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ON THE USE OF STANDARD GLASS POWDERS IN REFRACTIVE INDEX DETERMINATIONS

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The dispersion method, employed for the first time by H. E. Merwin[1] and developed* by S. Tsuboi,[2] is now used extensively. Any one who has tried this method knows how useful it is for the determination of refractive indices of minute crystals. Later the double variation method was proposed by R. C. Emmons,[3] and his apparatus is now on sale.

For a number of years the present writer has used an apparatus as shown in Fig. 1, which is a modification of Tsuboi's. It consists essentially of three thermostats containing water of different temperatures, and a system of water circulating tubes which connects the thermostats to a disk on the microscope stage. The disk is similar to that used by A. N. Winchell. The temperature on the stage can be quickly controlled by varying the temperature of the circulating water by means of a specially devised cock.

In connection with the dispersion method, or the double variation method, the Abbé refractometer is ordinarily used for determining the refractive indices of the immersion media. Accordingly, the method is not applicable to crystals having higher indices than 1.7. Also, there are certain inconveniences in the usual method.

* In spite of the opposition from G. Tunell and G. W. Morey (*Am. Mineral.*, Vol. **13**, 1932, p. 376) the present writer uses this statement, for S. Tsuboi showed for the first time how the principal refractive indices of double refracting crystals can be determined by the dispersion method. Moreover, Tsuboi proposed the use of Hartmann's dispersion net in the dispersion method, and simplified the procedure of finding graphically the refractive indices of powdered minerals for light of different wave lengths.

[1] H. E. Merwin and E. Posnjak, *J. Amer. Chem. Soc.*, Vol. **XLIV**, 1922, pp. 1965–1994.

[2] S. S. Tsuboi, *Miner. Mag.*, Vol. **18**, 1923, p. 108.
S. S. Tsuboi, *Japanese Journ. Geol. and Geogr.*, Vol. **III**, 1924, p. 19.
S. S. Tsuboi, *Journ. Geol. Soc. Tokyo*, Vol. **XXXII**, 1925, pp. 1–6.
S. S. Tsuboi, *Journ. Geol. Soc. Tokyo*, Vol. **XXXVII**, 1930, p. 39.

[3] A. N. Winchell and R. C. Emmons, *Am. Mineral.*, Vol. **XI**, 1926, p. 115.
R. C. Emmons, *Am. Mineral.*, Vol. **XIII**, 1928, p. 504 and **XIV**, 1929, p. 441.

In the first place, there is often a large difference between the temperature on the microscope stage and that of the Abbé refractometer, which makes the result unreliable, though the correction may be obtained by determining the temperatures with the thermocouple of copper and constantan, as E. H. Ashton and W. C. Taylor[4] have suggested. In the second place, the immersion liquid may partially evaporate and its composition and refractive index may change during the experiment, especially at the higher temperatures. We may overcome this trouble by using pure compounds as immersion media instead of mixtures as pointed out by Winchell and Emmons[5]; but pure liquids with appropriate refractive indices are sometimes very difficult to prepare.

In order to avoid the inconveniences enumerated above, the writer proposes here the use of *standard glass powders* in refractive index determinations with the microscope. By this method the refractive index and dispersion of the immersion liquid can be determined simultaneously with those of the crystal mounted on the microscope stage, without using the Abbé refractometer.

In the method here proposed we have no need of using numerous thermostats and water circulating systems, as it is not necessary to keep the refractometer and the microscope stage at the same temperature. It is only necessary to have such apparatus as will maintain the microscope stage at a constant temperature. An electric heating stage with a device for automatic temperature control, such as an Eisenberg or a Walton stage (Leitz), for instance, will meet this need.

I. Isotropic Crystal

Immerse the crystal under investigation in liquid X with proper standard glass powders, A and B, and the refractive index of the crystal as well as those of the glass powders are compared with that of the liquid, using a petrographic microscope and a Fuess monochromater. The refractive indices and dispersions of the standard glasses must have been determined previously and plotted on a Hartmann's dispersion net (Fig. 3). Suppose the refractive index of the liquid X matches those of the glass powders A and B, respectively, at wave lengths a and b. Then, locate the points X_a and X_b (Fig. 2) corresponding to a and b, on the disper-

[4] E. W. Ashton and T. C. Taylor, *Am. Mineral.*, Vol. **XIII**, 1928, p. 411.
[5] *Op. cit.*

sion lines A and B; the connecting line X_aX_b represents the refractive index and dispersion of the liquid X.

