A STUDY OF THE SCRATCH HARDNESS OF CERTAIN GLASSES

BY

KENNETH CASSINGHAM LYON

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THESIS

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I HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER MY SUPERVISION BY Kenneth C. Lyon

ENTITLED A Study of the Scratch Hardness of Certain Glasses

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C. W. Parmelee
In Charge of Thesis

C. W. Parmelee
Head of Department

Recommendation concurred in*

C. W. Parmelee
R. K. Hursh
T. E. Phipps
J. I. Andrews
H. C. Rask

Committee on Final Examination*

*Required for doctor's degree but not for master's.

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There is an obligation to those firms and individuals who supplied the glass samples used in the tests. The author wishes that he could present the name of each. Where the choice lies between naming these benefactors and listing the compositions of their glasses, the worthwhile course is obvious.

I. INTRODUCTION

The physical property of "hardness" is usually considered to be the resistance of a substance to deformation, cutting, or scratching. The property has never been so clearly defined that its value is independent of the method of measurement. Thus it is that we have "indentation", "impact", "scratch", "abrasive", and other types of hardness, depending upon the particular method used in evaluation.

The subject of hardness has been given some consideration in the study of metals, and the property is appraised as a control method in their thermal treatment. An indentation test is generally used for this purpose. The numerical value from such a test depends on the particular procedure as well as the condition of the metal. The indentation hardness values obtained from different procedures may sometimes be related empirically.

While there may be a concordance of results within the bounds of any one method of measurement, e. g. indentation hardness, the results from different methods frequently are neither proportional nor even of the same order of degree. This is particularly true when the series tested contains both ductile and brittle materials.

In considering the hardness of glass, the resistance to scratching seems to be of greater practical importance than indentation and impact properties. Polished glass artware, containers, tableware, and flat glass are presumably of greater

aesthetic value if they remain unscratched, or if the scratches may be confined to such size as to be invisible to the eye. Scratching of the surface markedly weakens glass, and the scratching of optical glass frequently makes it unusable.

An impact test would be appropriate for the comparatively small amount of glass which is subject to similar treatment in use. If it were possible to get permanent deformation of glass in the absence of rupture, an indentation test could be made. This would have little practical significance.

The purpose of this investigation was to determine the relative surface scratch hardness of various types of glasses. The width of scratch produced when a glass sample was drawn beneath a loaded diamond point served as the means of measurement.

A number of typical commercial glasses of known composition or surface preparation, and a series of glasses of varying alumina content were tested. Some data are presented to permit a comparison between scratch hardness and modulus of rupture.

The scratch hardness of the glasses tested is expressed in terms of the Mohs scale.

II. REVIEW OF LITERATURE

The first recorded attempt to study the hardness of glass seems to have been made by Auerbach[1] who used an indentation test to give a value which he considered to be the characteristic hardness. The test, originally treated theoretically by Hertz[2], is made by observing the effect of a sphere pressed against a plate of the same material. After Auerbach had tested a number of glasses by the indentation method, he prepared sharp-pointed specimens with which he scratched other samples. The widths of the scratches produced were obtained by the use of a microscope and the values used in arranging the glasses in order according to their scratch hardnesses. There was incomplete agreement between the results of the two tests.

Auerbach calculated coefficients for the hardness of glass as determined by its composition. In the table of coefficients, all of the oxides commonly used in glasses are given positive values except Na_2O and CaO which are represented as negative.

Values for the hardness of the minerals of the Mohs[3] scale are given in one of Auerbach's works. The common glasses lie between 4-1/2 and 7 in hardness, and clear silica glass has a hardness of about 5.

In 1911, Schneider[4] published a description of a hardness test in which a steel ball was allowed to fall on a glass plate from increasing heights until the surface layer was fractured. The hardness was taken as a function of the rebound.

Le Chatelier[5] reported the results of abrasive hardness tests carried out by Lecrenier on a number of glasses. There

was no general agreement between the values found for the abrasive hardness and those obtained by use of the Auerbach method. From the data given, it would seem that small amounts of silica replaced by lime on a weight basis produced a glass more resistant to abrasion.

Berndt[6] measured the scratch hardness of a borosilicate glass using a Martens[7] sclerometer with a 90° conical diamond point, and found the load necessary to produce a scratch 10 microns in width. The scratch hardness was found to be the same for annealed and unannealed samples of glass.

Hâlle[8] proposed a hardness scale depending upon the loss of weight undergone by the glass sample when pressed against a rotating abrasive disc.

Scott[9] devised an apparatus for the measurement of abrasive hardness of glazes. The hardness value was determined from the loss of weight occasioned when sand was allowed to fall on the test piece.

Lecrenier[10] determined the abrasive hardness of glass by pressing a sample against a grinding wheel and abrading about equal amounts under constant pressure. He worked out a relation between abrasive hardness and composition, and found soda glasses harder than potash, lime giving harder glasses than soda, boric acid increasing hardness, and soda and lime increasing the hardness of lead glasses.

Gehlhoff and Thomas[11], using a Martens apparatus, found the scratch hardness of a number of acid-treated glasses, and expressed their results in l/mm. of scratch width for twenty

grams loading on the scratching point. The authors state that all glasses had approximately the same hardness until after the acid etching. The scratch width varied between 3.9 and 9.1 microns. Factors are given for the calculation of scratch hardness from composition using the method of permutation. Contrary to Auerbach's findings, additions of CaO increased the hardness of glasses, and K_2O acted in the opposite manner. From a practical standpoint, the advisability of the use of the preliminary acid treatment might be questioned.

Graf[12] investigated the relative resistance of glass and other building materials to sand-blasting.

Lai and Silverman[13] determined the hardness of a series of beryllium glasses by finding the load on a diamond point necessary to produce a microscopically visible scratch. By use of minerals they correlated their results to the Mohs scale and found that glasses of the Na_2O-BeO-SiO_2 group had hardnesses varying between 6.2 and 6.7.

Navias[14] used a set of standards in hand scratching tests and found bottle glass to lie below 5 on the Mohs scale and chemical glass to be between 5 and 6.

Tammann and Klein[15] carried out some scratching tests on a group of organic glasses and a lead silicate glass to study the effect of temperature, speed of scratching, load, and angle of inclination of the scratching point on the character of the scratch produced. The evaluation of hardness was not attempted.

Becker[16] used a method somewhat like that of Gehlhoff and Thomas to find the effect of additions of beryllium oxide on

the scratch hardness of a glass. Using a loading of 15 grams on the scratching point, the results are expressed in 1/mm. of scratch width. Beryllium oxide, in terms of mol percent addition, has about the same effect as magnesium oxide in increasing hardness. The measurements were carried out on polished surfaces of optical quality.

III. APPARATUS

A. SCRATCH HARDNESS

The apparatus used in making the scratch hardness tests is shown in Figure 1. It is a modification of the Martens[7] sclerometer* and an instrument devised by Talmage[17]. It was constructed in the Physical Plant Machine Shop, of the University of Illinois, from an original design.

The specimen to be tested for hardness was mounted in fusible alloy (Wood's metal) and clamped in the brass holder of 3.1 cm. diameter by pressure from the screw s. It was possible to move the holder and specimen laterally on the carriage by use of the adjusting screw a. The carriage c was made of brass, 7.6 cm. in width, 5.6 cm. in length and 1.3 cm. in thickness. It was supported and moved longitudinally on four steel balls of 0.6 cm. diameter resting in 90-degree V-shaped grooves cut parallel in the lower side of the carriage c and the upper surface of the platform p. The platform p was 7.6 cm. in width, 1.2 cm. in thickness, and 8.1 cm. in length. It rested upon a brass column 2.5 cm. in diameter, which was fastened rigidly to the brass base plate.

Mounted on the end of the platform p was a screw d which threaded into the carriage block c. The rotation of this screw, brought about by the operation of the pulley q driving through a worm and gear g, caused the carriage to move longitudinally.

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*A history of the development of the sclerometer is given in Die Härte der festern Körper, by Viktor Pöschl, Dresden, 1909.

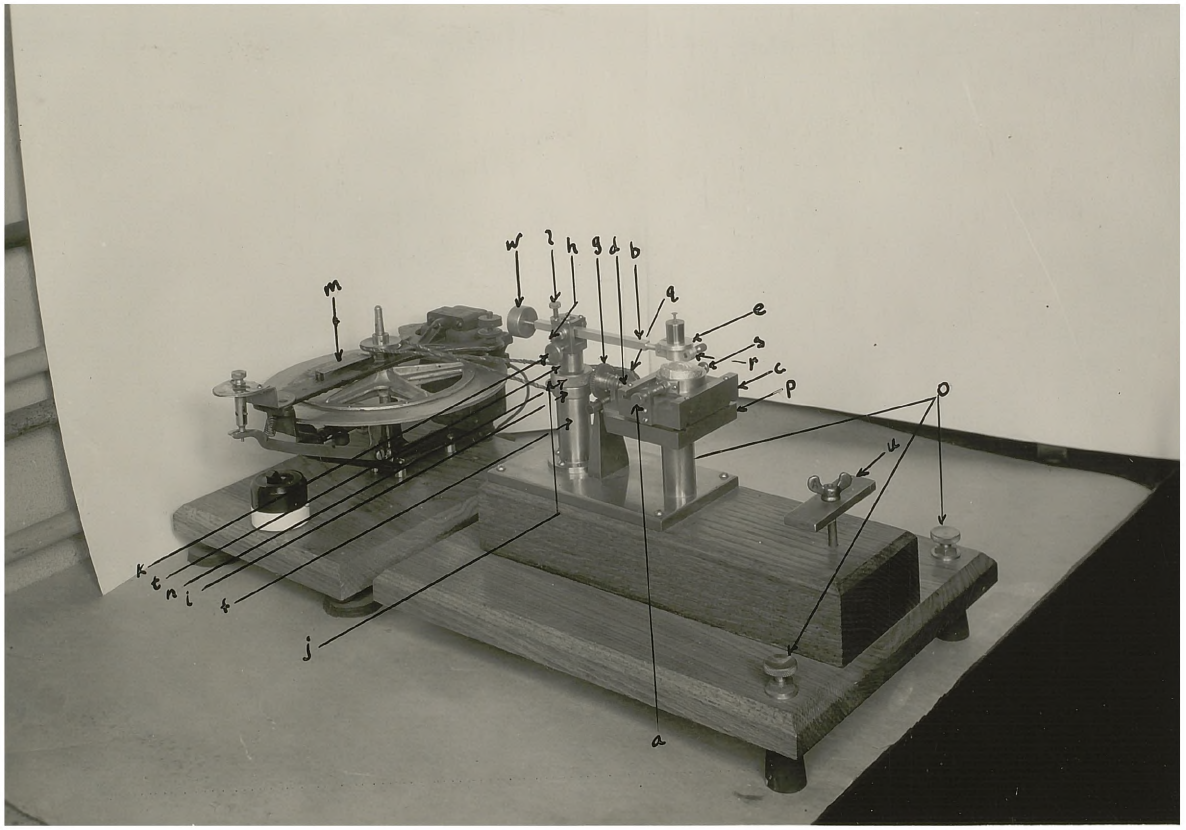

Figure 1

APPARATUS USED IN DETERMINING THE SCRATCH HARDNESS OF GLASSES

The pulley q was belt-driven from a governor-regulated induction-type electric motor m, designed for the operation of a phonograph.

The scratching point was mounted in a metal fitting 1.3 cm. in diameter, by 0.9 cm. in depth. This was firmly clamped to the end of the supporting beam b in a contracting ring r. Immediately above the scratching point was a stage s, of 2.5 cm. diameter, upon which weights were placed for loading the point as shown in the illustration. The supporting beam b was made 0.6 cm. square and was 10.2 cm. in length from the center of the stage s to the supporting bearings h, the total length being 13.5 cm. The supporting beam b, stage s, and ring r were made from Duralumin alloy to decrease their weight. The weight of the supporting beam and scratching point was counterbalanced by a brass weight w, adjusted by a screw threading into the end of b.

