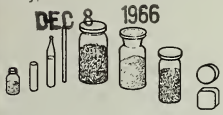

NBS MISC. PUBL. 260-11

Standard Reference Materials:

VISCOSITY OF A STANDARD LEAD-SILICA GLASS

U.S. Department of Commerce
National Bureau of Standards

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Standard Reference Materials:

VISCOSITY OF A STANDARD LEAD-SILICA GLASS

A. Napolitano and E. G. Hawkins

Institute for Materials Research
National Bureau of Standards
Washington, D.C.

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PREFACE

Within the framework of the NBS Institute for Materials Research the area of standard reference materials is a broad and important one, including the preparation, characterization and distribution of a wide variety of materials in such diverse fields as metallurgy, polymers and inorganic materials. In carrying out such a program there is much interaction with representatives of industry and science, beginning with discussions as to which primary standard materials will do most to advance technology, the furnishing of materials and fabrication of samples, and the characterization and certification of the materials by cooperative efforts. The many groups participating in a standards program are very interested in detailed information on specific aspects of the program -- but to date there has been no publication outlet for such written discussions.

To meet this need, NBS Miscellaneous Publication 260 has been reserved for a series of papers in the general area of "standard reference materials". This series will present the results of studies and investigations undertaken within the Institute for Materials Research with emphasis on the preparation and characterization of standard reference materials. This subject-oriented series will provide a means for rapid dissemination of this detailed information and we hope will stimulate the use of standard reference materials in science and industry.

W. Wayne Meinke, Chief
Office of Standard Reference Materials.

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STANDARD REFERENCE MATERIALS: VISCOSITY OF A STANDARD LEAD-SILICA GLASS

A. Napolitano and E. G. Hawkins

Institute for Materials Research
National Bureau of Standards

ABSTRACT

The viscosity of a lead-silica glass has been measured at the National Bureau of Standards and seven other laboratories. Determinations were made in the range of 10^2 to 10^{15} poises (1350-400 °C). Measurements were made by the rotating cylinder, restrained sphere, fiber-elongation, and beam-bending methods. The results have been critically evaluated and the glass has been issued as Standard Reference Material No. 711.

Key words: Beam bending, fiber elongation, glass, glass standard, glass viscosity, lead-silica glass, restrained sphere, rotating cylinders, standard, standard reference material, viscosity, viscosity standard.

1. INTRODUCTION

In a previous paper [1] the viscosity of a standard soda-lime-silica glass, No. 710 was reported as part of the program of physical property measurements on a series of glasses for calibrating instruments and comparing results between laboratories. As a continuation of this program, this report concerns the viscosity of another commercial glass, a lead-silica type, that has been measured at the

National Bureau of Standards and seven other participating laboratories.*

The lead-silica glass was selected as an additional standard because it duplicated another important type of commercial glasses that are made in large quantities. It can be produced as a homogeneous glass and is stable in storage.

The results submitted by the participating laboratories, as well as those of the National Bureau of Standards, have been analyzed and tabulated in a "Certificate of Viscosity Values" and the glass has been issued as Standard Reference Material No. 711.

2. GLASS SAMPLE

Every effort was made at the start to obtain a quantity of glass of the lead-silica type with the greatest possible homogeneity throughout the lot. The lot consisted of 1300 kg of glass in the form of bars having a cross-section of about 5 cm by 5 cm and ranging in length from 45 cm to 60 cm. The bars were marked with the hour of manufacture and numbered consecutively to show the sequence of production. Except for one short period at the beginning of the production run, figure 1, there was no break in the continuous process. The bars numbered 76-79 inclusive were discarded by the producer because the index was outside the specified limits. The bars numbered 1-46 were also not included in the shipment by the producer because they were either off index or were discarded for other reasons.

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*List of participating laboratories which made viscosity and other property measurements on Standard Reference Material No. 711: Bausch & Lomb, Inc., Rochester, N. Y.; Brockway Glass Co., Inc., Brockway, Pa.; Corning Glass Works, Corning, N. Y.; Emhart Manufacturing Co., Hartford, Conn.; General Electric Co., Cleveland, Ohio; National Bureau of Standards, Washington, D. C. (Lab. A); Owens-Illinois, Toledo, Ohio; Owens-Corning Fiber Glass Corp., Granville, Ohio; Thatcher Glass Manufacturing Co., Inc., Elmira, N. Y.