Further procedure is quite similar. The wave length x, for which

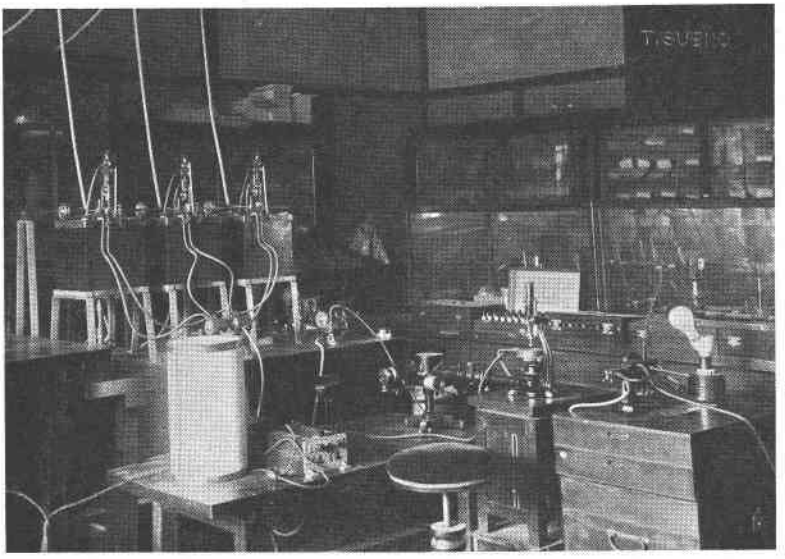

FIG. 1

TABLE I

	Li	Na	Tl		Li	Na	Tl
No. 1	1.6690	1.6764	1.6834	No. 16	1.7178	1.7263	1.7344
2	1.6715	1.6790	1.6862	17	1.7212	1.7299	1.7382
3	1.6740	1.6815	1.6887	18	1.7253	1.7342	1.7424
4	1.6770	1.6847	1.6919	19	1.7296	1.7383	1.7467
5	1.6804	1.6879	1.6950	20	1.7337	1.7425	1.7510
6	1.6830	1.6906	1.6977	21	1.7382	1.7472	1.7556
7	1.6867	1.6943	1.7016	22	1.7429	1.7520	1.7606
8	1.6902	1.6980	1.7052	23	1.7473	1.7566	1.7654
9	1.6936	1.7016	1.7090	24	1.7506	1.7599	1.7689
10	1.6977	1.7056	1.7132	25	1.7540	1.7636	1.7724
11	1.7018	1.7099	1.7174	26	1.7581	1.7681	1.7771
12	1.7038	1.7120	1.7198	27	1.7642	1.7741	1.7834
13	1.7078	1.7160	1.7239	28	1.7690	1.7790	1.7886
14	1.7116	1.7202	1.7281	29	1.7744	1.7847	1.7944
15	1.7152	1.7239	1.7319	30	1.7776	1.7881	1.7979

the refractive index of the crystal matches that of the liquid X, is determined. Then, the experiment is repeated either with the same liquid at another temperature, or with another liquid, Y, and the wave length y determined, at which the matching occurs. To obtain n_D, join the points $n_x n_y$ corresponding to x and y, respectively, to the dispersion lines of X and Y. The connecting line $n_x n_y$ represents the refractive index and dispersion of the crystal, and the intersection of this line with the D line will give n_D.