The beam b was supported on a brass column which was held closely in a sleeve f. The column could be raised and lowered by turning the nut n, and could be locked in position by tightening the collar i, using screw j. Loosening the setscrew k permitted the supporting beam to be swung aside when it was desired to change samples or make measurements with the microscope. The screw l, bearing on the supporting beam b allowed the loaded scratching point to be lowered gently to the test surface.

The scratching apparatus was firmly fastened to an oak board which could be leveled by adjustment of the three screws

g, which rested on rubber bottle stoppers. This arrangement was necessary to prevent undue vibration of the apparatus.

The microscope, fitted with a filiar micrometer eyepiece, and a vertical illuminator, was used in measuring the widths of the scratches produced in the testing. The base of the microscope was held in the clamp u. A magnification of approximately 1000X was used in making the measurements. This was obtained by the use of a 12.5X micrometer eyepiece, a No. 7 (Leitz) dry objective, and the full extension of the microscope tube.

The scratches were lighted by use of a Leitz vertical illuminator. The axis of the prism was placed at an angle of about 60° to the direction of the scratch. A special attachment permitted the use of a six-volt coiled filament lamp for lighting.

In the majority of the tests reported, a diamond, lapped to form a point made by three planes at an angle of 130° to each other, was used in making the scratches. A micro-photograph of the point, taken with the negative perpendicular to the intersection of two of the planes is given in Figure 2. Each division on the scale shown represents 10 microns.

B. MODULUS OF RUPTURE

The apparatus used in making the modulus of rupture tests is shown schematically in Figure 3. It was a modification of the machine used for determining the modulus of rupture of unburned clay bars, and consisted of an arrangement of levers which applied a load to the specimen when water was admitted

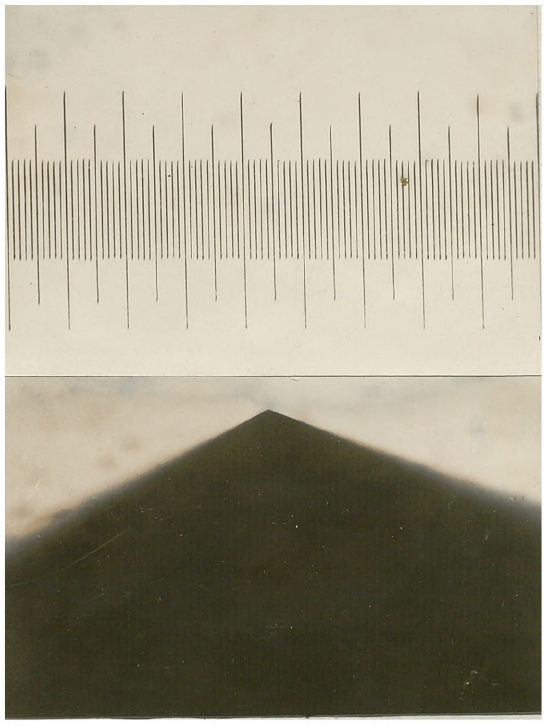

Figure 2

Silhouette of scratching point perpendicular to intersection of two planes. Magnification approximately 80 X.

Modulus of Rupture Apparatus

Scale :- $\frac{1}{8}$" = 1"

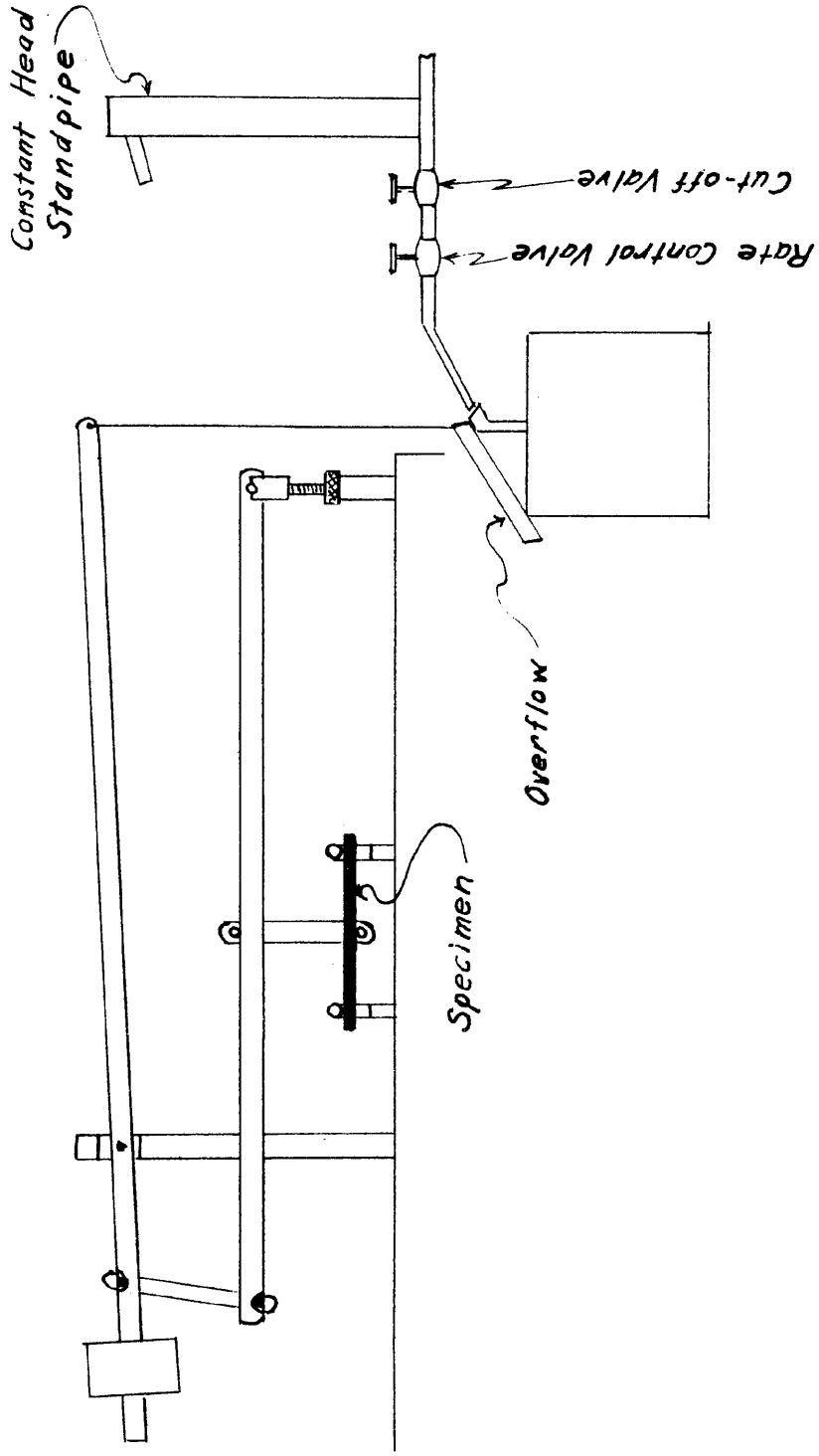

Figure 3

to the receiving vessel. When the specimen broke, the water was carried off through the overflow trough. The rate of flow was suitably controlled by adjustment of the valve. A seven-inch (17.8 cm.) span was used in breaking the specimens which were supported on steel rods, 1/2-inch (1.2 cm.) in diameter. The supports were covered with a layer of rubber 1.5 mm. in thickness.

IV. EXPERIMENTAL

A. SCRATCH HARDNESS

1. Technic of Testing

Except for specific instances which will be noted as their cases arise, the procedure followed in testing the scratch hardness of the materials reported was as outlined in the following:

To mount the diamond properly in its holder at r (see Figure 1), the assembly r, b, etc. was removed and placed on the stage of a low-powered microscope in such a manner that the position of the facets with relation to the beam b could readily be seen. The point was then turned so that the intersection of two of the planes forming it extended in the direction of the supporting beam b. The metal mounting of the diamond was marked so that one particular orientation could always be obtained.

With the diamond point mounted, the apparatus was re-assembled, and the correct position of the beam was secured by locating it parallel to the driving screw d. The beam was locked in place by tightening screw k, and was balanced by adjusting w until a weight of 0.07 grams placed on e caused a slow deflection and a weight of 0.15 grams on w resulted in a deflection of about the same speed. (A weight of 0.153 grams would have been precisely correct in the latter case.)

The glass samples were variously prepared. Those which had to be melted, either because of the condition of the material available, i. e. powder, or because of the need of a

fire-polished* surface, were heated in a platinum crucible in an electric resistance furnace to a temperature sufficiently high to permit pouring on a metal sheet. These castings were subsequently annealed by cooling from 550° C. to 400° C. at a rate of 19° C. per hour and by allowing them to remain in the furnace while it returned to room temperature. Specimens annealed were examined in polarized light to check removal of strain.

The samples which were polished in the laboratory were first ground with fine-grained silicon carbide abrasive to give a flat surface and then polished with wet rouge on felt. Some glass samples were too large for the sclerometer to accomodate. These were cut to proper size with a wet silicon carbide cutting wheel. A few of the samples were given special treatment which will be mentioned in the section devoted to the description of samples.

The glass samples of suitable size were mounted in fusible alloy (Wood's metal) for testing by placing them face downward in a round plaster of Paris mold and pouring the molten metal over them to the proper depth. The sample was held in position until the metal had cooled and solidified. When this method of mounting was not convenient, the metal was first poured into the mold and the glass sample inserted into the surface of the partially cooled metal and held until solidification

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*As used in this dissertation the term "fire-polished" refers to the condition of a glass surface prepared by cooling from a melt. Contact with flame or with products of combustion is not requisite to this terminology.

had taken place.

After the mounting material had completely cooled, the glass sample was placed in the specimen holder and firmly secured by tightening the screw s. The diamond point was caused to rest lightly on the surface of the sample by loading the beam with a small weight. The nut n was then adjusted to bring the beam b into a horizontal position. After this was done the screw j was tightened to hold the column t rigidly, and the beam was lifted by use of the screw l.

Before making a test the glass surface was wiped with acetone to clean it, and the carriage was placed in a position to locate the beginning of the scratch at the desired point. The beam was then loaded with a weight on e, the motor was started to cause the carriage to move forward, and the point was lowered to the surface. The scratches were made with the carriage moving only in the direction toward the leading edge of the intersecting planes on the diamond point; the return of the carriage was made with the scratching point lifted from the surface.

A single test usually consisted of making six parallel scratches at short distances apart, weights of ten, twenty, thirty, fifty, eighty, and one hundred grams, respectively being used for loading the point. The scratches were about 0.5 cm. in length, and were made using a carriage speed of 0.27 cm. per minute. This resulted with a motor speed of 100 r. p. m. The motor speed did not vary more than one r. p. m. with the standard setting of the governor.

Three tests were usually run on the same sample of glass, the scratches for all three tests being made at one time.

After making the scratches, the point was swung aside, and the microscope was set to focus on the scratches. The filiar eyepiece of the instrument was calibrated using an average from a 1/100 mm. stage micrometer made by the Bausch and Lomb Optical Co., and a similar standard ruled in the Physics Laboratory of the University of Illinois. The calibration was found to be 12.32 microns per large eyepiece division.

Seven measurements were made along each scratch except where chipping conditions were so bad that the path of the scratching point was not visible. From three tests on any one sample there were, then, twenty-one values for each loading. In some instances many more than twenty-one observations were made in testing the reproducibility of values and comparing values from different regions in a larger sample of glass plate. In other cases, due to excessive chipping, fewer than twenty-one measurements were made for a given loading on any one sample.