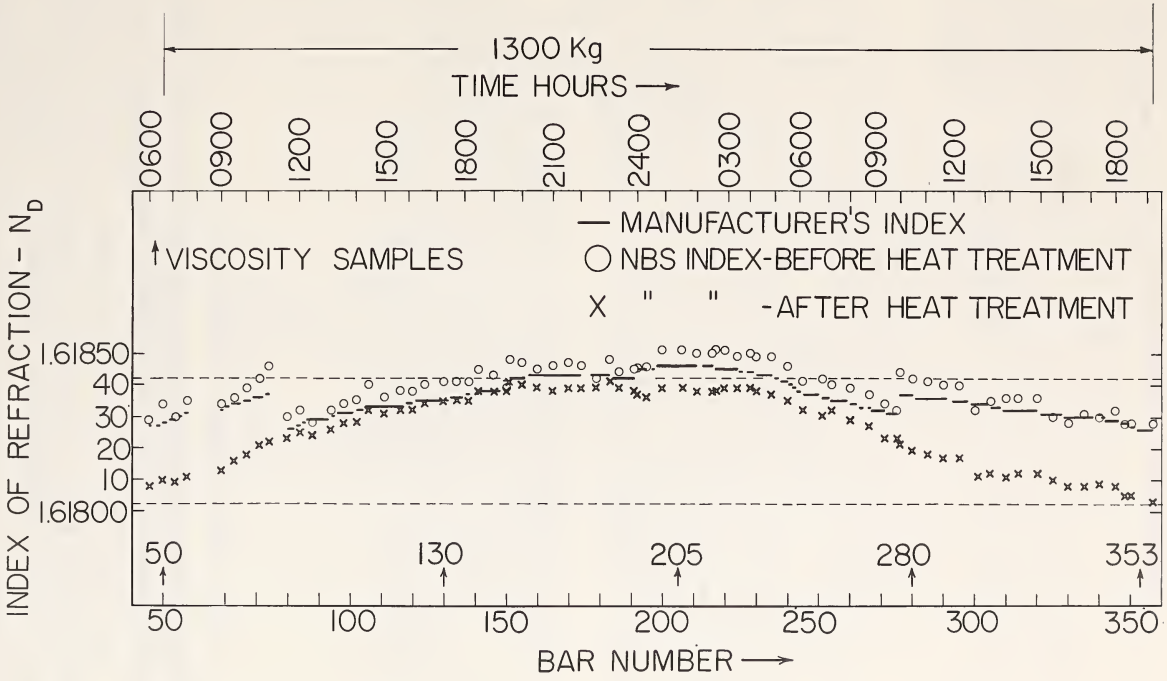

Figure 1. Index variation of Standard Glass No. 711.

The homogeneity of the glass was checked by measuring the index of refraction (N_D-line) on specimens taken from about every 20 kg. This corresponded to an average of two index samples for every hour of the production run. The specimens were measured [2] both in the condition as received and after they all had been given the same heat treatment. These index measurements are shown in figure 1. Also shown in figure 1 are the index measurements reported by the producer.

The variation in index of refraction of the 1300 kg of glass was determined to be $\pm$.0002 after fine annealing at the National Bureau of Standards. Such a variation in index for such a large quantity of glass is within acceptable tolerances. It was subsequently found that no significant variation in viscosity was associated with those portions of glass having different indices (Lab. A). Even though the uniformity of index is not necessarily a unique indicator of uniformity of composition, it is most unlikely that in a continuous production of such a large quantity of glass with no deliberate changes in batch composition, that a significant change in composition would not be reflected by an associated significant change in index. This has been discussed previously [1].

The chemical composition* of the glass as analyzed by one of the participating laboratories** was as follows:

SiO_2	- 46.0 %
PbO	- 45.32
K_2O	- 5.62
Na_2O	- 2.50
R_2O_3	- 0.56

As a further check on the uniformity of the lot, five samples were selected from the lot for viscosity measurements.

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*This glass is not intended as a standard for chemical analysis.

**Laboratory E.

3. APPARATUS AND METHOD OF MEASUREMENT

3.1 Laboratory A

The measurement of viscosity at the National Bureau of Standards was made by the rotating cylinder and fiber elongation methods. The equipment for these measurements has been described in detail in the previous papers [1,3].

3.2 Other Participating Laboratories

All of the other participating laboratories that made viscosity determinations at high temperatures used the rotating cylinder method. The techniques and modifications in the method of making these measurements have also been reviewed briefly in the previous work [1]. Laboratories C, F and G used a Brookfield RVT viscometer to make torque measurements. In addition to the rotating cylinder data, laboratory C submitted another set of data made by the restrained sphere method [4].

With one exception those laboratories that submitted data for low temperatures (below the softening point) used the fiber elongation method. Laboratory D made measurements by a beam-bending method [5] and data was obtained over the range of viscosities $\log_{10}$ 8 to $\log_{10}$ 14 poises*. Measurements were made by laboratory D while the temperature was being held constant and also while the glass was being heated and cooled at a constant rate.

4. RESULTS

4.1 Viscosity Measurements Made at NBS (Lab. A)

The results of viscosity measurements on five samples selected from the lot of glass are given in table 1. Two samples (figure 1), bars No. 50 and No. 353 were taken from regions of lowest index of refraction, bars No. 130 and

*One poise is 1 g/cm s which is equal to 0.1 kg/m s.

Table 1. Viscosities of five samples of glass selected from glass No. 711.

Test method: fiber elongation

Bar No. 50		Bar No. 130		Bar No. 205		Bar No. 280		Bar No. 353	
Temp °C	$Log_{10}\eta$ obs	Temp °C	$Log_{10}\eta$ obs	Temp °C	$Log_{10}\eta$ obs	Temp °C	$Log_{10}\eta$ obs	Temp °C	$Log_{10}\eta$ obs
411.1[a]	14.451	420.7[a]	13.927	422.3[a]	13.869	431.5[a]	13.429	396.3[a]	15.321
414.5[a]	14.299	441.3[a]	12.902	441.9[a]	12.911	460.7[a]	12.042	451.4[a]	12.441
470.1	11.631	482.2	11.202	475.3	11.509	466.2	11.856	488.2	10.987
501.5	10.565	521.9	9.897	506.5	10.399	492.8	10.840	511.9	10.204
526.7	9.727			535.7	9.506	495.4	10.720		
						516.9	10.065		

Test method: rotating cylinder

788.8	5.063	912.0	3.982	899.0	4.079	876.2	4.260	799.7	4.955
805.8	4.898	970.7	3.582	952.3	3.709	907.8	4.010	847.7	4.500
865.3	4.353	1032.2	3.225	1009.0	3.360	997.2	3.428	925.0	3.893
908.2	4.010	1089.9	2.920	1019.6	3.304	1012.0	3.330	997.7	3.416
924.0	3.900	1126.7	2.759	1099.4	2.885	1067.7	3.034	1051.0	3.123
979.8	3.528	1203.6	2.439	1103.0	2.867	1108.0	2.835	1095.3	2.903
1046.9	3.149	1240.2	2.300	1197.9	2.461	1166.4	2.589	1164.0	2.595
1119.4	2.790	1298.3	2.100	1240.0	2.300	1197.8	2.453	1195.0	2.467
1158.2	2.621	1349.0	1.936	1300.3	2.094	1273.6	2.188	1237.0	2.302
1217.5	2.375			1352.3	1.925	1321.0	2.020	1285.3	2.135
1266.7	2.206					1343.3	1.947	1325.0	2.011
1297.9	2.096							1352.5	1.923
1320.1	2.026								