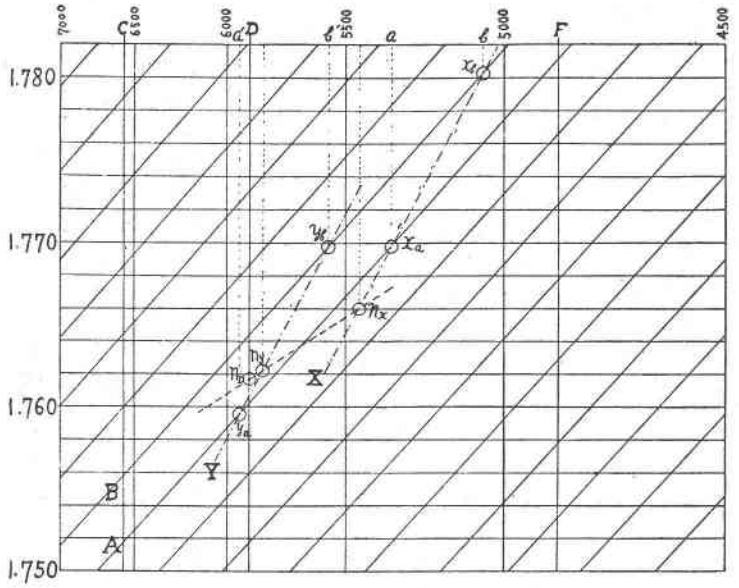

FIG. 2

II. UNIAXIAL CRYSTAL[6]

In order to find the ω and ϵ indices of a uniaxial crystal, it is best to follow the method outlined by S. Tsuboi. The following example will suffice to demonstrate that method. The powders of a crystal of corundum and standard glasses, Nos. 23 and 24 whose dispersions are represented in Fig. 3, were immersed in a suitable liquid. Examinations were made in monochromatic light to determine at which wave lengths the refractive indices of the standard glasses and n_1 and n_2 of each powdered grain of mineral would

[6] The following procedures are quite similar to those described in S. Tsuboi's paper, *Journ. Geol. Soc. Tokyo*, Vol. **XXXII**, 1925, pp. 2–6.

TABLE 2

Standard glasses Liquid	No. 23 550μμ	No. 24 490μμ
Corundum	n_1	n_2
	525μμ	495μμ
	521	494
	513	495
	510	495
	530	494
	511	495
	522	495
	513	495
	509	494
	497	495
	504	495
	520	495
	515	495
	542	494
	520	495
	500	495

TABLE 3

Standard glasses Liquid	No. 23 604μμ	No. 24 545μμ
Corundum	n_1	n_2
	533μμ	513μμ
	529	513
	535	513
	541	513
	563	513
	545	512
	563	513
	543	513
	556	512
	535	512
	547	513
	552	513
	553	513
	525	513
	557	513

TABLE 4

Standard glasses Liquid	No. 24 572μμ	No. 25 513μμ
Corundum	n_1	n_2
	546μμ	522μμ
	555	520
	574	522
	544	523
	572	523
	546	522
	550	520
	533	522
	532	520
	527	522
	546	521
	552	522
	557	522
	545	522
	549	522
	540	522

TABLE 5

Standard glasses Liquid	No. 7 514μμ	No. 8 478μμ
Hyper-sthene	n_1	n_2
	506μμ	480μμ
	538	violet
	536	475
	506	477
	518	485
	518	violet
	500	violet
	496	violet
	500	475
	513	472
	502	violet
	527	473
	497	470
	547	477
	529	violet

