1934) has listed 12 extensive groups of organic compounds which he considered fertile fields for investigation because these groups of compounds would tend to have relatively high indices of refraction.

Liquids of high index of refraction will generally be composed of substances that have high indices of refraction. In the preparation of these liquids the solvent should be a liquid of relatively high index of refraction, or a solid of high index of refraction whose melting point is very close to room temperature, so that if one dissolves some solute in it, the freezing point will be depressed below room temperature and a liquid will result. Of all the elements, white phosphorus (melting point, 44° C.) is the only one that can and has been used as a solvent to prepare a liquid of high index of refraction. (For references, see Meyrowitz, 1955.)

The low-melting inorganic compounds will generally be covalent or nonpolar. Covalent compounds are those in which the bonds holding the atoms together are formed by the sharing of electrons. Covalent compounds are not electrical conductors in the liquid state, either when pure or dissolved in a suitable solvent (Glasstone, 1946, p. 66), and they have low melting and boiling points because the external fields of force which

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exist between the molecules in the solid state are small (Glasstone, 1946, p. 67). They are also generally soluble in organic compounds.

Covalent inorganic compounds are usually composed of elements whose electronegativities are of the same magnitude. Electronegativity is defined as the power of an atom in a molecule to attract electrons to itself (Pauling, 1940, p. 58). As the difference in electronegativity of the atoms in a molecule decreases, the molecule becomes more covalent. Stable compounds composed of atoms having electronegativity values of the same magnitude are found primarily among the nonmetallic group of atoms (the carbon, nitrogen, oxygen, and fluorine groups of the periodic table).

Compounds that have both low melting points (close to room temperature) and high indices of refraction are generally composed of these elements. Examples are (1) arsenic tribromide, AsBr_3 , melting point 33°C ., (2) selenium monobromide, Se_2Br_2 , liquid at room temperature, and (3) stannic bromide, SnBr_4 , melting point 31°C . Two of these compounds, namely arsenic tribromide and selenium monobromide, have been used as solvents to prepare immersion liquids of high index of refraction. (For references, see Meyrowitz, 1955.)

Organic compounds are generally covalent, and very many organic liquids are good solvents for covalent solids. The range of the indices of refraction of organic liquids, however, is 1.30 to 1.80 and of organic solids 1.3 to 2.5 (Weissberger, 1949, p. 1143). Relatively few of the thousands of organic liquids have indices of refraction higher than 1.74. Similarly most of the organic solids have relatively low indices of refraction. From the following empirical relations, one can define the type of organic compound that would have a high index of refraction and probably be suitable as a solvent.

The index of refraction of a compound depends upon (*a*) the elements present, (*b*) their number, and (*c*) the manner in which they are combined. The homologous series of compounds in Table 1 shows that as one element in the compound is replaced by another the index of refraction changes. The homologous series of compounds in Table 2 shows that as the number of atoms of any one element present changes, the index of refraction changes. If the replacement in a compound of one element by another results in an increase in the index of refraction, generally the addition of more atoms of the element (where possible) will increase the index of refraction. The series of compounds in Table 3, each series having the same chemical composition, show that the index of refraction also depends on the manner in which the elements are combined.

TABLE 1. SERIES OF COMPOUNDS SHOWING THE CHANGE IN INDEX OF REFRACTION AS ONE ELEMENT IN A COMPOUND IS REPLACED BY ANOTHER