The great difficulty in properly viewing the scratch width cannot be too strongly emphasized. On first thought it would be said that three lines should be visible -- the two edges, and the line at the bottom formed by the intersection of the side planes of the scratch. There were visible, running parallel to these, other markings due, presumably, to reflections, light interference phenomena, and minute imperfections in the face of the diamond scratching point. One must make many ob-

servations before he can feel that he is making comparable measurement of scratch widths with any degree of certainty.

A number of different procedures were tried in attempting to make the scratch more readily definable. The glass was given a thin coating of petrolatum with the thought that the scratching point would break through the layer and leave a clearly defined interface between the grease and glass. It was observed that the effect of breaking the film was, apparently to form it in droplets on the glass surface. The test piece was given a thin coating of smoke by holding it over a gas flame. Under the high magnification the coating was resolved into particles which were too large and which were distributed at too great intervals to be of aid in defining the scratch. The glass surface to be tested was given a light mirror coating with silver. In forming the scratch the silver tore irregularly along the side of the depression. The best coating to define the edge of the scratch was obtained by rubbing the finger in dirt and smearing it on the test piece. The danger of the presence of abrasive particles which might tend to vitiate the results of the tests led to the abandonment of the practice. It was possible to gain some improvement of visibility conditions by rubbing the glass surface with a dirty finger after the scratches had been made. Except in extreme cases this practice was not followed.

Very late in the experimental work it was found that some improvement in the definition of the scratch could be obtained if it was formed with the carriage moving opposite to the

usual direction. One of the flat faces of the diamond point, instead of the intersection of two planes, met the oncoming glass, and a scratch resulted which showed much less chipping along the edge and somewhat better definition.

In addition to attempting to improve the visibility of the scratch edge by treating the surfaces of the test specimens, there was an opportunity to observe the scratches in dark-field illumination, with polarized light, and with an oil-immersion objective. It was found that the system with bright-field illumination, with the dry objective, and with non-polarized light, was best suited to the work.

2. Calibration

The reduction of test results to numerical form is not necessary, but it is extremely convenient. Verbal explanations of degree are unsatisfactory because the words used cannot have exactly the same meaning to the reader as to the writer. There are several ways in which hardness may be expressed numerically. Each has its faults and limitations.

The Mohs[3] scale of hardness has long been used for roughly evaluating the scratching properties of minerals. This scale has been chosen as a basis to permit a simple expression of the relative hardness of the glasses studies in this investigation. At least in one respect -- their brittleness -- the minerals of Mohs' scale are adapted for evaluating the scratch hardnesses of glasses.

It is well known that the properties of minerals (except for those of the isometric system) vary directionally[18].

It is also possible that a mineral contain impurities which affect its physical properties. Thus the scratch hardness may vary with the direction and with the sample.

An extensive study of the hardness of the calibrating minerals might be desirable. This would include specimens from different geographical localities and scratching in several directions on the principal crystal faces. Provided that specimens of suitable size and perfection could be obtained, this study in itself would furnish a sizeable research project.

To simplify the testing procedure, only one sample of each mineral has been used. This sample has been scratched in one direction on one crystal face. The general scratching procedure was the same as that used for the standard tests on glasses.

The minerals, their sources, the test faces, and directions of scratching are given:

Mineral	Mohs Scale No.	Geographic Source	Crystal Face Scratched	Direction of Scratching
Fluorite	4	Southern Illinois	111	Perpendicular to trace of cleavage
Apatite	5	Austria	$1\bar{1}00$	Parallel to a-axis
Orthoclase	6	Madagascar	001	Parallel to a-axis
Quartz	7	Arkansas	$1\bar{1}00$	Parallel to a-axis

These minerals were chosen for calibration purposes because previous investigators had found that the hardness of glass falls within the included range.

In observing the scratches made on these minerals, it was

found that all, except fluorite*, tend to chip more than glasses. To get any scratch width values for loadings in the range of thirty to one hundred grams, it was necessary to make measurements on regions which would not have been used if the material had been one of the test glasses. To make these measurements, the procedure suggested by Hodge and McKay[19] was followed; namely, the measurements were made at points where the widths of the chipped area seemed to be the least.

After the measurements had been made, the usual mathematical treatment (see the next section) was applied to the results. When the squares of the scratch widths had been plotted against the load on the scratching point, smoothed curves were drawn as shown in Figure 4a. Points were taken from these curves and their square roots were replotted against the Mohs scale number of the mineral as shown in Figure 4b. The Curves of Figure 4b were used to translate scratch width values into hardness numbers.

In examining Figure 4b, it may be noted that there are inflections in the curves for the three heavier loadings. It will be seen also that if the scratches on fluorite were of greater width the inflections would not be found. There is no reason to believe that there should be a definite relation between the widths of scratches made on the minerals of the Mohs scale because of its arbitrary nature. However if there seems to be some orderly relation with the lower loadings (10, 20,

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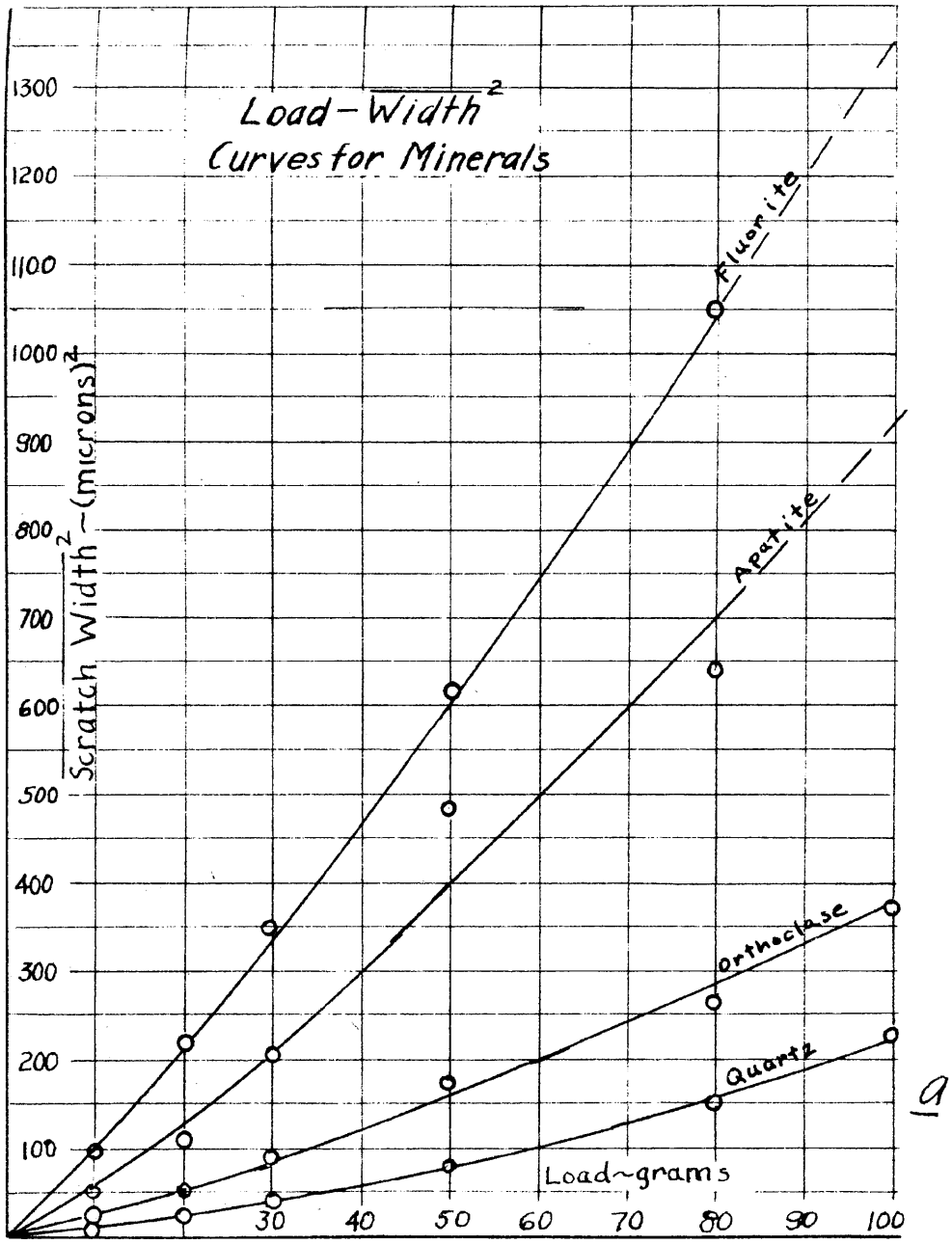
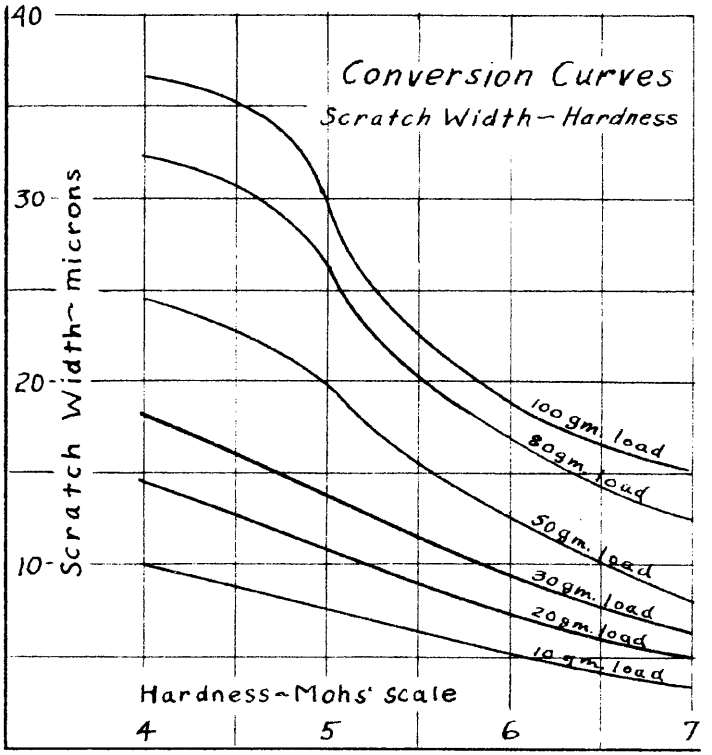
*No chipping was evident for fluorite with loads up to 80 grams.

Figure 4


Load – Width² Curves for Minerals
Scratch Width² – (microns)²
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
Fluorite
Apatite
Orthoclase
Quartz
30
40
50
60
70
80
90
100
Load~grams

a


Conversion Curves
Scratch Width – Hardness
Scratch Width~ microns
40
30
20
10
100 gm. load
80 gm. load
50 gm. load
30 gm. load
20 gm. load
10 gm. load
Hardness~Mohs' scale
4
5
6
7

b

and 30 grams) on the scratching point this would be expected to continue in the higher ranges of loading. Mention has been made of the marked tendency of apatite, orthoclase, and quartz to chip more than fluorite. It was observed that the tendency to chip increased with the load. An attempt will be made later to show that, in the case of glass, the action of chipping is to increase the scratch width. Had the fluorite chipped as did the other minerals, the orderly relation shown for the lower loadings (Figure 4b) might be found for the loadings of 50, 80, and 100 grams on the scratching point. Tammann[20] characterizes fluorspar (fluorite) as plastic. This would account for its behavior being different from the other, brittle minerals with respect to chipping.

3. Method of Calculation

As stated above, the width of each scratch was measured at a number of points using the micrometer microscope. For any one sample the values obtained with equal loadings on the scratching point were averaged. The standard deviation and probable error of any one observation were found by standard methods. The variations of the observations from the mean were examined by use of Chauvenet's[21] criterion of rejection, and values of too great divergence were discarded. Finally the probable error of the mean was calculated. Student's[22] factors were used in cases where less than thirty observations were available.

