

3.3. Measurement of refractive index*

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3.3.1. Introduction

The optical properties of crystals are complex, and it is planned to include a full account in Volume D. What follows is restricted to a brief description of immersion media for use in the measurement of indices of refraction. **WARNING. Many of the media, particularly those of high refractive index, are poisonous, or corrosive, or both.**

3.3.2. Media for general use

The immersion media listed in Table 3.3.2.1 are easily prepared, stable, and venerated satisfactory. They were selected because they require only a small number of liquids for their preparation. In general, each liquid is miscible in the liquids with higher and lower indices of refraction so that any intermediate mixture can be easily prepared.

The liquids up to $n = 1.770$ are measured on an Abbe refractometer; those with higher values for n are measured in a hollow glass prism prepared from selected object glasses and mounted on a goniometer or on a spectrometer (Butler, 1933; Larsen & Berman, 1934, pp. 18–20).

A set of immersion liquids with indices of refraction differing by one unit in the second decimal place (1.510, 1.520, 1.530, ...) is used for routine work. The liquids are best kept in 15 ml dropping bottles with plastic caps and glass dropping rods. These bottles are more satisfactory than the more expensive dropping bottles with solid glass stoppers and ground-glass caps because there is less trouble with the stopper cementing to the bottle. The bottle should be about half full.

Some crystals dissolve rapidly in the liquids tested. A measurement can usually be made by performing the reading rapidly. If the crystal and the liquid have nearly the same indices of refraction, the index of the liquid is not much changed by the solution of the crystal.

* Editorial condensation of the entry by E. S. Larsen Jr and R. Meyrowitz in Volume III (*IT* III, 1962).

Table 3.3.2.1. *Immersion media for general use in the measurement of index of refraction*

Note: Further lists are given by Hartshorne & Stuart (1960).

	$n_D^{20^\circ\text{C}}$	Temperature coefficient (dn/dT)	Dispersion
Water	1.333	1×10^{-4}	Slight
Glycerol	1.473	2.2×10^{-4}	Slight
<i>n</i> -Octane	1.400	4.8×10^{-4}	–
<i>n</i> -Hexadecane	1.434	3.8×10^{-4}	Slight
Kerosene (Paraffin)	1.448	3.5×10^{-4}	Slight
Petroleum oil (Nujol)	1.477	4×10^{-4}	Slight
α -Chloronaphthalene	1.626	4×10^{-4}	Moderate
Methylene iodide	1.740	6.4×10^{-4}	Rather strong
Methylene iodide saturated with sulfur	1.778	6×10^{-4}	Rather strong
AsBr ₃ plus 10% sulfur (mix with methylene iodide or α -bromonaphthalene for lower n)	1.814 (25°C)	7×10^{-4}	Rather strong
2S, 2As ₂ S ₂ , 6AsBr ₃ (mix with 10% sulfur in AsBr ₃ for lower n)	2.003 (25°C)	6×10^{-4}	Rather strong
2Se, 2As ₂ S ₂ , 6AsBr ₃ (mix with 10% sulfur in AsBr ₃ for lower n)	2.11 (25°C)	6×10^{-4}	Rather strong

Table 3.3.4.1. *Aqueous solutions for use as immersion media for organic crystals*

Salt	$n_D^{20^\circ\text{C}}$ of saturated solution
Lithium iodide	1.490
Sodium iodide	1.496
Potassium iodide	1.456
Barium iodide	1.528
Tetrasodium dioxypentathioannate	1.615

Table 3.3.4.2. *Organic immersion media for use with organic crystals of low solubility*

Compound	$n_D^{20^\circ\text{C}}$
Diethyl oxalate	1.41
Di- <i>n</i> -butyl carbonate	1.41
Triethyl citrate	1.44
Tri- <i>n</i> -butyl citrate	1.44
<i>n</i> -Butyl phthalate	1.49
α -Bromonaphthalene	1.66
α -Iodonaphthalene	1.70
Methylene iodide	1.74

3.3.3. High-index media

Refractive indices greater than 2.1 present special difficulties. Merwin & Larsen (1912) used melts of sulfur and selenium, satisfactory up to index 2.72. Mixtures of selenium and As₂Se₃ can be used up to index 3.17 (Larsen & Berman, 1934). Above about 2.2, the index must be determined at a wavelength for

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which selenium is transparent; the red line of lithium is convenient.

3.3.4. Media for organic substances

Organic substances are usually soluble in organic liquids, but not usually in aqueous solutions. The saturated solutions listed in Table 3.3.4.1 (Jelley, 1934, p. 245) are generally satisfactory. For substances of low solubility, the organic liquids listed in

Table 3.3.4.2 may be useful; the refractive index of diluted aqueous solutions changes with time because of evaporation.

If the index of refraction is greater than about 1.7, the media at the end of Table 3.3.2.1 have to be used. These usually dissolve organic crystals, so that the immersion liquid becomes saturated with the compound. The refractive index of the saturated medium is then measured with a microrefractometer (Jelley, 1934, pp. 236–240).