[a] Time held at indicated temperature before viscosity measurements were made are as follows: 411.1, 90 hrs.; 414.5, 260 hrs.; 420.7, 65 hrs.; 441.3, 4 hrs.; 422.3, 210 hrs.; 441.9, 4 hrs; 431.5, 20 hrs.; 460.7, 15 hrs.; 396.3, 335 hrs.; and 451.4, 2 hrs.

No. 280 from regions where the rate of change of index was increasing and decreasing respectively, and, finally, bar No. 205 from the region of highest index. (The uniformity of these samples was such that the viscosity values were within $\pm$2% of the mean at the high temperatures.)

The data from both the low temperature fiber elongation method and the high temperature rotating cylinder method for each of the five samples were combined and fitted to the Fulcher equation [6] by a least squares calculation*. This equation has the form:

$$\text{Log}_{10}\ \eta = A+B/T-T_o \qquad (1)$$

where T = temperature in ^oC

η = viscosity in poises

and A, B, and T_o for each of the five samples and also for the five samples combined are given in table 2.

Table 2. Fulcher equation constants.

Bar No.	Laboratory A (NBS)		
	A	B	T_o
50	-1.653	4319	146.5
130	-1.637	4295	148.4
205	-1.655	4318	148.0
280	-1.663	4330	146.0
353	-1.651	4310	147.6
Combined	-1.654	4317	147.0

*The values reported in this paper were obtained by a method suggested by R. W. Dougles [7]. Equation (1) was written in the form:

$$T \log_{10}\eta = T_o \log_{10}\eta + AT + C$$

where $C = B - AT_o$, and the usual least squares method was used to obtain estimates of T_o, A and C. The values in table 3 and 8 agree, to the number of significant digits reported, with values obtained by the Gauss-Newton iterative method applied to the model, as given in equation (1), although the coefficients differed somewhat (maximum difference was 2.2 standard deviation).

Using these constants, temperatures for specified $\log_{10}$ viscosity values were calculated and are given in table 3. Inspection of table 3 reveals that the reproducibility of temperatures at certain $\log_{10}$ viscosities between samples is within 1 °C. Because of this reproducibility temperatures are cited to the nearest .1 °C throughout this paper.

Table 3. Comparison of temperatures corresponding to specified viscosities calculated from Fulcher equation parameters for each of the five samples.

	Temperature °C					
Bar No.: $\log_{10}\eta$	50	130	205	280	353	Combined
1.90	1362.0	1362.8	1362.5	1361.2	1361.4	1361.8
2.00	1328.7	1329.4	1329.3	1328.0	1328.2	1328.5
2.25	1253.0	1253.4	1253.6	1252.5	1252.5	1252.9
2.50	1186.4	1186.7	1187.7	1186.0	1186.0	1186.3
2.75	1127.4	1127.5	1128.1	1127.1	1127.0	1127.3
3.00	1074.7	1074.7	1075.5	1074.5	1074.3	1074.7
3.25	1027.3	1027.3	1028.2	1027.3	1027.1	1027.4
3.50	984.6	984.5	985.5	984.6	984.4	984.7
3.75	945.8	945.7	946.8	945.9	945.7	945.9
4.00	910.5	910.4	911.5	910.6	910.4	910.6
4.50	848.4	848.3	849.5	848.5	848.3	848.5
5.00	795.6	795.6	796.8	795.8	795.7	795.8
5.50	750.3	750.2	751.4	750.4	750.4	750.5
6.00	710.8	710.8	712.0	711.0	711.0	711.1
6.50	676.2	676.3	677.4	676.4	676.4	676.5
7.00	645.6	645.7	646.8	645.8	645.8	645.9
8.00	593.9	594.1	595.2	594.1	594.2	594.2
9.00	551.9	552.2	553.2	552.0	552.3	552.2
10.00	517.1	517.5	518.4	517.2	517.6	517.5
11.00	487.8	488.3	489.2	487.9	488.3	488.2
12.00	462.8	463.4	464.2	462.9	463.4	463.2

Two representative temperatures were selected to determine the overall precision of measurement and to compare the two methods of measuring viscosities, rotating cylinder at 1200 °C and fiber elongation at 500 °C.

The $\log_{10}$ viscosity values along with their standard deviations (from the Fulcher equation) for each of the five samples and also for the five samples combined for the representative temperatures, 1200 and 500 °C are given in table 4*. For both temperatures, 1200 and 500 °C, the $\log_{10}$

Table 4. Comparison of values of $\text{Log}_{10}\ \eta$ and standard deviations calculated at 500 and 1200 °C from the data of each participating laboratory.