Where the rejection of any value was indicated, the whole series was examined for uniformity, and, if several values

were found to lie near the limit permitted from the calculation, no rejection was made. However if the value whose rejection was indicated was clearly alone in its eccentricity, there was no hesitation in eliminating it from the group. In the cases where rejection was carried out it was assumed that an error had been made in observing the scratch boundary at the time of measurement.

The means of the micrometer measurements and the probable errors of the means were transformed to micron units by multiplying with the factor 12.72. After the scratch width (in microns) had been found it was changed to Mohs scale hardness by referring to the curves of Figure 4b.

The hardness value for the sample was found by averaging the values for each of the different loadings. The system of weighting suggested by Mellor[23] was used in obtaining the average. The hardness value for each loading was weighted by the amount of the reciprocal of the square of the percent probable error of the mean of the scratch width. As used in this investigation, this method of weighting depended on dispersion, number of observations, and ratio of the size of the dispersion to the size of the measurement.

The value which resulted from these calculations was considered to be an expression of the hardness of the sample.

4. General Considerations

a. Effect of Speed of Scratching: While no extensive study of the effect of speed was made, there were some observations of scratches made with a motor speed of 65 r. p. m.

There was no evident difference between the widths of these scratches and those made at the standard speed of 100 r. p. m.

Since time is an important element in the testing of glassware[24], increasing the speed of scratching might be expected to result in a reduced scratch width.

b. Effect of Shape of Scratching Point: Scratch hardness tests are not standardized. In terms of scratch width, the results of each test depend upon the geometric shape and the size of the angles of the point.

In addition to the standard scratching point used throughout the investigation, another point, made by three planes at and angle of 107°, was obtained. The widths of the scratches made by these two points are tabulated:

Load on Point grams	Scratch Width - microns 130° point	107° point
10	6.69	6.36
20	9.28	8.54
30	11.45	9.54
50	15.33	13.45

A series of tests was made with the 130° diamond, running the carriage in reverse of its usual direction, so that a flat face rather than an intersection of planes met the oncoming glass. The scratch widths obtained in these tests were almost identical with those from the regular procedure when loadings of 10, 20, and 30 grams were used. The same sample of glass was used for all three series of measurements.

The depths of the scratches for the 130° and the 107° points were calculated to be 0.163 and 0.274 of the width, respectively.

One of the earliest observations made in this investigation was that the square of the scratch width was nearly proportional to the load on the point (see Figure 5). This relation has been used in obtaining the calibration curves (Figure 4a).

Some attempts were made to analyze the action of the points in forming the scratches. The comparisons of the results from different procedures did not lead to any definite conclusions.

It was found that the scratch made with the 130° diamond was markedly less chipped when the flat face met the oncoming glass. Peters[25] reported that a diamond tool forming a shallow scratch gave the best ruling for optical gratings. This tool was made with an angle of about 135° at the bottom of the scratch, and the leading face was flat and inclined at about 40° to the vertical.

c. The Effect of Chipping: Figure 5 shows graphically the relation between the squares of the scratch widths obtained for the 130° diamond point with different procedures. The upper curve was drawn by plotting the squares of the scratch widths resulting from the standard procedure. The lower curve shows a similar plotting of values obtained when the direction of the motion of the sample with respect to the scratching point is reversed. Lesser loadings produce identical curves because there is no chipping. As the loading is increased, the chipping becomes more evident with the standard procedure, and the two curves separate.

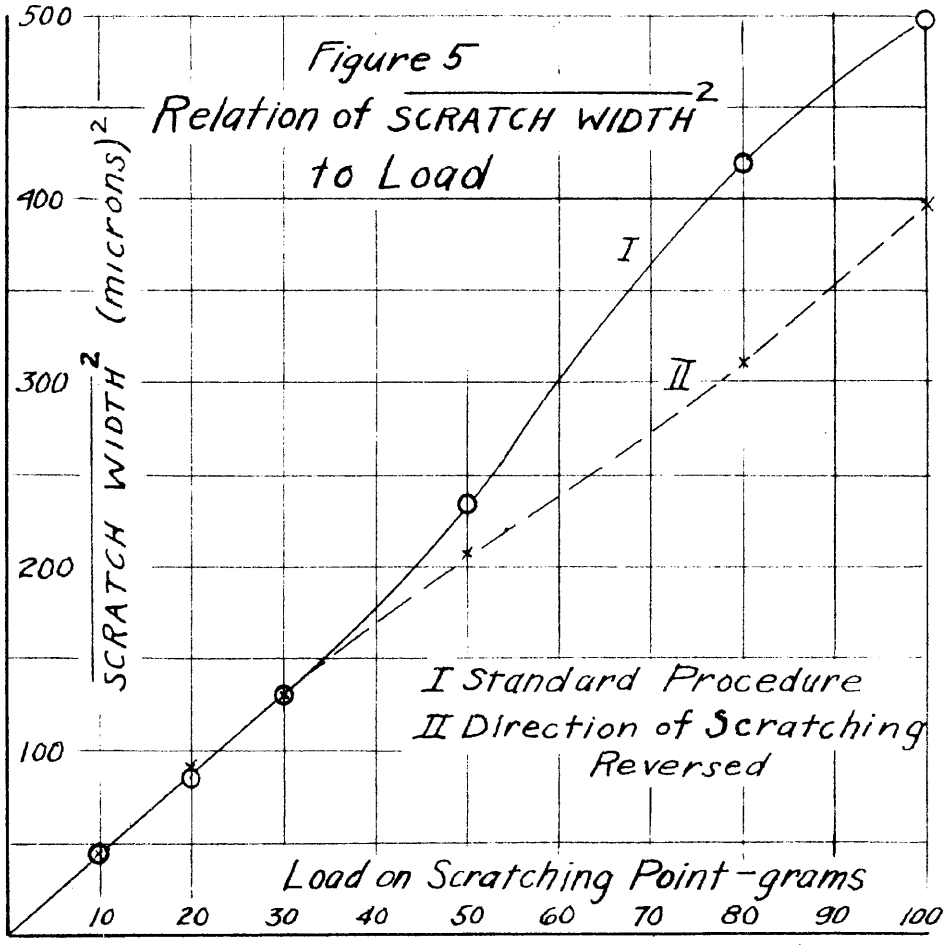


Figure 5
Relation of $\overline{\text{SCRATCH WIDTH}}^2$ to Load
$\overline{\text{SCRATCH WIDTH}}^2$ (microns)^2
500
400
300
200
100
I
II
I Standard Procedure
II Direction of Scratching Reversed
Load on Scratching Point - grams
10
20
30
40
50
60
70
80
90
100

The development of chipping with increasing loads is shown in the photographs of Figure 6. The first shows a scratch made with a loading of ten grams. There is no evidence of chipping. The second shows the type of chipping which develops in this particular sample when the load is increased to twenty grams. The third photograph shows a badly chipped scratch made with a load of fifty grams on the point. The fourth photograph shows the end of a badly chipped scratch. The crack leading from the end of the scratch indicates how the chipping is started. If the glass is fractured ahead of the scratching point, then it is not surprising that the scratch is wider where chipping occurs.

8. Effect of Wear on the Diamond Point: In making a test of scratch hardness, the effect of wear on the scratching point must always be considered. The diamond point used in this investigation was examined periodically for evidence of wear. A microscope was used for this purpose. The appearance of the point after the completion of the scratching tests is shown in Figure 7. This may be compared with Figure 2 which shows the same view of the point before the tests were begun.

In addition to the optical examinations, periodic scratch hardness tests of sample No. 4 were made. The results from some of these tests are given:

Sample No 4

Test made after regular test No.	Hardness
100	5.54
200	5.52
250	5.53

Scratch made with 10 grams load on scratching point. No chipping.

Scratch made with 20 grams load on scratching point. Slight chipping.

Scratch made with 50 grams load on scratching point. Badly chipped but path of diamond is visible.

End of badly chipped scratch. Note crack which preceded the scratching point.

Figure 6
Magnification approx. 650 X

Figure 7

Silhouette of scratching point perpendicular to intersection of two planes. Photograph made at the completion of the scratching tests. Magnification approximately 80 X.

The amount of wear which occurred made no material difference in the results of the tests.

e. The Relation Between Scratch Width and Load on the Scratching Point: Figure 5 shows the relation between the square of the scratch width and the load on the scratching point. Figure 8 shows the relation of width to load. The form of the curve of Figure 5 suggests that the relation shown in Figure 8 is parabolic in nature although there is departure from a true parabola.

The very short dotted branch of Figure 8 shows the form of curve which resulted from some measurements made with small loads in the early part of the investigation. This form of curve could result from a very thin, soft surface layer on the glass. The relatively high error of measurement of such a narrow scratch leads one to question the significance of the observation.

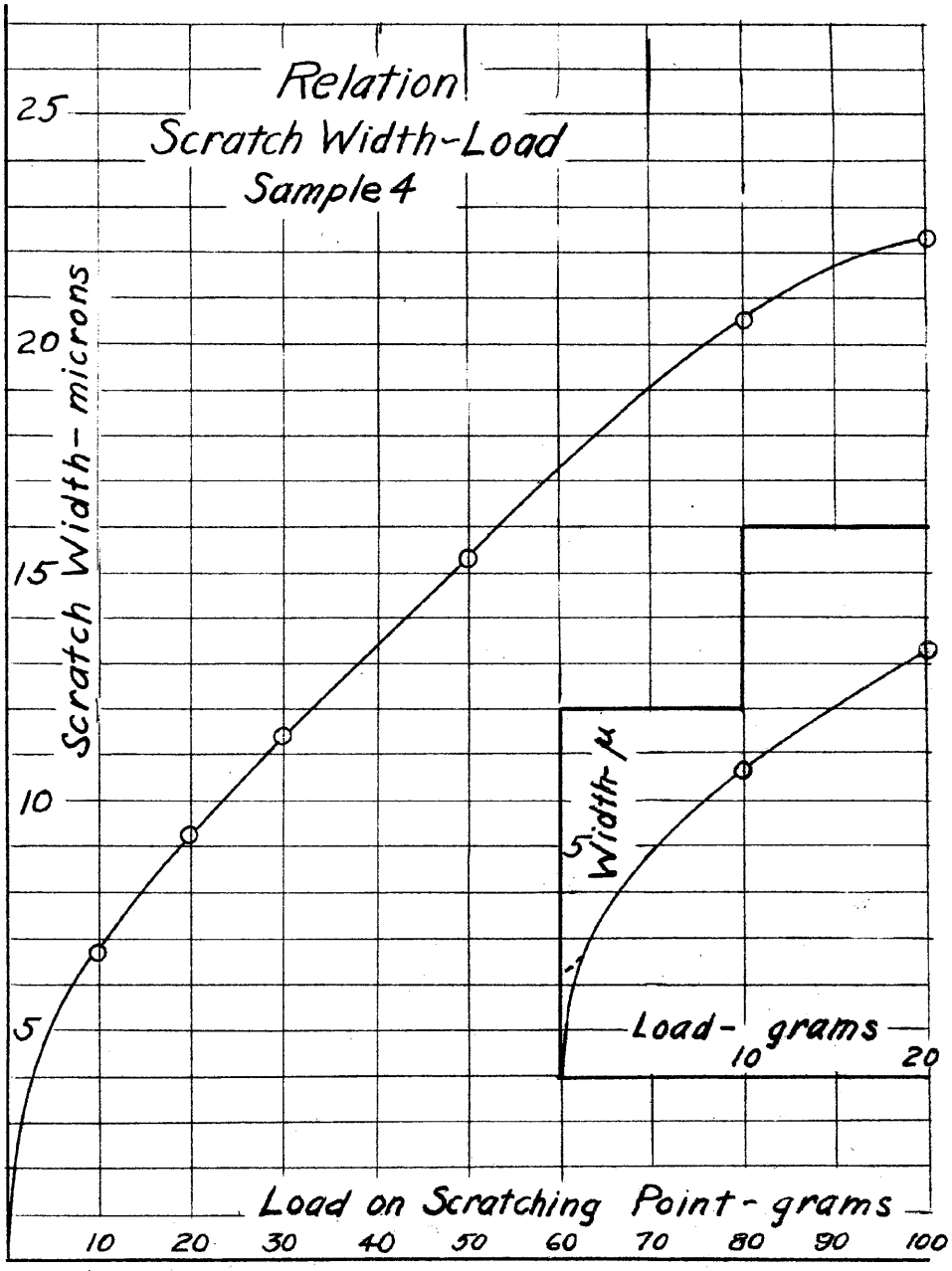

Relation
Scratch Width-Load
Sample 4
Scratch Width- microns
25
20
15
10
5
Width-μ
5
1
Load- grams
10
20
Load on Scratching Point - grams
10
20
30
40
50
60
70
80
90
100

Figure 8

5. Description of Samples

A listing of the samples tested in this investigation will be found immediately below. The compositions of those samples which are marked with an asterisk (*) may be found following the general listing.