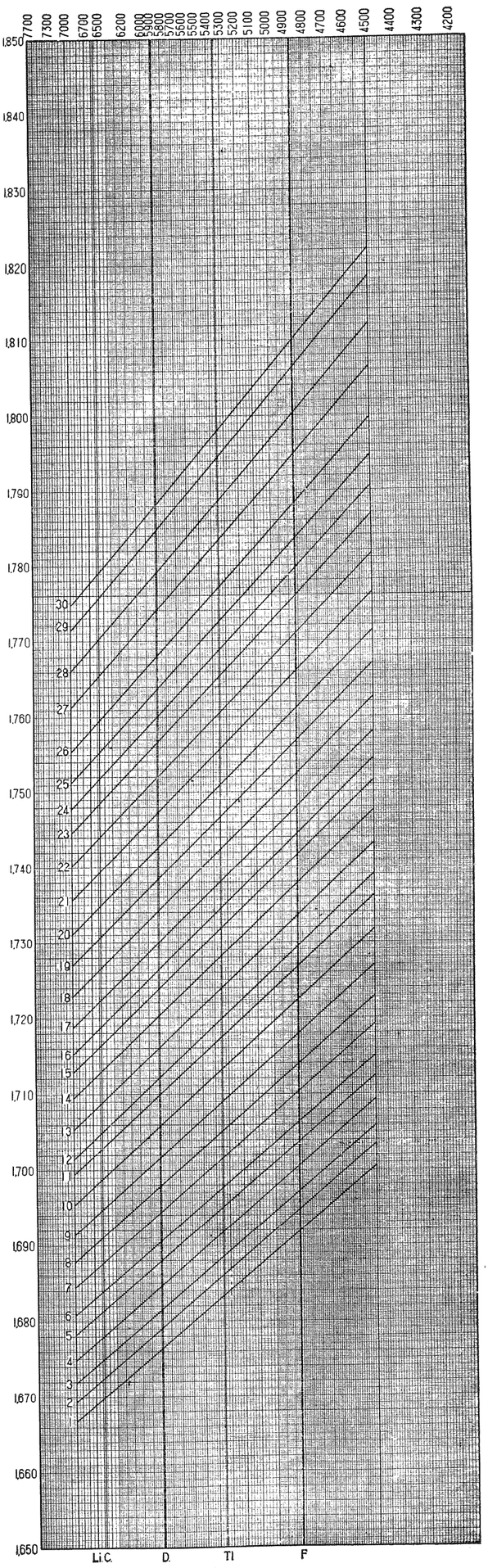
7700 7300 7000 6700 6500 6200 6000 5900 5800 5700 5600 5500 5400 5300 5200 5100 5000 4900 4800 4700 4600 4500 4400 4300 4200
1.850 1.840 1.830 1.820 1.810 1.800 1.790 1.780 1.770 1.760 1.750 1.740 1.730 1.720 1.710 1.700 1.690 1.680 1.670 1.660 1.650
30 29 28 27 26 25 24 23 22 21 20 19 18 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1
Li C.
D.
Tl
F

FIG. 3

match that of the liquid. The observations were made on 15 grains of the mineral chosen at random and it was found that the matching occurred for the wave lengths given in Table 2.

The experiment was then repeated at another temperature, or by immersing the powders in another liquid, and the results as shown in Table 3 were obtained.

To find the dispersion of the liquid, mark the points corresponding to 550 $\mu\mu$ and 490 $\mu\mu$, respectively, on the lines Nos. 13 and 14, and connect them.

As can be seen in the above tables the value for n_2 is constant in every grain, whatever its orientation. This corresponds to that of ω.

To obtain ω_D mark the points corresponding to 495 $\mu\mu$ and 513 $\mu\mu$ (the mean of the wave lengths in the column for n_2 in Tables 2 and 3) on the lines representing the first and second liquids in Fig. 4 and connect them; then the intersection of this line with the D line will give ω_D of the mineral.

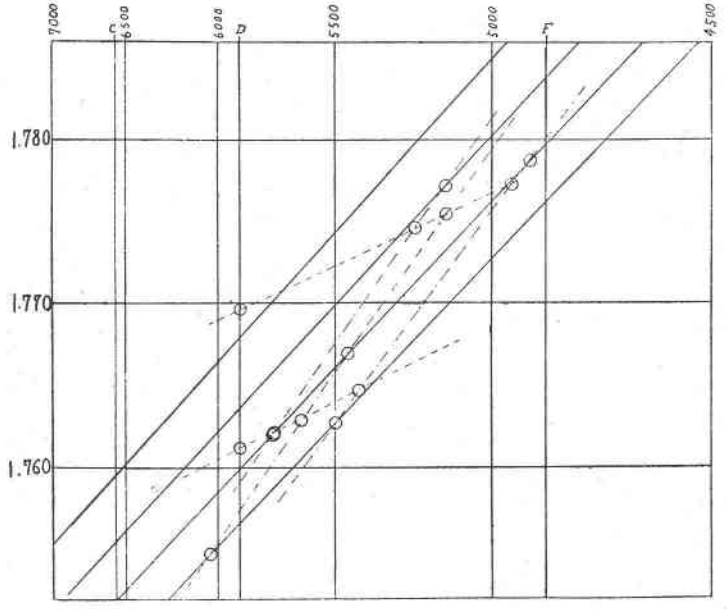

FIG. 4

The value of n_1 observed in each grain is that for the extraordinary ray, and varies from ω to ϵ, ϵ being, in this case, the minimum value of n_1. Of the various values for n_1 given in Table 2, the value