Laboratory		$\text{Log}_{10}\eta$ 1200 °C	σ	Laboratory		$\text{Log}_{10}\eta$ 500 °C	σ
A	Bar No. 50	2.447	.004	A	Bar No. 50	10.565	.060
	130	2.447	.005		130	10.579	.035
	205	2.450	.003		205	10.612	.027
	280	2.445	.006		280	10.569	.045
	353	2.444	.004		353	10.579	.025
A	Combined	2.446	.005	A	Combined	10.575	.040
B		2.436	.005	B		10.580	.015
C		2.412	.006	C		10.619	.030
D		2.431	.005	D		10.633	.073
E		2.421	.010	E[a]		---	---
F		2.443	.006	F[a]		---	---
G		2.559	.003	G[a]		---	---
H[a]		---	---	H		10.612	.022
Combined, A-H[b]		2.448	.033	Combined, A-H[b]		10.610	.056
Combined, except G[c]		2.440	.012	Combined, except G[c]		10.610	.056

[a] No data.
[b] Equation 2
[c] Equation 3

viscosity values for each sample may be said to agree with the corresponding values from the combined equation (five samples) using plus or minus one standard deviation from the value of $\log_{10}$ viscosity of each sample and from the combined equation as the test of agreement. Since these samples were selected

*These standard deviations as well as the others given in Table 4 were computed by combining deviations in the ranges where the changes in the function are small, which were 1075-1375 °C and 475-600 °C, respectively.

from the lot where the index of refraction showed the greatest spread, figure 1, it may be concluded that the combined equation represents to within its uncertainty the $\log_{10}$ viscosity of any of the five samples and also it is assumed of any other sample selected from the lot.

The proportional error of measured viscosity is a function of temperature, the standard deviation of the viscosity being about 1.2% at 1200 °C and about 10% at 500 °C. The measurements at the higher temperatures by the rotating cylinder method are more precise by a factor of eight than those at the lower temperatures by the fiber elongation method.

4.2 Viscosity Measurements Made at Participating Laboratories

The results of viscosity measurements submitted by each participating laboratory are given in table 5 (high temperature data) and table 6 (low temperature data). The data obtained by the restrained sphere method (Lab. C) and that by the beam-bending method using the heating and cooling rates (Lab. D) were not used in the analysis of the data.

The data submitted by each laboratory was fitted to the Fulcher equation by the least squares calculation in the same manner as that used for Laboratory A and the values of the constants A, B, and T_o derived from each each laboratory's data are given in table 7. A comparison of the data from each laboratory is given in table 8 by calculating temperatures for specified values of $\log_{10}$ viscosity using these constants.

The data from all of the participating laboratories was combined and fitted to the Fulcher equation. This gave:

$$\log_{10} \eta = -1.607 + 4249/T^{\circ}C-152.2 \qquad (2)$$

The values of $\log_{10}$ viscosity and their standard deviations for the two representative temperatures T = 1200 °C and T = 500 °C for each laboratory and for the combined data were determined and are given in table 4.

Table 5. High temperature viscosities of glass No. 711.

Rotating cylinder

Laboratory B		Laboratory C		Laboratory D	
Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs
767.2	5.332	966.0	3.580	695.1	6.175
814.4	4.818	1014.5	3.283	742.5	5.553
815.6	4.821	1070.5	2.983	784.2	5.075
900.0	4.093	1114.0	2.771	825.9	4.659
900.6	4.069	1154.6	2.595	874.6	4.238
986.1	3.489	1200.4	2.413	931.5	3.809
987.2	3.480	1244.5	2.257	974.1	3.534
1036.7	3.213			1028.5	3.216
1038.3	3.192			1092.9	2.889
1043.9	3.162			1167.4	2.568
1102.8	2.866			1201.8	2.428
1117.2	2.798			1236.0	2.294
1194.4	2.455			1269.6	2.182
1200.0	2.437			1306.6	2.061
1249.4	2.243			1343.0	1.947
1255.6	2.229			1375.1	1.859
1303.3	2.064				
1314.4	2.031				
1354.4	1.894				

Laboratory E		Laboratory F		Laboratory G	
748	5.517	921	3.90	924.4	4.00
806	4.862	961	3.63	963.9	3.75
902	4.021	1015	3.29	1003.9	3.50
992	3.408	1066	3.04	1050.0	3.25
1092	2.868	1122	2.77	1098.9	3.00
1192	2.464	1181	2.52	1152.8	2.75
1254	2.235	1237	2.30	1213.9	2.50
1301	2.070	1292	2.12	1283.3	2.25
1357	1.900			1362.8	2.00

It is noted in table 4 that the value of log_{10} viscosity at 1200 °C for laboratory G is three standard deviations higher than the value of log_{10} viscosity from the combined data. If the high temperature data from Laboratory G (no low temperature data submitted) is discarded then the above equation becomes:

$$log_{10}\ \eta = -1.621 + 4255/T^{\circ}C - 152.1 \qquad (3)$$

Table 6. Low temperature viscosities of glass No. 711.

Fiber elongation

Laboratory B		Laboratory C		Laboratory H	
Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs
406.1[a]	14.720	455.7	12.267	441.0	12.920
419.7[a]	13.964	471.2	11.782	447.0	12.650
435.3[a]	13.221	479.6	11.326	456.0	12.245
450.0[a]	12.503	493.0	10.865	470.0	11.710
475.0[a]	11.501	496.7	10.720	471.5	11.250
488.6[a]	10.980	505.7	10.409	499.5	10.620
505.8	10.361	511.6	10.222	508.0	10.340
522.2	9.877	516.6	10.047	534.0	9.570
532.5	9.599	527.9	9.726	557.0	8.870
551.9	9.022	542.9	9.260	568.5	8.590
559.4	8.825	562.3	8.776	574.0	8.420
593.3	8.023	563.6	8.733		
		584.5	8.237		

[a]Time held at indicated temperatures before viscosity measurements were made are as follows: 406.1, 137 hrs.; 419.7, 92 hrs.; 435.3, 24 hrs.; 450.0, 16 hrs.; 475.0, 15 hrs.; and 488.6, 2 min.