Sample Number	Description
1*	Rolled, unpolished plate glass. Tested on the rough surface.
2*	The same as No. 1. Tested on a smooth edge produced by breaking.
3*	The same as No. 1. Rouge-polished in the laboratory. Tested on the rouge-polished surface.
4*	Polished plate glass. Tested on the rouge-polished surface.
5*	Polished plate glass. Tested on the rouge-polished surface after it had been coated with grease.
6*	Polished plate glass. Tested on a smooth edge produced by breaking.
7*	Fire-polished glass. Tested on the fire-polished surface.
8*	Unpolished plate glass, original surface abraded away. Tested on the surface produced by treating for 45 seconds with a solution of sulfuric acid (1 part) and hydrofluoric acid (2 parts).
9*	Polished glass surface produced by rolling quickly with a polished metal roll. Tested on the polished surface.
10*	The same as No. 9. Tested on a smooth edge produced by breaking.
11*	Fire-polished window glass used for modulus of rupture tests. Tested on the fire-polished surface.
12*	The same as No. 11. Strained by quick cooling from low red heat. Tested on the fire-polished surface.
13*	Cast and polished glass. Tested on the rouge-polished surface.

Sample Number	Description
14*	Flint bottle glass. Tested on the mold surface.
15*	Flint bottle glass. Tested on the fire-polished surface.
16*	Amber bottle glass. Tested on the mold surface.
17*	Amber bottle glass. Tested on the fire-polished surface.
18*	Green bottle glass. Tested on the mold surface.
19*	Green bottle glass. Tested on the fire-polished surface.
20	Clear glass taken from a water pitcher. The surface tested was formed against an iron mold.
21	The same glass as that of Sample 20. The surface tested was formed against a paste mold.
22	Polished plate glass received for modulus of rupture tests. Tested on the rouge-polished surface.
23	Polished plate glass received for modulus of rupture tests. Tested on the rouge-polished surface.
24*	Optical glass received from the National Bureau of Standards. Tested on a rouge-polished surface.
25*	Optical glass received from the National Bureau of Standards. Tested on a rouge-polished surface.
26*	Optical glass received from the National Bureau of Standards. Tested on a rouge-polished surface.
27*	Optical glass received from Bailey and Sharp Co., Hamburg, N. Y. Tested on a rouge-polished surface.
28*	Optical glass received from Bailey and Sharp Co. Tested on a rouge-polished surface.
29*	Optical glass received from Bailey and Sharp Co. Tested on a rouge-polished surface.
30*	Optical glass received from Bailey and Sharp Co. Tested on a rouge-polished surface.

Sample Number	Description
31* 32* 33* 34* 35*	Experimental glasses made in the laboratories of the Department of Ceramic Engineering. In this series amounts of alumina were added to a base glass of the type used for making bottles. All were tested on a fire-polished surface.
36* 37* 38* 39* 40* 41*	Experimental glasses received from Bailey and Sharp Co., Hamburg, N. Y. The group was selected to show the effect of variation of composition upon the hardness. All were tested on a fire-polished surface.
42	Rod of chemical glassware. Tested on the fire-polished side surface.
43	Rod of chemical glassware. Tested on the end on the smooth surface produced by breaking.
44	Tempered sheet glass. Tested on the polished surface.
45	Clear rod of fused silica. Tested on the fire-polished surface.
46	Green, translucent condensation product. Tested on the smooth surface.
47	Clear, transparent condensation product. Tested on the smooth surface.
48	Red sealing wax. Tested on the smooth surface produced by cooling from the melt.

Compositions of the glasses:

The approximate analysis of the glass of samples No. 1 to No. 10 inclusive, was furnished by the manufacturer:

	%
SiO_2	71.76
Na_2O	13.46
CaO	13.01
MgO	0.09
Na_2SO_4	0.87
$NaCl$	0.07
Al_2O_3	0.26
Fe_2O_3	0.48
	100.00

The analysis of the glass of samples No. 11 and 12 was furnished by the manufacturer:

	%
SiO_2	72.43
Na_2O	13.62
CaO	9.84
MgO	3.20
Na_2SO_4	0.59
NaCl	0.10
Al_2O_3	0.16
Fe_2O_3	0.06
	100.00

The approximate analysis of the glass of sample No. 13 was furnished by the manufacturer:

	%
SiO_2	70.80
Na_2O	7.82
CaO	12.05
K_2O	6.87
Na_2SO_4	0.35
NaCl	0.88
Sb_2O_5	1.06
Fe_2O_3	0.04

The analyses of the glasses of samples 14 to 19 inclusive, were furnished by the manufacturer:

	Percentage Composition		
Sample No.	14-15	16-17	18-19
Silica	73.90	71.73	71.73
R_2O_3	0.46	1.55	1.16
Calcium	5.88	9.12	5.82
Magnesium	4.06	0.08	4.18
BaO	0.47	---	0.46
Na_2O	15.18	17.36	16.41

The analyses of the glasses of samples 24, 25, and 26 have been calculated from their batch compositions. They were furnished by the National Bureau of Standards.

Sample No.	Percentage Composition 24	25	26
SiO_2	39.0	66.6	68.5
Na_2O	3.0	9.8	11.0
K_2O	4.0	5.9	5.0
PbO	49.0	- -	- -
BaO	- -	7.8	10.6
ZnO	- -	1.9	1.5
CaO	4.0	- -	- -
As_2O_3	- -	0.2	0.4
B_2O_3	- -	7.8	3.0
Sb_2O_5	1.0	- -	- -

The analyses of the glasses of samples 27 to 30, inclusive, have been calculated from their batch compositions. They were furnished by Bailey and Sharp Co., Hamburg, N. Y.

Sample No.	Percentage Composition 27	28	29	30
SiO_2	56.0	48.0	40.0	27.0
R_2O_3	- -	1.0	3.0	0.0
CaO	- -	- -	- -	- -
PbO	8.5	- -	- -	70.0
ZnO	8.5	9.0	9.0	- -
B_2O_3	- -	4.0	6.0	- -
K_2O	8.0	7.0	- -	3.0
Na_2O	5.0	1.0	- -	- -
BaO	14.0	30.0	42.0	- -

The analyses of the glasses of samples No. 31 to 35, inclusive, were made by Bailey and Sharp Co., Hamburg, N. Y.

Sample No.	Percentage Composition 31	32	33	34	35
SiO2	73.50	70.92	69.90	69.73	69.00
CaO	6.07	5.88	5.75	4.95	5.61
MgO	4.38	4.14	3.92	3.60	3.90
R2O3	0.35	3.36	4.75	6.33	7.71
Na2O	15.68	15.70	15.68	15.39	13.78

The analyses of the glasses of samples No. 36 to 41, inclusive, have been furnished by the Bailey and Sharp Co.

Sample No.	Percentage Composition 36	37	38	39	40	41
SiO_2	72.7	72.5	72.7	72.6	71.6	72.5
R_2O_3	1.2	0.7	0.7	1.5	2.3	2.3
CaO	10.7	11.2	12.9	10.5	10.6	11.5
Na_2O	15.4	15.6	13.7	15.4	15.3	13.7

B. MODULUS OF RUPTURE

The apparatus shown schematically in Figure 3 and described on page 10, was used for the determination of the modulus of rupture of the several glasses. Before using the apparatus, the ratio of the load on the end of the beam to the load on the specimen was determined. This was found to be 1.000 to 10.00. The machine was set so that there would be no load on the test piece when the loading vessel was empty.

The glasses used in the modulus of rupture tests were received from two manufacurers and are described as Samples No. 4, 11, 22, and 23 in the listing beginning on page 33. They were in the form of strips one inch (2.5 cm.) in width, eight inches (20.3 cm.) in length, and of different thicknesses. These samples apparently had been cut from larger sheets of glass by scratching and breaking. When received each piece was separated from its neighbor by a sheet of paper, and care was taken not to touch the samples near the center before they had been broken.

To make a test, the glass specimen was placed in the holding device, and water was admitted to the loading vessel at a

predetermined rate. When the sample had broken, the pieces were removed and the breadth and the thickness of the glass at the point of breaking were measured with a micrometer. The weight of water in the receiving vessel was found to the nearest 1/2 ounce (14 grams).

From the data obtained the modulus of rupture was calculated by the formula:

$$M = \frac{3}{2} \frac{P\ l}{b\ d^2}$$

in which P was the load (in pounds), l the length of span (7 inches), b the breadth of the sample (in inches), d the thickness of the sample (in inches). The modulus of rupture has been converted to equivalent units of the metric system in reporting the results.

Since the specimens were not of the same thickness an attempt was made to apply the stress rather than the load at a uniform rate. The average rate of application of stress was 3010 lb. per sq. in. per minute. Rates actually used for each series of samples will be given in reporting the results of the tests.

Statistical values were calculated for the modulus of rupture data. Rejections were made on the basis of Chauvenet's criterion.

V. RESULTS

A. SCRATCH HARDNESS

The results of the determinations of hardness for the several glasses tested in this investigation are tabulated below. The mean scratch width, the probable error of the mean, the number of scratches made, the total number of measurements made, the Mohs scale hardness, and the average Mohs scale hardness are given. The weighting procedure described in the previous chapter was used in arriving at an average value for hardness.