Compound	Formula	n_D	Reference
Arsenic trihydride	AsH ₃	gas	Lange, 1949, p. 186
Arsenic trichloride	AsCl ₃	1.62	Hodgman, 1950, p. 456
Arsenic tribromide	AsBr ₃	1.78 (24° C.)	Borgström, 1929, p. 59
Arsenic triiodide	AsI ₃	solid	Lange, 1949, p. 186
Phosphorus trihydride	PH ₃	gas	Lange, 1949, p. 248.
Phosphorus trichloride	PCl ₃	1.52	Lange, 1949, p. 1012
Phosphorus tribromide	PBr ₃	1.70	Lange, 1949, p. 1012
Phosphorus triiodide	PI ₃	solid	Lange, 1949, p. 250
Phosphorus trichloride	PCl ₃	1.52	Lange, 1949, p. 1012
Arsenic trichloride	AsCl ₃	1.62	Hodgman, 1950, p. 456
Antimony trichloride	SbCl ₃	solid	Lange, 1949, p. 184
Methane	CH ₄	gas	Lange, 1949, p. 561
Methylene chloride	CH ₂ Cl ₂	1.42	Lange, 1949, p. 1007
Methylene bromide	CH ₂ Br ₂	1.55	Timmermans, 1950, p. 216
Methylene iodide	CH ₂ I ₂	1.74	Lange, 1949, p. 1007
Benzene	C ₆ H ₆	1.50	Lange, 1949, p. 943
Chlorobenzene	C ₆ H ₅ Cl	1.53	Lange, 1949, p. 949
Bromobenzene	C ₆ H ₅ Br	1.56	Lange, 1949, p. 945
Iodobenzene	C ₆ H ₅ I	1.62	Lange, 1949, p. 989
Naphthalene	C ₁₀ H ₈	1.58 (98° C.)	Lange, 1949, p. 1007
α -Chloronaphthalene	C ₁₀ H ₇ Cl	1.63	Lange, 1949, p. 950
α -Bromonaphthalene	C ₁₀ H ₇ Br	1.66	Lange, 1949, p. 945
α -Iodonaphthalene	C ₁₀ H ₇ I	1.71	Lange, 1949, p. 989
Tetramethyl silicon	(CH ₃) ₄ Si	1.359	Beilstein, 1929, p. 579
Tetramethyl germanium	(CH ₃) ₄ Ge	1.387	Beilstein, 1942, p. 1008
Tetramethyl tin	(CH ₃) ₄ Sn	1.520	Beilstein, 1922, p. 632
Tetramethyl lead	(CH ₃) ₄ Pb	1.512	Heilbron and Bunbury, 1943, p. 502
Tetraethyl silicon	(C ₂ H ₅) ₄ Si	1.425	Beilstein, 1929, p. 580
Tetraethyl germanium	(C ₂ H ₅) ₄ Ge	1.44	Beilstein, 1942, p. 1008
Tetraethyl tin	(C ₂ H ₅) ₄ Sn	1.514	Beilstein, 1922, p. 632
Tetraethyl lead	(C ₂ H ₅) ₄ Pb	1.514	Beilstein, 1922, p. 638

TABLE 2. SERIES OF COMPOUNDS SHOWING CHANGE IN INDEX OF REFRACTION AS ELEMENT NUMBER CHANGES

Compound	Formula	n_D	Reference
Benzene	C_6H_6	1.50	Lange, 1949, p. 943
Chlorobenzene	C_6H_5Cl	1.53	Lange, 1949, p. 949
<i>o</i> -Dichlorobenzene	$C_6H_4Cl_2$	1.55	Lange, 1949, p. 954
Trichlorobenzene	$C_6H_3Cl_3$	1.57	Lange, 1949, p. 1020
Diethyl sulfide	$(C_2H_5)_2S$	1.44	Mumford and Phillips, 1950, p. 80
2:2' Dichlorodiethyl sulfide	$(C_2H_4Cl)_2S$	1.53	Mumford and Phillips, 1950, p. 80
1:1:2:2:2:2' Hexachlorodiethyl sulfide	$(C_2H_2Cl_3)_2S$	1.57	Mumford and Phillips, 1950, p. 80

The particular elements present in a compound have a much greater effect on the index of refraction of the compound than the manner in which these elements are combined. The increase in index of refraction

TABLE 3. INDICES OF REFRACTION OF SEVERAL SERIES OF COMPOUNDS, THE COMPOUNDS IN EACH SERIES HAVING THE SAME CHEMICAL COMPOSITION

Compound	Formula	n_D	Reference
Dipropargyl	C_6H_6	1.44	Lange, 1949, p. 969
Benzene	C_6H_6	1.50	Lange, 1949, p. 943
tert-Butyl chloride	C_4H_9Cl	1.387	Lange, 1949, p. 946
sec-Butyl chloride	C_4H_9Cl	1.395	Lange, 1949, p. 946
iso-Butyl chloride	C_4H_9Cl	1.397	Lange, 1949, p. 946
<i>n</i> -Butyl chloride	C_4H_9Cl	1.402	Lange, 1949, p. 946
<i>p</i> -Dibromobenzene	$C_6H_4Br_2$	1.574	Lange, 1949, p. 953
<i>m</i> -Dibromobenzene	$C_6H_4Br_2$	1.608	Lange, 1949, p. 953
<i>o</i> -Dibromobenzene	$C_6H_4Br_2$	1.612	Lange, 1949, p. 953
Bromopropylene (2)	C_3H_5Br	1.447	Lange, 1949, p. 945
Bromopropylene (α)	C_3H_5Br	1.455	Lange, 1949, p. 945
Allyl bromide	C_3H_5Br	1.514	Lange, 1949, p. 940
Propionic acid	$C_3H_6O_2$	1.387	Lange, 1949, p. 1013
Methyl acetate	$C_3H_6O_2$	1.359	Lange, 1949, p. 992
Ethyl formate	$C_3H_6O_2$	1.360	Lange, 1949, p. 977
Acetol	$C_3H_6O_2$	1.430	Lange, 1949, p. 938