for the light of wave length 542 $\mu\mu$ (the longest wave length) is the lowest, since the dispersion of the immersion liquid is stronger than that of the crystal. Likewise, of the values of n_1 in Table 3, 563 $\mu\mu$ is the lowest. Assuming that these lowest values of n_1 represent approximately ϵ, ϵ_D can be obtained graphically in a manner similar to that indicated for ω_D.

The values of ω_D and ϵ_D thus obtained are:

$$\omega_D = 1.769$$
$$\epsilon_D = 1.761$$

And the dispersion is: $\omega_F - \omega_C = 0.013$

III. Biaxial Crystal

To demonstrate the method for obtaining α_D, β_D, and γ_D of biaxial crystals, a hypersthene crystal from Taihoku, Taiwan, Japan, will be taken as an illustration. The powders of a hypersthene crystal and standard glasses Nos. 7 and 8, whose dispersions are represented in Fig. 3, were immersed in liquids, and the refractive indices of the mineral, standard glasses and liquids were compared in the same way as in the case of the corundum. The results are given in Tables 5, 6, and 7.

Table 6

Standard glasses Liquid Hypersthene	No. 7 580$\mu\mu$ n_1	No. 8 526$\mu\mu$ n_2
	564$\mu\mu$	503$\mu\mu$
	531	509
	587	499
	570	509
	540	*526*
	533	517
	537	518
	572	506
	527	500
	587	503
	545	*491*
	529	495
	578	502
	537	515
	528	509

Table 7

Standard glasses Liquid Hypersthene	No. 9 610$\mu\mu$ n_1	No. 10 537$\mu\mu$ n_2
	red	559$\mu\mu$
	625$\mu\mu$	565
	610	558
	red	576
	red	560
	red	554
	red	565
	red	549
	638	570
	red	*553*
	red	557
	red	580
	red	*609*
	613	567
	red	566

S. Tsuboi has shown the following relationship $\alpha \leqq n_1 \leqq \beta \leqq n_2 \leqq \gamma$ holds for a grain of a biaxial crystal of any orientation. Because of this relationship it is clear that the refractive indices of the mineral that match n of the first liquid for light of wave lengths 547 $\mu\mu$, and 496 $\mu\mu$, as well as those that match n of the second liquid for 587 $\mu\mu$, and 527 $\mu\mu$, are α, and β respectively. And the refractive index that matches n of the second and the third liquids, respectively, for 526 $\mu\mu$, and 553 $\mu\mu$ corresponds to γ. Marking the points representing these wave lengths on the line of liquids in Fig. 5 and

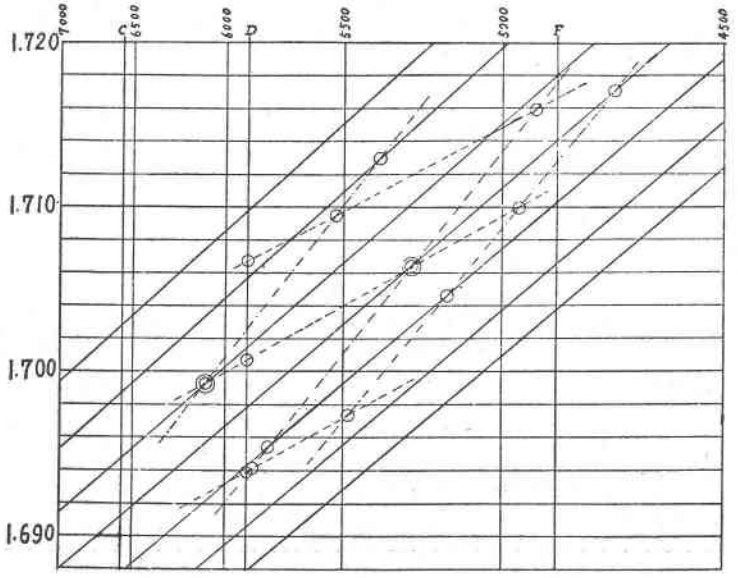

FIG. 5

applying the same graphical method as in the preceding examples, the following values were obtained:

$$\alpha_D = 1.694;\ \beta_D = 1.701;\ \gamma_D = 1.707;\ \beta_F = \beta_C = 0.015.$$