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measurements	Mohs Scale Hardness Value	Average Hardness Mohs Scale
1	10	6.26	0.053	11	108	5.60	5.63
	20	9.06	0.051	12	104	5.54	
	30	11.01	0.075	11	84	5.65	
	50	14.73	0.056	4	34	5.65	
	80	18.82	0.069	2	17	5.66	
	100	21.62	0.067	2	15	5.61	
2	10	6.02	0.067	3	21	5.60	5.66
	20	9.10	0.069	4	34	5.53	
	30	10.77	0.080	3	21	5.71	
	50	14.14	0.114	4	34	5.74	
	80	18.61	0.118	4	34	5.70	
	100	21.61	0.241	4	20	5.61	
3	10	6.65	0.050	3	21	5.44	5.52
	20	9.57	0.055	3	21	5.39	
	30	11.35	0.068	3	21	5.58	
	50	15.26	0.073	3	21	5.58	
	80	19.64	0.122	3	9	5.56	
	100	22.67	0.197	3	7	5.46	
4	10	6.69	0.030	20	153	5.43	5.52
	20	9.28	0.025	21	152	5.47	
	30	11.45	0.024	20	137	5.55	
	50	15.33	0.032	17	119	5.57	
	80	20.48	0.066	14	73	5.45	
	100	22.32	0.145	14	37	5.53	

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measurements	Mohs Scale Hardness Value	Average Hardness Mohs Scale
5	10	6.74	0.058	3	20	5.40	5.64
	20	9.63	0.052	3	20	5.38	
	30	11.24	0.043	3	20	5.60	
	50	14.49	0.123	3	9	5.69	
	80	18.66	0.041	3	17	5.69	
	100	20.99	0.101	3	11	5.70	
6	10	6.46	0.050	3	20	5.52	5.62
	20	9.52	0.050	3	20	5.41	
	30	10.99	0.043	3	20	5.66	
	50	14.57	0.075	3	16	5.68	
	80	18.90	0.156	3	15	5.66	
	100	21.42	0.335	3	4	5.63	
7	10	6.75	0.042	4	21	5.40	5.47
	20	8.99	0.057	4	26	5.55	
	30	12.15	0.155	4	19	5.40	
	50	16.11	0.087	4	28	5.45	
	80	18.80	0.436	4	20	5.67	
	100	21.61	0.607	4	20	5.75	
8	10	6.10	0.100	1	8	5.65	5.48
	20	9.04	0.100	3	21	5.54	
	30	10.80	0.102	1	8	5.70	
	50	16.13	0.167	3	21	5.45	
	80	20.61	0.192	3	21	5.44	
	100	23.86	0.256	3	11	5.37	
9	10	6.05	0.035	8	92	5.68	5.74
	20	8.69	0.046	10	84	5.64	
	30	10.93	0.064	8	52	5.67	
	50	14.70	0.116	6	47	5.66	
	80	17.71	0.084	3	19	5.85	
	100	20.22	0.124	3	17	5.80	
10	10	7.08	0.048	3	23	5.36	5.64
	20	9.83	0.147	3	20	5.32	
	30	11.39	0.068	3	18	5.57	
	50	15.30	0.163	3	17	5.57	
	80	18.86	0.128	3	18	5.66	
	100	21.41	0.237	3	7	5.63	
11	10	6.28	0.048	15	102	5.59	5.61
	20	8.98	0.040	14	101	5.56	
	30	11.14	0.030	15	99	5.62	
	50	14.88	0.037	13	81	5.63	
	80	19.24	0.065	9	45	5.61	
	100	22.39	0.084	9	40	5.52	

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measurements	Mohs Scale Hardness Value	Average Hardness Mohs Scale
12	10	6.29	0.064	3	21	5.58	5.46
	20	9.49	0.042	3	20	5.42	
	30	11.94	0.079	3	20	5.45	
	50	15.45	0.119	3	6	5.55	
	80	-- --	- ---	3	0	- --	
	100	-- --	- ---	3	0	- --	
13	10	6.39	0.033	9	71	5.54	5.61
	20	9.00	0.049	9	69	5.55	
	30	10.91	0.044	9	62	5.68	
	50	14.58	0.056	6	39	5.68	
	80	18.52	0.058	6	38	5.58	
	100	21.45	0.099	6	32	5.63	
14	10	6.49	0.061	6	41	5.51	5.60
	20	8.65	0.076	6	42	5.65	
	30	10.87	0.070	6	42	5.69	
	50	15.00	0.065	6	41	5.62	
	80	19.87	0.137	6	35	5.52	
	100	21.78	0.090	6	19	5.59	
15	10	6.80	0.030	9	74	5.38	5.55
	20	9.33	0.030	9	65	5.46	
	30	11.17	0.039	9	62	5.62	
	50	15.03	0.061	8	58	5.61	
	80	19.85	0.153	6	42	5.53	
	100	22.70	0.089	6	33	5.48	
16	10	6.12	0.036	5	49	5.65	5.65
	20	8.75	0.049	6	44	5.62	
	30	10.83	0.063	6	39	5.69	
	50	14.40	0.056	4	21	5.70	
	80	18.89	0.148	3	20	5.56	
	100	21.49	0.094	3	12	5.62	
17	10	6.22	0.053	5	41	5.61	5.77
	20	8.83	0.071	5	41	5.60	
	30	11.52	0.078	5	36	5.54	
	50	12.89	0.066	4	21	5.93	
	80	17.10	0.131	3	21	5.95	
	100	19.89	0.143	3	18	5.86	
18	10	6.48	0.042	5	41	5.51	5.52
	20	9.56	0.055	5	41	5.40	
	30	11.56	0.041	5	40	5.53	
	50	15.50	0.076	5	36	5.54	
	80	19.75	0.436	3	5	5.54	
	100	-- --	- ---	3	0	- --	

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measurements	Mohs Scale Hardness Value	Average Hardness Mohs Scale
19	10	6.43	0.061	8	63	5.52	5.47
	20	9.66	0.066	8	62	5.37	
	30	11.78	0.075	8	61	5.48	
	50	15.99	0.071	8	68	5.47	
	80	20.25	0.130	6	35	5.48	
	100	22.90	0.115	6	37	5.46	
20	10	5.60	0.045	3	21	5.86	5.73
	20	8.53	0.075	3	22	5.69	
	30	9.92	0.095	3	24	5.90	
	50	14.34	0.117	3	22	5.71	
	80	18.79	0.153	3	4	5.67	
	100	21.18	0.113	3	12	5.67	
21	10	5.82	0.065	3	21	5.77	5.61
	20	9.03	0.071	3	21	5.54	
	30	11.55	0.110	3	20	5.53	
	50	14.43	0.182	3	15	5.70	
	80	18.89	0.654	3	5	5.66	
	100	20.93	0.594	3	2	5.70	
22	10	6.98	0.060	3	21	5.31	5.46
	20	9.91	0.055	3	21	5.30	
	30	11.64	0.085	3	16	5.51	
	50	15.26	0.097	3	13	5.58	
	80	19.40	0.164	3	2	5.59	
	100	21.29	0.245	3	4	5.64	
23	10	6.78	0.059	3	21	5.39	5.53
	20	9.76	0.069	3	21	5.34	
	30	10.94	0.121	3	21	5.67	
	50	15.11	0.091	3	16	5.60	
	80	19.30	0.123	3	15	5.60	
	100	21.52	0.192	3	5	5.62	
24	10	7.55	0.043	2	14	5.08	5.32
	20	10.34	0.041	3	21	5.17	
	30	12.70	0.112	2	14	5.27	
	50	16.21	0.076	3	20	5.41	
	80	22.09	0.158	2	12	5.38	
	100	26.08	0.161	2	10	5.18	
25	10	5.34	0.047	3	21	5.96	5.82
	20	8.38	0.076	3	21	5.74	
	30	9.79	0.074	3	21	5.94	
	50	14.26	0.067	3	20	5.73	
	80	16.75	0.456	3	3	5.84	
	100	-- --	- ---	3	0	- --	

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measure-ments	Mohs Scale Hardness Value	Average Hardness Mohs Scale
26	10	6.36	0.047	6	42	5.56	5.62
	20	9.28	0.044	6	42	5.48	
	30	10.64	0.048	6	41	5.74	
	50	15.00	0.055	6	41	5.62	
	80	19.00	0.499	6	26	5.64	
	100	-- --	- ---	6	0	- --	
27	10	6.41	0.041	3	20	5.54	5.65
	20	9.02	0.062	3	19	5.54	
	30	10.87	0.036	3	20	5.68	
	50	14.48	0.050	3	15	5.69	
	80	-- --	- ---	3	0	- --	
	100	-- --	- ---	3	0	- --	
28	10	6.64	0.030	3	19	5.45	5.60
	20	9.11	0.034	3	21	5.52	
	30	10.82	0.033	3	21	5.69	
	50	14.69	0.099	3	21	5.66	
	80	18.64	0.116	3	10	5.69	
	100	-- --	- ---	3	0	- --	
29	10	6.85	0.022	3	20	5.37	5.50
	20	9.54	0.050	3	20	5.40	
	30	11.40	0.058	3	19	5.56	
	50	15.10	0.076	3	21	5.60	
	80	19.38	0.042	3	20	5.59	
	100	22.23	0.100	3	21	5.53	
30	10	8.04	0.022	3	21	4.87	4.95
	20	11.47	0.048	3	21	4.86	
	30	14.33	0.052	3	20	4.91	
	50	19.02	0.075	3	21	5.11	
	80	25.19	0.127	3	19	5.05	
	100	28.15	0.190	3	15	5.06	
31	10	6.65	0.057	3	20	5.42	5.42
	20	9.24	0.103	3	21	5.49	
	30	12.14	0.070	3	20	5.40	
	50	16.36	0.126	3	21	5.42	
	80	20.27	0.675	3	20	5.48	
	100	24.87	0.856	3	3	5.28	
32	10	6.83	0.092	3	20	5.37	5.51
	20	9.14	0.171	3	20	5.51	
	30	12.42	0.125	3	20	5.33	
	50	16.13	0.287	3	17	5.44	
	80	19.08	0.184	3	7	5.63	
	100	22.27	0.083	3	6	5.53	

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measurements	Mohs Scale Hardness Value	Average Hardness Mohs Scale
33	10	6.58	0.070	4	28	5.47	5.65
	20	8.73	0.091	4	27	5.63	
	30	11.59	0.102	4	23	5.52	
	50	14.83	0.235	4	19	5.64	
	80	17.36	0.150	4	12	5.90	
	100	-- --	- ---	4	0	- --	
34	10	6.23	0.077	3	21	5.60	5.56
	20	8.91	0.065	3	20	5.58	
	30	11.80	0.112	3	18	5.48	
	50	15.25	0.116	3	20	5.58	
	80	17.81	0.302	3	4	5.66	
	100	22.00	0.120	3	6	5.56	
35	10	6.02	0.052	3	21	5.69	5.73
	20	8.18	0.058	3	21	5.79	
	30	11.01	0.089	3	22	5.66	
	50	14.85	0.150	3	10	5.64	
	80	18.29	0.031	3	5	5.75	
	100	21.61	0.167	3	6	5.61	
36	10	6.56	0.081	3	21	5.48	5.48
	20	9.43	0.080	3	22	5.43	
	30	11.73	0.105	3	20	5.49	
	50	14.30	0.282	3	8	5.72	
	80	-- --	- ---	3	0	- --	
	100	-- --	- ---	3	0	- --	
37	10	5.75	0.065	6	42	5.80	5.51
	20	9.32	0.079	6	42	5.46	
	30	12.05	0.075	6	41	5.42	
	50	15.59	0.094	3	21	5.53	
	80	-- --	- ---	3	0	- --	
	100	-- --	- ---	3	0	- --	
38	10	5.80	0.054	3	21	5.77	5.81
	20	8.31	0.082	3	19	5.76	
	30	10.76	0.112	3	12	5.71	
	50	13.84	0.180	3	18	5.79	
	80	16.83	0.147	3	15	5.98	
	100	20.37	0.225	3	6	5.78	
39	10	6.68	0.058	3	19	5.43	5.40
	20	9.92	0.074	3	21	5.30	
	30	11.95	0.096	3	21	5.44	
	50	15.77	0.163	3	9	5.50	
	80	-- --	- ---	3	0	- --	
	100	-- --	- ---	3	0	- --	