Beam bending

Laboratory D

Heating		Cooling		Equilibrium	
Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs
515	10.068	520	9.986	416	14.137
520	9.996	517	10.127	425	13.760
520	9.954	513	10.179	428	13.638
520	9.849	509	10.371	436	13.140
525	9.806	505	10.538	445	12.736
525	9.795	500	10.740	458	12.185
525	**9.732**	**495**	**10.892**	**465**	**11.901**
530	9.662	490	11.072	469	11.845
530	9.640	480	11.425	470	11.773
530	9.610	475	11.650	476	11.603
535	9.530	470	11.872	476	11.550
535	9.498	465	12.057	482	11.436
535	9.493	460	12.190	483	11.340
540	9.417	460	12.220	485	11.170
540	9.386	460	12.248	487	11.179

Table 6. Low temperature viscosities of glass No. 711 (con't).

Beam bending

Laboratory D

Heating		Cooling		Equilibrium	
Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs	Temp. °C	Log_{10} η obs
540	9.334	455	12.373	491	10.988
545	9.301	455	12.422	497	10.728
545	9.253	455	12.456	503	10.567
550	9.164	450	12.577	503	10.425
555	9.041	450	12.640	509	10.217
560	8.901	450	12.656	512	10.324
565	8.774	445	12.790	515	10.072
565	8.751	445	12.811	516	10.182
570	8.654	445	12.836	521	9.899
570	8.587	440	12.987		
575	8.498	440	13.000		
575	8.449	440	13.004		
580	8.348	440	13.000		
580	8.314	435	13.161		
585	8.233	435	13.201		
590	8.149	435	13.179		
595	8.045	430	13.340		
600	7.931	430	13.365		
605	7.803	430	13.334		
610	7.692	425	13.505		
		420	13.689		
		415	13.823		
		410	13.965		
		405	14.107		
		400	14.265		
		395	14.442		
		390	14.619		

The values of log_{10} viscosity at 1200 and 500 °C along with their standard deviations from equation (3) for all laboratories (except G) are also given in table 4. In the last column of table 8 temperatures at the specified log_{10} viscosities are given using equation 3.

Table 7. Fulcher equation constants from the data submitted by each laboratory.

Laboratory	Range, °C	A	B	T_o
A[a]	460-1360	-1.654	4317	147.0
B	475-1360	-1.753	4440	140.0
C	470-1250	-1.659	4264	152.7
D	460-1375	-1.526	4106	162.3
E	745-1360	-1.404	3866	189.2
F	920-1300	-1.587	4238	148.4
G	925-1365	-1.830	4871	90.1
H	470- 575	-5.341	7482	31.0
Combined, A-H[b]	460-1375	-1.607	4249	152.2
Combined, except G[c]	460-1375	-1.621	4255	152.1

[a] Data from five samples combined.
[b] Equation 2
[c] Equation 3

The calculated $\log_{10}$ viscosities from 460 to 1380 °C, using equation 3, are represented by a straight line at zero ordinate in figure 2. The differences in the observed and calculated $\log_{10}$ viscosities ($\Delta \log_{10} \eta$) of each laboratory's data using this equation are shown in figure 2. The arbitrary limits ± 5 °C and $\pm 10\%$ viscosity in poises have also been shown for this glass to indicate the magnitude of scatter [1].

The scatter in the observed $\log_{10}$ viscosities with this glass was considerably greater than those of the soda-lime-silica glass. This is especially true at the lower temperatures where the results obtained by the two methods (fiber elongation and beam bending) overlap each other and show an equal amount of scatter. For these lower temperatures only the data obtained at equilibrium are shown in figure 2.

The observed values of $\log_{10}$ viscosity obtained by each laboratory have been plotted against the function of temperature $4255/T^{\circ}C - 152.1$ from equation 3. The high temperature data (rotating cylinder method) is shown in figure 3 and the low temperature data (fiber elongation and beam-bending method) is shown in figure 4.

Table 8. Comparison of temperatures for specified viscosities calculated from appropriate Fulcher equation parameters.

$\log_{10}\eta$ viscosity poise	Temperature °C								
	Laboratories								
	A	B	C	D	E	F	G	H	Combined equation
1.90	1361.8	1355.4		1360.8	1359.3				1360.5
2.00	1328.5	1323.0		1326.8	1324.9	1330.0	1361.8		1327.1
2.25	1252.9	1249.2	1243.6	1249.7	1247.2	1253.0	1283.9		1251.2
2.50	1186.3	1184.0	1178.0	1182.2	1179.5	1185.4	1214.9		1184.5
2.75	1127.3	1126.0	1119.9	1122.6	1119.9	1125.7	1153.5		1125.5
3.00	1074.7	1074.1	1068.0	1069.5	1067.1	1072.4	1098.5		1072.8
3.25	1027.4	1027.5	1021.3	1022.0	1019.9	1024.6	1048.9		1025.6
3.50	984.7	985.2	979.3	979.3	977.5	981.6	1003.9		982.9
3.75	945.9	946.8	941.0	940.6	939.3	942.5	963.0		944.3
4.00	910.6	911.8	906.2	905.3	904.6	907.0	925.5		909.0
4.50	848.5	850.1	845.0	843.7	844.0				847.2
5.00	795.8	797.5	793.1	791.5	792.9				794.7
5.50	750.5	752.2	748.3	746.7	749.2				749.6
6.00	711.1	712.7	709.5	707.9					710.4
6.50	676.5	678.0	675.3	673.9					676.0
7.00	645.9	647.2	645.2	643.9					645.6
8.00	594.2	595.2	594.2	593.3				591.8	594.3
9.00	552.2	552.9	552.8	552.4				552.7	552.7
10.00	517.5	517.8	518.4	518.5				518.7	518.2
11.00	488.2	488.1	489.5	490.1				488.8	489.2
12.00	463.2	462.8	464.9	465.9				462.4	464.5