that is obtained by replacing one element of a compound by another suitable one is usually relatively large (a few units in the first decimal place, Table 1) as compared to the increase obtained by changing the manner in which the elements of a compound are combined (a few units in the second decimal place, Table 3). Inasmuch as it is desirable to use compounds having the highest practical indices of refraction in the preparation of immersion liquids of high index of refraction and inasmuch as changes in molecular structure affect the index of refraction to a relatively small extent, the discussion of changes in molecular structure as they affect the index of refraction will be omitted.

A metal-organic compound will generally have a much higher index of refraction than the organic compound from which it is derived. (See Table 1.)

If an element in any given compound is replaced by a denser element that belongs to the same group in the periodic table, there will usually be an increase in the index of refraction of the new compound as compared to the previous one. Increases in density, melting point, and boiling point generally accompany the increase in index of refraction. However, the advantage of an increase in index of refraction which results from the replacement of one element by another may be of no value in the search for a suitable solvent, because the melting point of the new compound may be considerably higher than room temperature. This is illustrated in inorganic compounds by the following series of compounds:

Compound	Formula	n_D	Reference	Melting point, ° C.	Reference
Arsenic trichloride	AsCl ₃	1.62	Hodgman, 1950, p. 456	-18	Lange, 1949, p. 187
Arsenic tribromide	AsBr ₃	1.78	Borgström, 1929, p. 59	31	Lange, 1949, p. 187
Arsenic triiodide	AsI ₃	$\omega=2.59$ $\epsilon=2.23$	Lange, 1949, p. 899	146	Lange, 1949, p. 187
Phosphorus tribromide	PBr ₃	1.70	Lange, 1949, p. 1012	-40	Lange, 1949, p. 249
Arsenic tribromide	AsBr ₃	1.78	Borgström, 1929, p. 59	31	Lange, 1949, p. 187
Antimony tribromide	SbBr ₃	— ¹	Slawson, 1922, p. 175	96.6	Lange, 1949, p. 185

¹ "... the indices of refraction were considerably higher than that of the liquid (1.74)."

and in organic compounds by these series of compounds:

Compound	Formula	n_D	Reference	Melting point, ° C.	Reference	Boiling point, ° C.	Reference
Chloroform	CHCl ₃	1.46	Lange, 1949, p. 950	-63.5	Lange, 1949, p. 427		
Bromoform	CHBr ₃	1.60	Lange, 1949, p. 945	8-9	Lange, 1949, p. 405		
Iodoform	CHI ₃	$\omega = 2.11$ $\epsilon = 1.77$ 1.57	West, 1937, p. 463-464 Beilstein, 1942, p. 979	119	Lange, 1949, p. 553		
Dichloromethylarsine	CH ₃ AsCl ₂	1.57	Beilstein, 1942, p. 979	— ¹	Beilstein, 1942, p. 979	133 (760 mm.)	Beilstein, 1942, p. 979
Dichloromethylstibine	CH ₃ SbCl ₂	— ¹		— ²	Friend, 1936, p. 184	115-120 (60 mm.)	Friend, 1936, p. 184
Dichloromethylbismuthine	CH ₃ BiCl ₂	— ¹		242	Friend, 1936, p. 269-270		

¹ Not available.

² Does not solidify at approximately 0° C.

Although arsenic triiodide, antimony tribromide, iodoform, and dichloromethylbismuthine have or should have high indices of refraction, they could not be used as solvents because their melting points are much higher than room temperature. With these counterbalancing factors of increase in index of refraction and increase in melting point in mind, one can say that the types of organic compounds that would be suitable as solvents for the preparation of immersion liquids of high index of refraction would be organic or metal-organic compounds containing arsenic in preference to bismuth, sulfur in preference to tellurium, and bromine in preference to iodine. These preferences would tend to insure a low-melting compound. The organic part of the compound should be as small as possible so that the highest possible index of refraction be obtained.