Sample Number	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measure-ments	Mohs Scale Hardness Value	Average Hardness Mohs Scale
40	10	6.97	0.026	3	21	5.32	5.38
	20	9.63	0.070	3	23	5.38	
	30	11.64	0.068	3	21	5.51	
	50	16.14	0.159	3	20	5.44	
	80	18.68	0.624	3	4	5.68	
	100	-- --	- ---	3	0	- --	
41	10	6.14	0.116	3	21	5.64	5.58
	20	8.99	0.105	3	20	5.56	
	30	11.05	0.114	3	20	5.64	
	50	15.49	0.131	3	19	5.54	
	80	-- --	- ---	3	0	- --	
	100	-- --	- ---	3	0	- --	
42	50	14.59	0.147	1	22	5.67	5.67
43	50	14.55	0.088	--	30	5.68	5.68
44	10	6.79	0.046	3	20	5.39	5.64
	20	8.89	0.052	3	23	5.58	
	30	10.85	0.057	3	21	5.69	
	50	14.38	0.108	3	22	5.71	
	80	18.80	0.064	3	22	5.67	
	100	21.12	0.096	3	19	5.67	
45	10	4.61	0.065	6	53	6.28	6.31
	20	6.77	0.085	4	22	6.22	
	30	8.36	0.701	4	3	6.80	
	50	- --	- ---	3	0	- --	
46	10	17.20	- ---	1	6	- --	- --
	20	26.8	- ---	1	6	- --	
	30	31.6	- ---	1	6	- --	
47	5	18.2	- ---	1	7	- --	- --
	10	27.3	- ---	1	7	- --	
	15	35.2	- ---	1	6	- --	
	20	45.5	- ---	1	6	- --	
	30	51.7	- ---	1	7	- --	
48	5	23.7	- ---	1	6	- --	- --
	10	32.5	- ---	1	6	- --	
	15	39.5	- ---	1	6	- --	

Sample Tested	Load on Point Grams	Mean Scratch Width Microns	Probable Error of Mean Microns	Number of Scratches Made	Number of Measurements	Mohs Scale Hardness Value	Average Hardness Mohs Scale
Quartz	10	3.02	0.033		20	6.99	7.02
	20	4.51	0.045		21	7.20	
	30	6.38	0.072		20	6.96	
	50	9.09	0.166		19	6.73	
	80	12.48	0.133		18	7.02	
	100	15.10	0.149		9	7.00	
Ortho-clase	10	3.98	0.093		21	6.58	6.04
	20	7.11	0.149		21	6.10	
	30	9.42	0.171		21	6.02	
	50	13.18	0.186		21	5.89	
	80	16.27	0.269		21	6.09	
	100	19.30	0.294		21	5.94	
Apa-tite	10	6.84	0.126		21	5.36	4.98
	20	10.40	0.175		17	5.16	
	30	14.41	0.249		33	4.90	
	50	22.01	0.415		26	4.69	
	80	25.31	0.393		15	5.04	
Fluor-ite	10	9.89	0.077		21	4.03	4.01
	20	14.49	0.080		20	4.04	
	30	18.68	0.154		21	4.00	
	50	24.60	0.202		21	3.97	
	80	32.42	0.249		19	3.99	

B. MODULUS OF RUPTURE

Sample No.	Modulus of Rupture lb/sq in	kg/sq cm	Prob. Error - Mean lb/sq in	kg/sq cm	Number of Specimens	Average Rate of Application of Stress lb/sq in	kg/sq cm
4	10,450	735	557	39.2	12	2875	202
11	10,578	743	393	27.6	36	2908	204
22	7,221	508	223	15.7	12	3000	211
23	7,728	543	484	34.0	24	3160	222

VI. ANALYSIS OF RESULTS

The following will be devoted largely to the comparison of the hardness values for the samples tested. The differences in hardness shown by the results will generally be treated as real and significant, since this is necessary for brevity.

An extended analysis of the significance of the differences in the scratch hardness of the samples has not been made. It might be possible to calculate the probable error of the average hardness value and study the significance of the results with its help. It would be possible to examine the significance of the differences of widths of scratches made with each loading on the scratching point. A difficulty which would arise in this latter case can be illustrated by an example:

The average result for sample 5 shows a substantial increase in hardness over sample 4, yet when the results for the loadings of ten and twenty grams are observed it would appear that No. 4 is harder than No. 5. The hardness values obtained from the higher loadings are the ones which produce the effect shown by the average.

Even though the differences in hardness may not have the desired degree of significance, the results are generally consistent.

There are three evident causes of dispersion in the measurements of this investigation: (1) the difficulty in properly defining the scratch width for measurement, (2) the dispersions arising from measuring technic, and (3) the variations which seem to be characteristic of glass subjected to any test de-

pending on its physical strength.

The difficulty of properly viewing the scratch boundaries has already been discussed.

Dispersions in the measurements are due chiefly to mechanical imperfections in the apparatus and the inability of the eye to sense the precise setting of the cross-hair of the micrometer. To these must be added the effect of focus, because it was necessary to re-adjust the microscope for practically every measurement. A number of measurements were made at identical points on three scratches to form an estimate of the amount of error introduced in the measuring of the scratch widths. The usual care was taken in making the measurements, and the microscope was re-focused before each reading was made. The results are tabulated:

	Mean Scratch Width Microns	Standard Deviation Microns	Number of Measurements
I	6.01	0.259	30
II	8.60	0.214	30
III	11.39	0.228	30

The scratches II and III were better defined than scratch I. To allow a ready comparison, some standard deviation data have been assembled:

Mean Scratch Width Microns	Standard Deviation Microns	Number of Measurements
6.69	0.55	153
9.28	0.44	152
11.45	0.42	137
15.33	0.65	119

Mean Scratch Width Microns	Standard Deviation Microns	Number of Measurements
6.41	0.25	20
9.02	0.25	19
10.87	0.22	20
14.48	0.27	15
5.82	0.42	21
9.03	0.45	21
11.55	0.68	21
14.43	0.98	15
6.46	0.31	20
9.52	0.31	20
10.99	0.26	20
14.57	0.41	16

These values have been selected at random to show that much of the dispersion in the test results can be attributed to the error of measurement. This is particularly true of the narrower scratches. The error of measurement might be reduced by obtaining a better micrometer, and by having absolute rigidity of the optical parts relative to the scratch.

The results of tests of physical strength of glass are prone to show large variations from one sample to another. It would not be surprising if a test of scratch hardness showed similar characteristics.

The tendency of individual glasses to chip may be found by comparing the number of measurements with the number of scratches for a particular loading. When it is considered that the practice was to make seven measurements for each scratch, the recording of less than that number per scratch indicates a tendency to chip. When the chipping was extensive the regions suitable for measurement were reduced in number.

A. PHYSICAL EFFECTS

When the results of the hardness measurements are reviewed the following may be noted:

Rouge-polishing a glass surface softens it. This is shown by a comparison of values obtained for samples 1, 3, and 4. Rouge-polishing also seems to increase the tendency to chip.

Beilby[26] studied the polishing of metals, minerals, and glass. He concluded that the high polish obtained by use of rouge resulted from the flow of the surface. The flow lowers the projections and fills the depressions. Work must be done on the glass surface to produce flow. If work is done, there are bound to be frictional effects which will convert some of the work into heat. A strained material will result if the liquid layer sets and cools to the temperature of the solid body. Hypothetically, at least, a rouge-polished glass surface is under tension.

Galladay and Twyman[27], who studied an optical glass, found that there was a horizontal tension condition immediately below a diamond cut in glass. The type of surface was not specified, but it may be assumed that it was either rouge- or fire-polished. An attempt will be made later to show that there is some similarity between these two types of polished surfaces. Preston[28] was of the opinion that the horizontal stress was due to tiny particles of glass which get into the crack formed by the diamond and act as wedges in maintaining a strained condition. It seems equally likely that the tension condition

which is found just below the diamond scratch is due to local release of strain in the outer layer and concentration in the plane where it has been observed.

Beilby[26] stated that the depth of mechanical disturbance in the polishing of calcite was from 500 to 1000 millimicrons. For the scratches of the present investigation, a simple calculation shows that the depth is 0.16 of the width. The surface layer would be penetrated with a loading of only ten grams on the scratching point. Thus, it develops that the results of the measurements made on the rouge-polished glass express a summation of the hardnesses of the surface layer and of the glass beneath. It is doubtful if the apparatus used for the measurements is sensitive enough to permit a close study of the hardness from the surface inward.

The above discussion attempts to explain the observations of scratch hardness of rouge-polished glass by assuming a surface layer under tension. An alternative explanation of the reason for this softening may be deduced as follows:

It is a common observation that glasses which are quite durable under ordinary conditions undergo a rapid disintegration when treated in the autoclave. In the rouge-polishing process, glasses are subjected to pressure and a somewhat elevated temperature in the presence of moisture. It seems possible that the conditions of the autoclave are found in rouge-polishing. If the surface has undergone any disintegration in the polishing process it cannot be detected by the ordinary means of examination. This does not preclude a hypothesis of sub-micro-

scopic disintegration whose effect is noted in the scratch hardness tests.

The results indicate that a fire-polished surface is softer than one which has been formed in contact with metal. This may be demonstrated in the comparison of values for sample 1 with 7, 9 with 7, 14 with 15, and 18 with 19. An exception is found when sample 16 is compared with 17.

It has been found that when an increase in alkali content of a glass is made, the surface tension falls[29] and the thermal expansion rises[30]. From observations on the behavior of surface tension, it is known that a dissolved substance which causes a lowering of surface tension tends to concentrate at the surface of the solution. The surface layer of a glass, which has been colle from a melt, must be under tension because its thermal contraction is greater in cooling than the contraction of the main body of the glass. This tension is in addition to the force exerted by the surface tension. If this hypothesis of surface strain in fire-polished glasses is admissible then the softening effect of strain can be noted as it was in the discussion of rouge-polished glass surfaces.

In the following section which is devoted to the effects of composition on the scratch hardness of glasses, it is brought out that additions of soda soften a glass. As has been indicated above, the free surface of a glass would be expected to have a higher soda content than the body of the glass. This fact, per se, may furnish adequate explanation of the softening effect of fire-polish.

Hardness tests made on the fractured edge of certain glass samples have been assumed to show the hardness of the glass as it is found in the interior of the body. The glass of the first ten samples listed is supposed to be of the same composition. Hardness tests have been made on the edges of three different samples of this glass. These results are listed for samples No. 2, 6, and 10. While there is not excellent agreement among these values, the general average is 5.64. This approaches the value of 5.63 which was found for sample 1. It would seem that the rolling process used in making plate glass leaves a product whose hardness throughout is nearly uniform. Sample No. 9, another rolled glass, is of superior hardness. From the brief description of the manufacturing process, there is no indication of a reason for this difference.

Comparison of the hardness of sample 8 with sample 1 shows that acid treatment softens this kind of glass.

The surfaces of container glassware are sometimes lubricated to reduce the scratching in the lehr. The results from the study of the hardness of samples 4 and 5 show that lubrication has an appreciable effect in increasing the apparent hardness of glass.

The difference in hardness between a fire-polished surface and one which has been formed in contact with metal (usually iron) has already been mentioned. In addition to the softening effect of fire-polishing, there is a possibility of a hardening effect due to the contact with the metal. By use of x-ray technic, Trillat[31] has found evidence of orientation in the surface structure of cast glass cylinders. This arrange-

ment would result in a more compact structure which would be expected to show an increased hardness.

Samples 20 and 21 show that an article formed in an iron mold is more difficult to scratch than one made in a paste mold, even when the glass composition is identical. From the method of formation it might be suspected that the glass made in the paste mold has a fire-polished surface. This surface, formed in steam, may have different properties from one formed in air. However there is also a possibility that the orienting effect of the mold is minimized by the layers of paste material and steam between it and the glass.

When the results of hardness measurements on samples 11 and 12 are compared, it may be seen that the strained sample is softer and has a greater tendency to chip. In this case the surface layers of the strained sample are under compression, because of the method of preparation. This effect of strain in reducing the hardness of glass is in accord with observations made by Littleton[32] who found that a rapidly moving cotton string produced more wear on a strained sample of glass than it did on an unstrained piece. The composition of the samples was identical, and the test conditions were comparable.