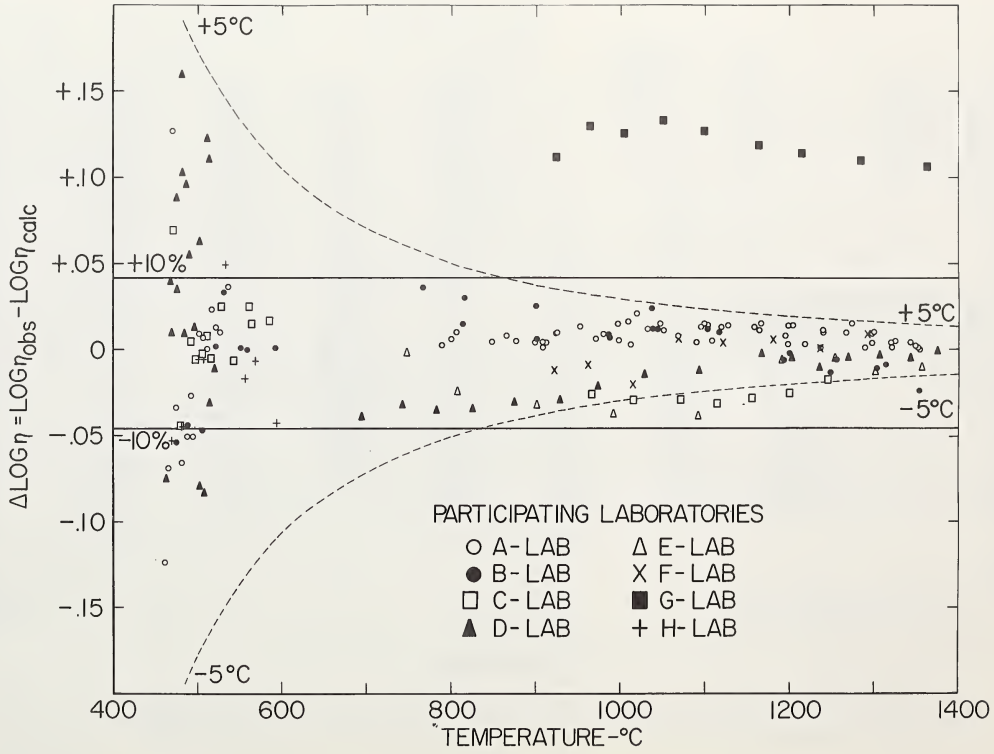

Figure 2. Differences in observed and calculated $\log_{10}$ viscosities of participating laboratories on Standard Glass No. 711.

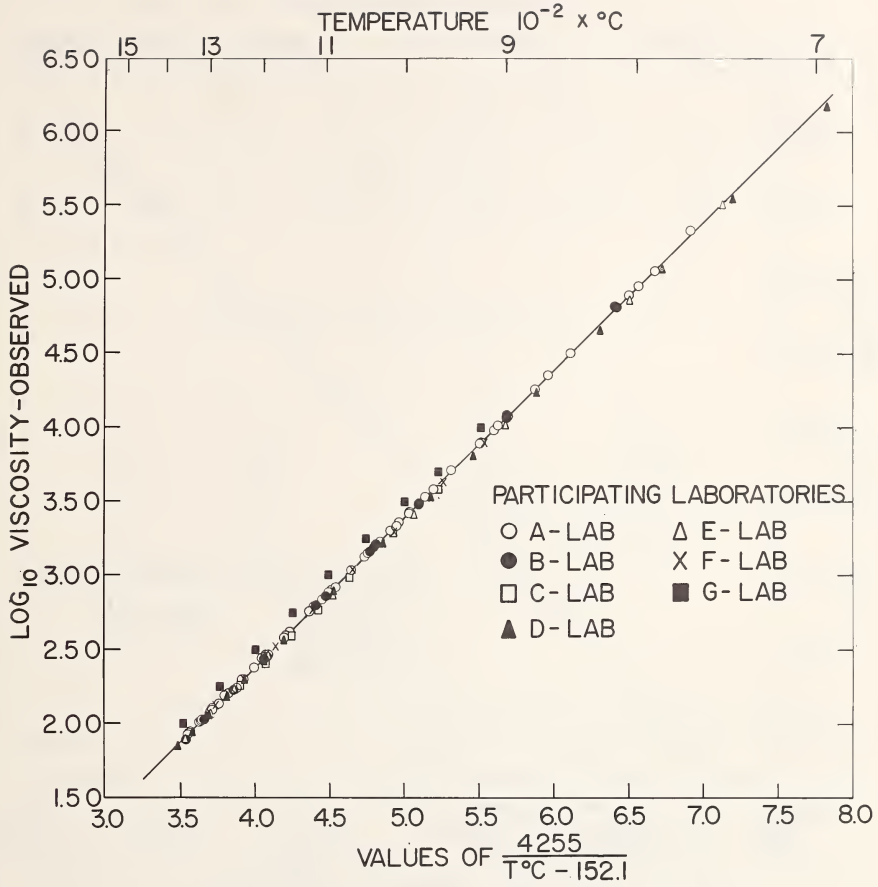

Figure 3. Observed values of $\log_{10}$ viscosity plotted against the function of temperature $4255/T\ ^{o}C - 152.1$ (700-1400 °C).