Organic and metal-organic compounds that have been used as solvents (for reference, see Meyrowitz, 1955) are:

1. α -Bromonaphthalene, C₁₀H₇Br
2. Cacodyl selenide, [(CH₃)₂As]₂Se
3. Carbon disulfide, CS₂
4. Diethylselenium, (C₂H₅)₂Se
5. Dimethylmercury, (CH₃)₂Hg
6. α -Iodonaphthalene, C₁₀H₇I
7. Methylene iodide, CH₂I₂
8. Phenyl-di-iodoarsine, C₆H₅AsI₂

It has been shown that the compounds, both inorganic and organic, that would be suitable for use as solvents would tend to be essentially covalent. Nonpolar or covalent solvents generally dissolve only nonpolar or covalent solutes. Solid compounds (as distinguished from low-melting solids) that are essentially covalent, that is, compounds containing weakly polar parts, would tend to be soluble in covalent inorganic, organic, and metal-organic compounds.

Some free elements are soluble in covalent solvents. They are found among the nonmetallic elements. The elements that have already been used as solutes (for references, see Meyrowitz, 1955) are:

1. Arsenic
2. Iodine
3. Phosphorus
4. Selenium
5. Sulfur

Although tellurium tends to be metallic, it might be possible to find a solvent containing one or more substances in which appreciable amounts of the brown amorphous form of tellurium would be soluble.

The covalent inorganic compounds will usually be composed of the nonmetallic elements (the carbon, nitrogen, oxygen, and fluorine groups of the periodic table). Mercury and thallium should be included among these elements because they can form covalent compounds (Partington, 1950, p. 770, 805).

The inorganic compounds that have already been used as solutes (for references, see Meyrowitz, 1955) are:

1. Antimony tribromide, SbBr_3
2. Antimony triiodide, SbI_3
3. Antimony trisulfide, Sb_2S_3
4. Arsenic disulfide, As_2S_2
5. Arsenic triiodide, AsI_3
6. Arsenic trisulfide, As_2S_3
7. Mercuric iodide, HgI_2
8. Mercuric sulfide, HgS
9. Stannic iodide, SnI_4

The types of organic compounds that would be suitable as solutes will be similar to those that would be suitable as solvents except that the inorganic and halogen portion of the compounds would be increased as much as possible and the organic portion of the molecule kept as small as possible. The limiting factor in the consideration of the compounds suitable for use as solvents is the melting point. Any change in the composition of a compound made in order to increase its index of refraction is to no advantage if the melting point of the new compound is much

above room temperature. The limiting factor for the solutes (inorganic as well as organic) is its solubility in the solvent.

As the melting points increase in a homologous series of compounds in which an element is replaced by another in the same group of the periodic table, there will generally be a decrease in the covalent properties of the compounds. This will usually result in a decrease in the solubility of the compounds in covalent solvents. The advantage of an increase in index of refraction will be of no practical value, if by replacing one element of a compound by another, we decrease the covalent character of the compound and so decrease to a large extent the solubility of the compound.

Organic compounds that have been used as solutes are:

1. Iodoform, CHI_3 .

2. Tetraiodoethylene, C_2I_4 (for references, see Meyrowitz, 1955). It will be noticed that the halogen fraction of the total mass of each of these two compounds is relatively large compared to the organic fraction.

The use of these types of covalent compounds which should be suitable as solvents and solutes for the preparation of immersion liquids of high index of refraction will be circumscribed by their instability, toxicity, and reactivity to the immersed phase.

It would be useful in the consideration of compounds for possible use as solvents and solutes for the preparation of immersion liquids of high index of refraction to have available their indices of refraction. Much of these data is not readily available, for a search of the chemical literature (especially that previous to the last 20 to 30 years) will reveal that the indices of refraction of relatively few organic and metal-organic solid compounds are recorded. The indices of refraction of many more liquid compounds than of solid compounds are given in the literature.

The index of refraction of a compound can be calculated by means of the Lorentz-Lorenz equation

$$R_D = \frac{n_D^2 - 1}{n_D^2 + 2} \cdot \frac{M}{d}$$

where (R_D) is the molar refraction for the D line of sodium, n_D is the index of refraction for the D line of sodium, (M) is the molecular weight, and (d) is the density; (n_D) and (d) are measured at the same temperature.

The molar refraction of compounds is usually not available, whereas the density is frequently available. However, the molar refraction of a compound can be calculated using the values for bond refractions, and in turn the index of refraction can be calculated with a reasonable degree of accuracy from the molecular refraction and density data.