The tests on sample 42 and 43 were made with the thought that if it were possible for the molecules in glass to have an orientation, the effect would be shown by differences in hardness between the side and the end of a rod*. The data show

* Since the units in metals and other substances take up preferred positions when formed into elongated bodies, it was thought that some similar phenomenon might occur in the drawing of glass rods.

that the rod is slightly softer on the side, but this is to be expected from the findings relative to the difference in hardness between fire-polished and fractured surfaces. If an orientation exists, as Trillat found[31], either it cannot be detected by the apparatus used, or it is not produced by the drawing of glass rods.

Sample 44 shows the result of a scratch hardness test on tempered window glass of foreign manufacture. Since the composition is unknown, the value is presented merely as a matter of interest.

The modulus of rupture of a material is a measure of the stress produced in the outermost fibers of a piece broken by bending. Because both the scratch hardness and the modulus of rupture measurements are dependent in a large degree upon the surface condition, it was thought that there might be some correlation between the two.

Sample Number	Modulus of Rupture kg. per sq. cm.	Hardness
4	735	5.52
11	743	5.61
22	508	5.46
23	543	5.53

As can be seen, the correlation is poor.

B. COMPOSITIONAL EFFECTS

When alumina* is added to a glass, the hardness is increased. Samples 31 to 35, inclusive, demonstrate this fact.

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*For the glasses of samples 31 to 41, inclusive, "R_2O_3" reported in the analyses was considered synonymous with "Al_2O_3". The samples 31 to 35 were made with unusual care from c. p. quality materials. The color of samples 36-41 plainly shows that they are low in iron, the other common "R_2O_3" element.

The data for composition and hardness have been plotted in Figure 9. It will be seen that there is a steady rise in the R_2O_3 content and a decrease in the amounts of the other oxides present. There is a break in the hardness curve at the position corresponding to sample No. 34. As will be shown later, this can be ascribed to the irregularity in the CaO (and MgO) composition curve.

The effects of several changes of composition can be noted in the hardness values for the series of samples numbered 36 to 41, inclusive. Rather than devote space to the comparison of the hardness values for different groupings of these glasses, the whole series can be summarized by the presentation of hardness factors for the different constituent oxides.

The relative effect on hardness of the use of one percent of each of the constituent oxides has been found for the group of samples 36 to 41. The factors are:

SiO_2	0.053
R_2O_3	0.147
CaO	0.167
Na_2O	-0.024

These are average values obtained by solution of simultaneous equations. As an example of the method used, the equations for samples 36 and 37, respectively, had the form:

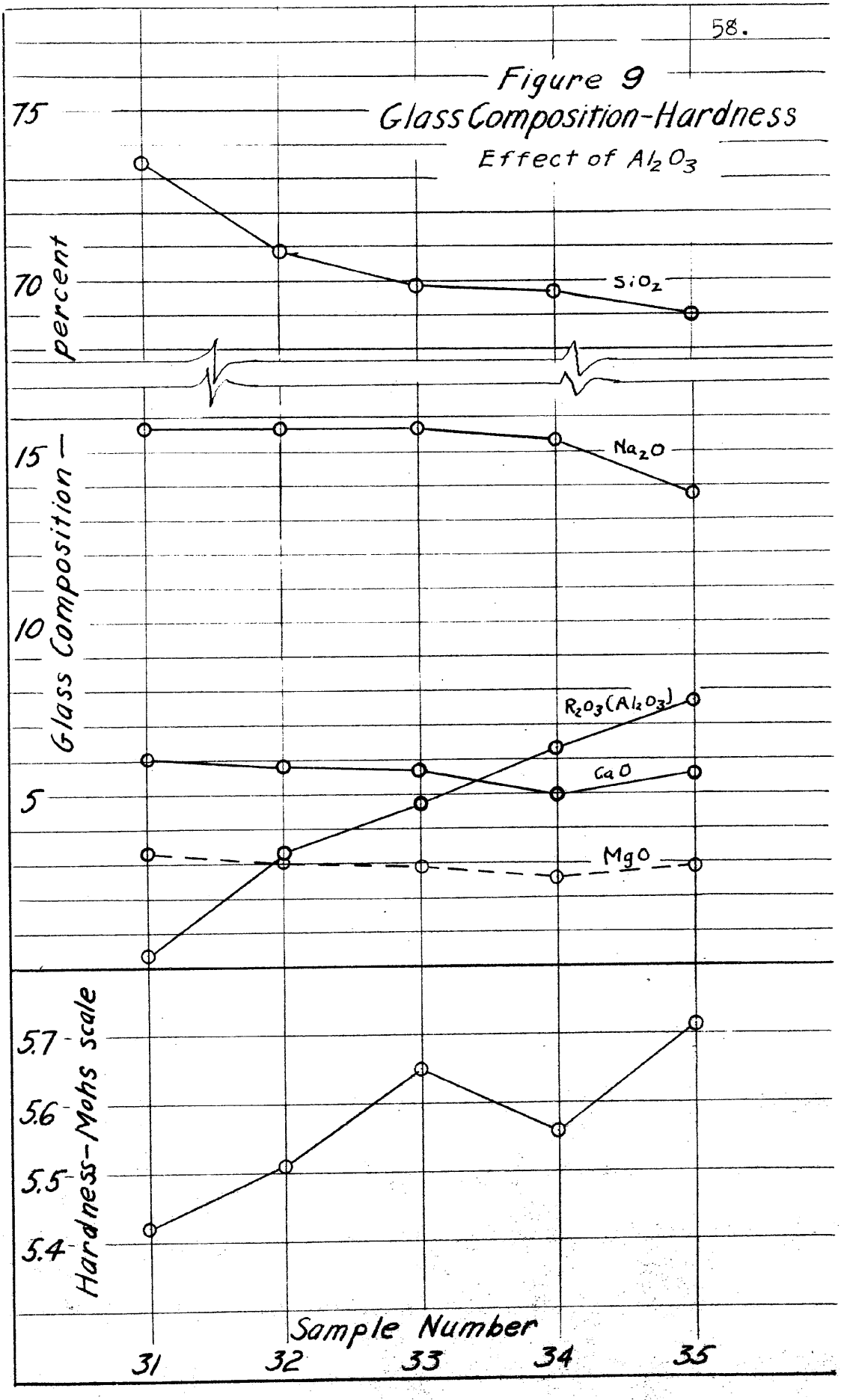
(36) $72.7\ SiO_2 + 1.2\ R_2O_3 + 10.7\ CaO + 15.4\ Na_2O = 5.48$

(37) $72.5\ SiO_2 + 0.7\ R_2O_3 + 11.2\ CaO + 15.6\ Na_2O = 5.51$

Other similar equations were set up and the whole group was solved simultaneously to obtain the factors for SiO_2, R_2O_3, etc.

The use of the factors should be limited to glasses of

Figure 9
Glass Composition-Hardness
Effect of Al_2O_3



the type from which they were calculated. Because of the small change in the amounts of SiO_2, CaO, and Na_2O present in these glasses, it is probably permissible to express their effects in terms of constants. Since the relative change in R_2O_3 is great, its effect should not be expressed as a constant. The proper expression would show a decrease in effect per unit added as the amount becomes greater.

Considering that the factor for Al_2O_3 (R_2O_3) is probably too high for the higher ranges of alumina content, and that lime is quite effective in increasing the hardness of glass, the break in the hardness curve shown in Figure 9 is comprehensible.

The remarks about the relation between composition and hardness are in agreement with the observations on samples 15, 17, and 19. Sample 15 is somewhat harder than No. 31, but there are some differences in composition. The hardness of No. 19 seems to be too high. The R_2O_3 group of the green and the amber glasses cannot be considered to include only Al_2O_3.

The optical glasses included in this study show a wide range of composition. These compositions are reported from the make-up of the batches, and do not represent the analyses of the founded glasses. For these reasons it is difficult to generalize upon the relation between their compositions and hardnesses. However the tendency of lead to decrease the hardness of glasses is unmistakable. The results show that optical glasses are not necessarily soft.

Silica glass has a hardness of 6.31 as shown by sample No. 45 which makes it desirable as an intermediate member between feldspar and quartz in the hardness scale.

As a matter of interest a few measurements were made on substances other than glass. The condensation products and the sealing wax were found to be softer than the range included in the calibration measurements, and no hardness values were obtained for them. Sealing wax is somewhat softer than samples 46 and 47 which were the condensation products. The clear condensation material was quite badly scratched by the relatively light loads on the diamond point. The scratches on the translucent material were of the same order of size, but were not easily visible because of the matt texture.

C. THE HARDNESS OF THE CALIBRATING MINERALS

It has been a matter of observation for a great many years that apatite is harder than fluorite, orthoclase is harder than apatite, and quartz is harder than orthoclase. Indeed, these observations were made by Mohs in setting up his scale of hardness. The results of the present investigation are in agreement with them.

Later years have seen the formation of generalizations relating crystal structure and hardness. Quartz and orthoclase are harder than fluorite because they have a silicate type of lattice and the fluorite lattice is ionic in type. Quartz is harder than orthoclase because the larger K^+ ion of the feldspar distorts the structure and weakens it. Apatite has a lattice of the ionic type but it is more complex than fluorite. The curves of Figure 4a show a wider spacing between the scratch widths of the silicate and ionic minerals than between the minerals of the same type.

Observations of scratch hardness of materials under conditions which are not carefully controlled are likely to lead to erroneous conclusions. When two materials have nearly the same hardness, either may be scratched by the other. This is particularly true when hand scratching tests are made. The following factors are pertinent to the problem:

Shape and perfection of the scratching point

Speed of scratching

Pressure on the point

Angle of inclination of the point to the surface.

In addition to these items there are others which must be considered. A scratch will be more readily visible on a highly polished surface than on one which is slightly roughened. It has been reported[73] that the use of alumina in glass results in a softer product. The true situation seems to be that the addition of alumina to the composition of a glass increases its surface tension and, hence, makes a more perfect surface. Thus scratches become more readily visible to the eye. Even though the size might be the same, a scratch would be more prominent on the surface of a glass of higher index of refraction. Chipping, too, enters into the question. Naturally, a glass which chips badly will seem to be softer than one of equal hardness in which chipping does not occur.

VII. CONCLUSIONS

The results of this investigation seem to justify the following conclusions:

1. The apparatus and the method are generally satisfactory for the determination of the scratch hardness of glasses. An improvement in the micrometer would be of benefit.

2. The hardnesses of the majority of the commercially valuable silicate glasses lie between 5.0 and 6.0 of the Mohs scale.

3. Rouge-polishing, fire-polishing, and straining of glasses are physical processes which result in softening.

4. When alumina is added to bottle glass compositions, there is an increase in hardness. In the general case, alumina is not as effective as lime in increasing the hardness of glass.

5. The following factors express the relative effect on the scratch hardness of a bottle glass of the addition of one percent of the oxide:

SiO_2	0.037
R_2O_3 (Al_2O_3)	0.147
CaO	0.167
Na_2O	-0.024

6. Optical glasses of high lead oxide content are markedly softer than the average bottle or window glass. However optical glasses may be comparatively hard.

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VITA

Kenneth Cassingham Lyon was born in La Harpe, Illinois, January 22, 1906. He attended the public schools of that city and was graduated from La Harpe High School in 1926. He attended Knox College from September, 1926 to June, 1928. The author matriculated at the University of Illinois in 1928 and was awarded the degree of Bachelor of Science, with High Honors in Ceramic Engineering, in 1931.

In September, 1931, he entered the Graduate School of the University of Illinois. During the academic years of 1931-32 and 1932-33, he served as Research Graduate Assistant in the Engineering Experiment Station. He was granted the Master of Science Degree in 1933. The author was appointed University Service Fellow for the academic years 1933-34 and 1934-35.

He is a member of the Society of the Sigma Xi, Phi Kappa Phi, Tau Beta Pi, Keramos, Sigma Tau, Phi Gamma Delta, and the American Ceramic Society.

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