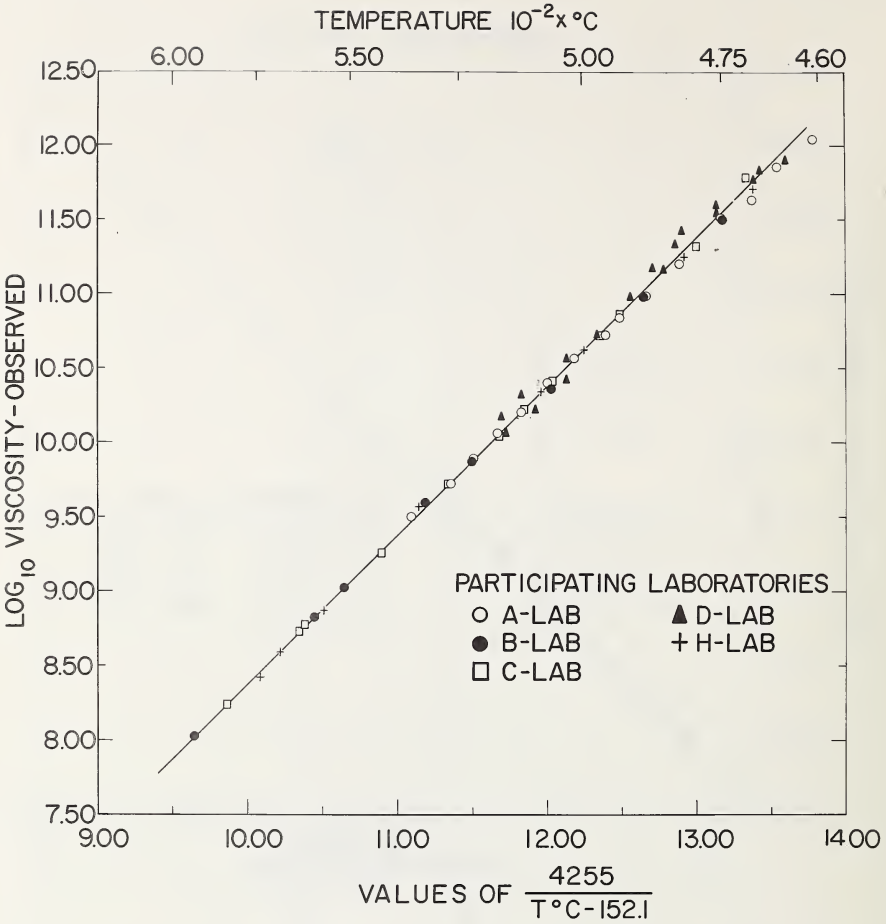

Figure 4. Observed values of $\log_{10}$ viscosity plotted against the function of temperature 4255/T °C - 152.1 (460-600 °C).

A comparison of the results (see table 9) by the restrained sphere method (Lab. C) and equation 3 is made in figure 5. The solid line curve represents calculated values from equation 3. The average departure of the results by the restrained sphere from those of the rotating cylinder is -3.5% with one point off by -10% and another off by -20% in viscosity. This is considered a relatively good agreement between the two methods.

Table 9. High temperature viscosities of glass No. 711 (restrained sphere, Lab. C).

Temp °C	$\text{Log}_{10}\,\eta$ obs	Temp °C	$\text{Log}_{10}\,\eta$ obs
889	4.144	1061	3.026
920	3.840	1064	3.073
932	3.827	1095	2.877
962	3.589	1118	2.749
987	3.442	1146	2.632
1009	3.317	1170	2.574
1037	3.168		

In the round robin tests made with the first Standard Reference Material No. 710, Hagy [5] made a comparison of the beam-bending and fiber elongation methods of measuring viscosities and found that the two methods were in agreement. With this glass, No. 711, at equilibrium temperatures, the beam-bending data showed a little more scatter i.e. $\pm$18% in viscosity as compared to $\pm$15% by the fiber elongation method (table 4). Since both methods of measuring viscosity cover essentially the same range of temperatures it is possible to derive a best curve from the data with a standard error of $\pm$14% in viscosity ($\pm$1.5 °C) at the lower temperatures.

The beam-bending data obtained while the sample was being cooled and heated is shown in table 6 and plotted in figure 6.

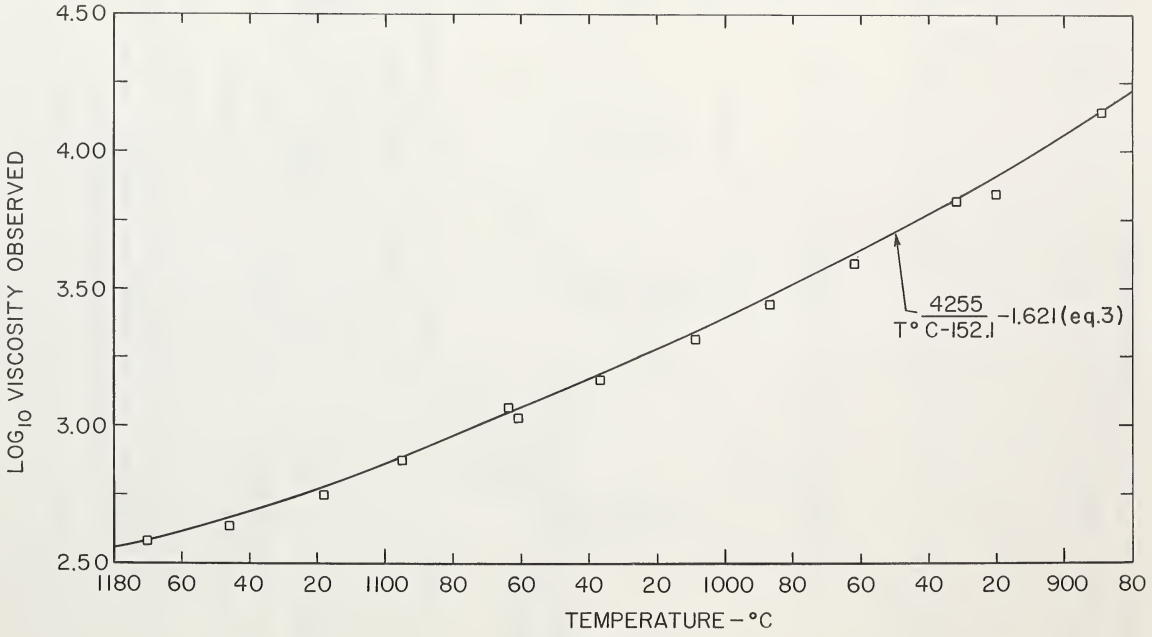

Figure 5. Comparison of $\log_{10}$ viscosities obtained by the restrained sphere and rotating cylinder methods on Standard Glass No. 711.