METHOD FOR PREPARING THE STANDARD GLASS

Any type of colorless and transparent glass with appropriate refractive index and dispersion, which is stable and not easily devitrified, may meet the need of a standard glass, without regard to its composition. However, a glass of weak dispersion is preferred, because then the difference in dispersion between the glass and

the immersion liquid is large and a comparatively small number of standard glasses are sufficient for our purpose.

The refractive indices and dispersions of the glasses prepared for the present purpose are shown in Table I, and are represented on Hartmann's net (Fig. 3).

The process of making a standard glass is as follows. At first two kinds of glasses of the following extreme compositions, *A* and *B*, are made in the glass furnace by mixing As_2O_3, quartz sand, Pb_3O_4 and KNO_3.

	Quartz	As_2O_3	PbO	K_2O	
A	38.0	0.3	56.7	5.0	(%)
B	23.0	0.3	75.0	1.7	(%)

When the mixture is heated caution must be taken against blackening due to the separation of metallic lead caused by the action of the reducing flame when used for a long period. The use of K_2CO_3 is avoided, as it may liberate carbon dioxide which sometimes reduces the lead oxides. For melting the glass a crucible of chamotte is used and it is heated for about two hours at about 1300°C. When the molten glass becomes free from bubbles, it is immersed into water and a frit obtained. The frit is crushed in a ball mill to a fine powder and sieved through 4900 mesh. It is important to render the powder extremely fine, for otherwise it may be difficult to obtain a homogeneous glass in the second heating.

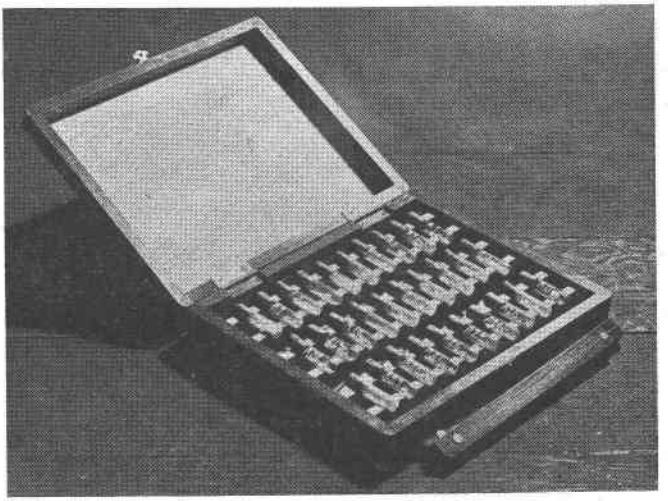

FIG. 6

Next the glass powders of the extreme compositions *A* and *B* are mixed in various proportions and a series of mixtures, 50 grams each, obtained. These are melted in a carbon resistance electric furnace, keeping the preparations at 1000°–1200°C, for about an hour and a half, stirring the melt from time to time with a small platinum spatula attached to the end of an iron rod in order to obtain a homogeneous melt. A small amount of arsenic and antimony oxides are added to facilitate and to expedite the refining of the glass. The crucible used is made chiefly of alumina—e.g., "S.M." of Samposha Company. After removing the crucible from the furnace, it is placed in a small dish of chamotte and cooled to room temperature in about 3 hours. The internal stresses produced upon cooling the glass causes no harm for our purpose, and there is little need for annealing. The outermost part, about 3 mm. thick, is removed, and the homogeneity of the remaining central part is ascertained by comparing the refractive indices of different portions of the mass with one another by the dispersion method. The refractive index and dispersion of the standard glass thus obtained are determined by the prism method. Table I shows the indices and dispersions of the standard glasses manufactured for the present purpose, and Fig. 3 represents the dispersion diagrams plotted on Hartmann's net.

Acknowledgments

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