The molar refraction is a physical property which is additive as well as constitutive, that is to say, the molar refraction of a molecule depends

not only upon the number and kind of atoms present, but also upon the particular arrangement of the atoms within the molecule. The molar refraction of a compound can be considered as the sum of the refractions of the atoms in the molecule. To each of the component atoms is assigned a definite part of this total refraction, and this part is referred to as the atomic refraction. However, inasmuch as the atomic refraction depends on the manner in which the atom is held in the molecule, it is more accurate to divide the molar refraction into bond refractions or electron groups refractions.

Although there is often no exact additivity of the bond refractions,

TABLE 4. EMPIRICAL AND CALCULATED INDICES OF REFRACTION OF SOME CHEMICAL COMPOUNDS

Compound	Formula	<i>M</i>	<i>R_D</i>	<i>d</i>	* C.	Empirical <i>n_D</i>	* C.	Calculated <i>n_D</i>	Melting point ° C.
1-Chloro-5-iodopentane	C ₅ H ₁₀ ClI	232.50	43.06	1.6789 ¹	25	1.5297 ¹	25	1.5252	
Dimethyl tin diiodide	Sn(CH ₃) ₂ I ₂	402.59	54.22	2.8582 ²	39.3	1.6906 ²	39.3	1.7137	
Germane, tetramethylsulfide	Ge(SCH ₃) ₄	261.00	66.63	1.4364 ³	25	1.6379 ³	25	1.6544	
Iodoform	CHI ₃	393.78	45.51	4.008 ⁴	20	2.00*		1.89	119 ⁴
Pentabromoethane	C ₂ HBr ₅	424.61	49.92	3.312 ⁵		—		1.71	56–57 ⁵
1-Chloro-1,1,2,2-tetrabromoethane	C ₂ HBr ₄ Cl	380.15	47.04	3.366 ⁵	16	—		1.77	32–33 ⁵

$$* \bar{n} \text{ (mean) calculated using } \bar{n} = \frac{2\omega + \epsilon}{2} = 2.00 \left\{ \begin{array}{l} n_{\omega} = 2.11 \\ n_{\epsilon} = 1.77 \end{array} \right\}^{\frac{1}{2}}$$

$$\bar{n} = \sqrt[3]{\omega^2 \epsilon} = 1.99$$

¹ Huber, 1951, p. 2731.

² Friend, 1928, p. 371.

³ Johnson, 1951, p. 292–293.

⁴ Hodgman, 1950, p. 949.

⁵ Beilstein, 1918, p. 95.

⁶ West, 1937, p. 463–464.

the deviations are so small as to be negligible in the application of these principles to the estimation of the order of magnitude of the index of refraction of a compound. A more detailed discussion of the Lorentz-Lorenz molar refraction, its additivity, and its limitations is given in Weissberger (1949, p. 1157–1177).

The molar refraction (*R_D*), density (*d*), index of refraction (empirical), and index of refraction (calculated using the Lorentz-Lorenz formula) of six compounds are listed in Table 4. The molar refraction was calculated using values for the bond refractions as given by Vogel et al. (1950) and Cresswell et al. (1953).

The agreement between the empirical and calculated indices of refraction of the first four compounds is relatively good. The last two com-

pounds, if stable, should be good solvents for they resemble bromoform and acetylene tetrabromide. Their indices of refraction as calculated by the Lorentz-Lorenz formula are relatively high, and their melting points are not very much above room temperature.

Other compounds that might be suitable as solvents and solutes are listed below.

Compound	Formula	Melting point ° C.	Reference
Phosphorus sesquisulfide	P_4S_3	172	Hodgman, 1950, p. 539
Phosphorus pentasulfide	$P_2S_5(P_4S_{10})$	276	Hodgman, 1950, p. 539
Stannic bromide	$SnBr_4$	31	Hodgman, 1950, p. 587
Methyltintribromide	$Sn(CH_3)Br_3$	50-55	Friend, 1928, p. 312
Methyltintriiodide	$Sn(CH_3)I_3$	82-85	Friend, 1928, p. 312
Diphenyltin dibromide	$Sn(C_6H_5)_2Br_2$	38	Friend, 1928, p. 324
Diiodo-p-iodophenylarsine	$(IC_6H_4)AsI_2$	80	Friend, 1930, p. 120
Bisdimethylstibine trisulfide	$[(CH_3)_2SbS]_2S$	76-78	Friend, 1936, p. 187
Diiodotriisobutylstibine	$(C_4H_9)_3SbI_2$	70	Friend, 1936, p. 289
Diphenylselenium disulfide	$(C_6H_5)_2SeS_2$	50-51	Friend, 1937, p. 29

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