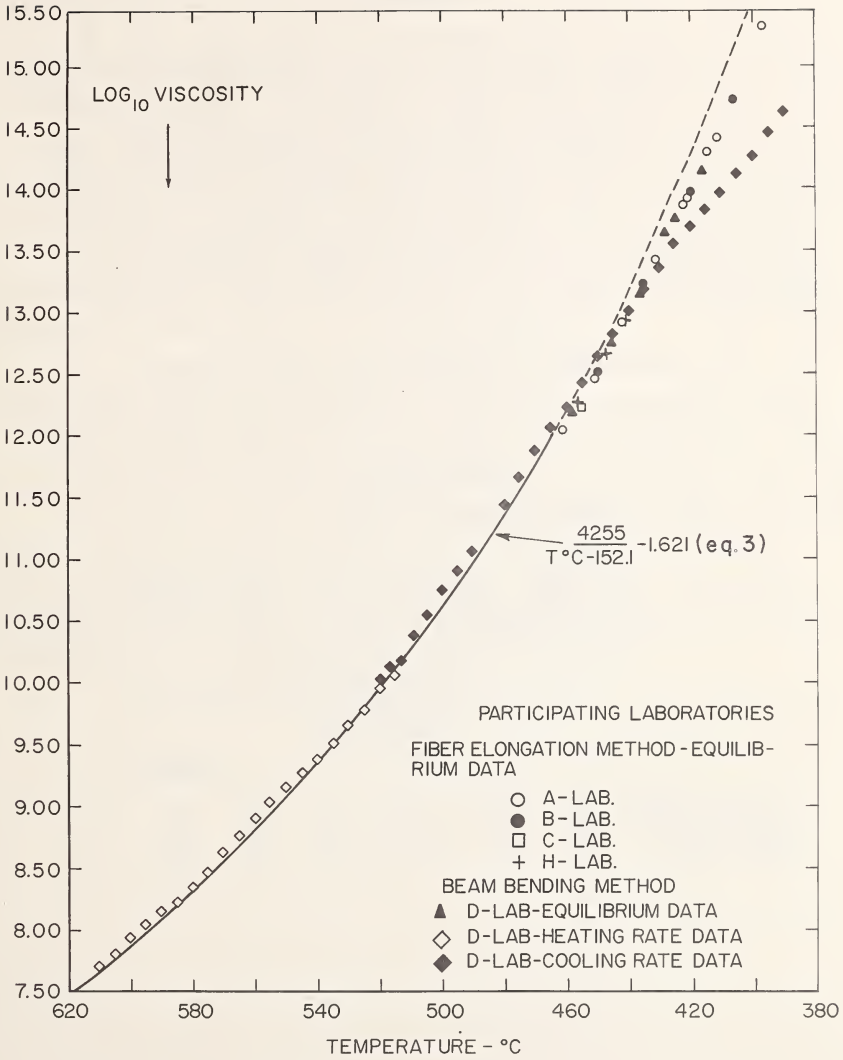

Figure 6. Comparison of $\log_{10}$ viscosities obtained by the beam-bending (heating and cooling cycles) and fiber-elongation methods on Standard Glass No. 711. Equilibrium values of $\log_{10}$ viscosity beyond $\log_{10} \eta = 12$ as obtained by several laboratories are also compared with the Fulcher equation (extrapolated).

The results are compared to the values calculated from equation 3 (table 8) with values extrapolated beyond $\log_{10}$ 12. In addition, viscosity values made under equilibrium conditions above $\log_{10}$ 12, are also shown in figure 6. If these data, above $\log_{10}$ 12, obtained under equilibrium conditions, had been included in the original least square calcualtion the data would not fit the curve (Fulcher equation) at the high temperatures (low viscosity) and at the low temperatures (high viscosity) as well as it does using only the data between $\log_{10}$ 2 and $\log_{10}$ 12.

4.3 Softening, Annealing and Strain Points

The softening, annealing and strain points were determined by five participating laboratories and are given in table 10. The definition of these points and methods of determining them are given in ASTM Standards [8,9].

Table 10. Softening, annealing, and strain points of standard glass No. 711.

	Temperature °C					
	Laboratories					
	A	D	E	F	I	Average
Softening point	603	602	599	603	603	602
Annealing point	433	431	429	432	435	432
Strain point	393	393	389	387	396	392

5. SUMMARY

(1) NBS has established an additional Standard Reference Material for viscosity of glass: No. 711 (Lead-Silica).

(2) Viscosity measurements have been made on this glass by eight participating laboratories. The temperature range covered was 390 to 1375 °C. The rotating cylinder, restrained sphere, fiber elongation and beam bending methods were used.

(3) A viscosity temperature curve was determined from these measurements by fitting the data points to the Fulcher equation by the method of least squares.

(4) The softening, annealing and strain points of this glass have been determined by five participating laboratories.

6. ACKNOWLEDGMENT

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