A NEW SERIES OF IMMERSION LIQUIDS*[a]

ROBERT MEYROWITZ

ABSTRACT

A new series of high-index immersion liquids (1.66–1.81) has been made. α-bromonaphthalene and a solution of precipitated sulfur (10 per cent) in arsenic tribromide are the end members. The mixing curve is not a straight line. After nine months the maximum change in index was −0.001. Stable liquids containing α-bromonaphthalene, arsenic tribromide, precipitated sulfur, and arsenic disulfide are discussed.

INTRODUCTION

A study of inorganic, organic, and metal-organic compounds that might be employed in the preparation of liquids of high refractive index is in progress at the U. S. Geological Survey. It is part of a program of research in the geochemistry of uranium that the Geological Survey is undertaking on behalf of the Atomic Energy Commission.

In a previous paper (Meyrowitz and Larsen, 1951) describing immersion liquids of high refractive index, a series of liquids containing methylene iodide, arsenic tribromide, and precipitated sulfur was introduced. The indices of refraction of this series range from 1.74 to 1.81. It was reported in that paper that these liquids darkened on standing because of the decomposition of methylene iodide. Nevertheless their indices of refraction have remained nearly constant for 15 months. As the decomposition seemed to be continuing, work was begun immediately on the development of a new set of immersion liquids in the range of 1.74 to 1.81, not containing methylene iodide. It was found that α-bromonaphthalene could be substituted for methylene iodide in these liquids.

The liquids made of α-bromonaphthalene and a 10 per cent solution of precipitated sulfur in arsenic tribromide have been used for nine months. They have shown no decomposition, and their indices of refraction have remained nearly constant. They may replace the methylene iodide liquids.

The new liquids are light yellow and after a period of nine months the maximum change in index was −0.001. They are stored in glass bottles with ground-glass stopper and a ground-glass dust cover. The 1.80, 1.81, and 1.82 liquids may become solid if the temperature at which they are stored falls much below that of normal room temperature (20 to 25° C). Gentle warming will effect complete solution and after

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thorough mixing the clear liquid will have its original refractive index.

The temperature coefficient (dn/dT) and the dispersion as measured by the difference in index between the "e" line of mercury (546) and the "D" line of sodium (589) are as follows:

	dn/dT	*Dispersion*
α-bromonaphthalene	4.8×10^{-4}	76×10^{-4}
	(Larsen and Berman, 1934)	
10 per cent sulfur in arsenic tribromide	6×10^{-4}	114×10^{-4}

Preparation of Immersion Liquids

Reagents

(1) α-bromonapthalene, Eastman Organic Chemical no. 46, supplied by Distillation Products Industries, Rochester 3, N. Y.
(2) Sulfur, precipitated, U.S.P.
(3) Arsenic tribromide, C. P., supplied by A. D. Mackay, Inc., 198 Broadway, New York 7, N. Y.

The end members are:

(1) α-bromonaphthalene, $n_{Na}^{25^\circ C}=1.655$
(2) 10 per cent sulfur in arsenic tribromide, $n_{Na}^{25^\circ C}=1.814$.

The sulfur and arsenic tribromide (10 to 90 by weight) are mixed in a glass-stoppered Erlenmeyer flask, and solution is effected by gentle warming. After standing at room temperature overnight, the solution is filtered by suction through a sintered glass of "medium" porosity.

10 per cent S in $AsBr_3$ (in grams)	α-bromonaphthalene (in grams)	$n_{Na}^{25^\circ C}$
28	272	1.662
69	231	1.673
88	212	1.679
118	182	1.690
142	158	1.700
163	137	1.710
184	116	1.721
200	100	1.729
216	84	1.739
233	67	1.752
244	56	1.760
256	44	1.769
268	32	1.780
276	24	1.788
287	13	1.799
296	4	1.809

TABLE 1. CHANGE IN REFRACTIVE INDEX OF LIQUIDS CONTAINING α-BROMONAPHTHALENE, ARSENIC TRIBROMIDE, SULFUR, AND ARSENIC DISULFIDE AFTER A PERIOD OF NINE MONTHS

Number	$n_{Na}^{25°\,C.}$ (initial)	$n_{Na}^{25°\,C.}$ (after nine months)	$\Delta n \times 10^{-3}$ (9 months)	$n_{Na}^{25°\,C.}$ of high end member ($AsBr_3-S-AsS$)
1	1.666	1.665	−1	1.924
2	1.672	1.672	0	1.924
3	1.683	1.683	0	1.924
4	1.692	1.691	−1	1.924
5	1.706	1.705	−1	1.924
6	1.719	1.718	−1	1.924
7	1.738	1.737	−1	1.924
8	1.750	1.749	−1	1.924
9	1.773	1.771	−2	1.924
10	1.792	1.791	−1	1.924
11	1.822	1.820	−2	1.924
12	1.848	1.846	−2	1.924
13	1.887	1.885	−2	1.982
14	1.923	1.921	−2	1.982
15	1.938	1.937	−1	1.982
16	1.968	1.966	−2	1.982
17	1.868	1.866	−2	1.999
18	1.897	1.896	−1	1.999
19	1.957	1.956	−1	1.999
20	1.979	1.977	−2	2.027
21	2.003	1.998	−5	2.027
22	2.018	2.015	−3	2.027

The filtrate is passed through the same filter until it is no longer turbid.

The mixing curve obtained with the two end members deviates considerably from a straight line, and the curve may be constructed from the table at the foot of the preceding page.

The necessary amounts by weight of the high and low end members are mixed together in glass-stoppered bottles or Erlenmeyer flasks to obtain the series of liquids of desired intervals. The refractive indices of the liquids are determined after thorough mixing.

Experiments indicate that a discontinuous series of liquids containing α-bromonaphthalene, arsenic tribromide, arsenic disulfide, and precipitated sulfur can be made. These liquids range in index from 1.66 2.02. This discontinuous series of liquids can probably be made up of two continuous series of liquids with α-bromonaphthalene as the lower

end member and two solutions both containing arsenic tribromide, sulfur, and arsenic disulfide in different proportions as high end members.

High end members with indices of 1.922, 1.936, 1.945, and 1.956 were used to make liquids with a refractive index of 1.660 that have remained stable for six months. If stable liquids at the low end of the series can be made, then it should be possible to produce a stable continuous series of liquids, as demonstrated by liquids 1 to 12 in Table 1. Stable liquids of refractive index 1.66 could not be prepared by adding α-bromonaphthalene to liquids of very high refractive index ($n = 1.982$ and 1.999) containing arsenic tribromide, sulfur, and arsenic disulfide.

The gap between 1.95 and 2.02 has been filled by using high end members with refractive indices of 1.999 and 2.027. Although a series from 1.66 to 2.02 can be made by using three high end members, the author is attempting to develop two high end members from which the entire series from 1.66 to 2.02 can be produced.

The stability of the liquids containing α-bromonaphthalene, arsenic tribromide, sulfur, and arsenic disulfide is shown in Table 1.

References

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