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SUMMARY

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Data on the absorption coefficients of fused silica samples at elevated temperatures are presented. From an initial set of 42 samples, representing two optical grades from one manufacturer and one optical grade from another manufacturer, a few samples, which exhibited "typical" spectral absorption, were selected for measurements at elevated temperatures. Among samples of the same optical grade from a given manufacturer, large variations occurred in the absorption at the ultraviolet absorption edge at 22° C; "typical" samples, as here defined, exhibited absorption coefficients lying close to the mean absorption coefficient of samples with the same optical grade and manufacturer, at a wavelength of 180 millimicrons. The three sample sets were Corning UV grade, Corning Optical grade (Corning Glass Works) and Suprasil (Engelhard Industries).

The measurements were taken over the spectral range 0.17 to 3.5 microns, at 22°, 260°, 538°, 760°, and 982° C sample temperatures. Construction and performance of the optical system, sample furnace, and power supply are described.

The data show a general decrease in transmission with increasing temperature. The ultraviolet cutoff wavelength moves approximately 30 millimicrons toward the infrared between 22° and 982° C. The absorption peak at 1.4 microns moves approximately 10 millimicrons toward the infrared, while passing through a maximum in absorption around 260° C. The absorption peak at 2.2 microns moves some 17 millimicrons toward the infrared, while passing through a minimum. The absorption peak at 2.7 microns moves toward the ultraviolet up to about 260° C, then moves toward the infrared with increasing temperature; it monotonically decreases in absorption between 22° and 982° C.

All the bands broaden with increasing temperature, causing an increase in the fraction of the incident light absorbed by a factor of two to four in the regions between absorption bands.

Author

INTRODUCTION

In applications such as envelopes for high-power xenon lamps, high-temperature photography, or photolysis at elevated temperatures, information on the temperature dependence of the optical transmission of window materials is of great interest. One of the most commonly used window materials in research and industrial apparatus is fused silica. While its optical properties are well known (refs. 1 and 2), data on its absorption spectrum at elevated temperatures have been fragmentary (refs. 3 and 4). Recently, Douglas and Gagosz (ref. 5) have presented absorption spectra (0.15 to 3.5 μ) of four 30-millimeter-thick samples of fused silica of different composition and origins at two temperatures (22° and 800° C) along with ultraviolet absorption spectra of a 2-millimeter sample and a fifth 30-millimeter sample at 22° and 800° C and infrared absorption spectra of a sixth 30-millimeter sample at 22° and 800° C.

The present work extends the investigation of reference 5. Absorption coefficient data from three optical grades of fused silica at five temperatures from room temperature to 982° C in the wavelength range of 0.17 to 3.5 microns are presented. The room-temperature transmittance of a large initial number of samples was measured, and transmittance measurements were taken at elevated temperatures on those which appeared to have "average" optical properties. Both thick and thin samples were included to obtain data in spectral regions where the absorption coefficient is, respectively, very low and very high. Comparison is made with the data of reference 5. Construction and performance details are given on the spectrophotometer and furnace used for these measurements.

The data are presented in the form of separate absorption spectra for the different spectral regions where measurable absorption occurs. A detailed examination of the measured location as a function of temperature of three infrared absorption peaks is included.

SYMBOLS

I intensity of light transmitted through sample

I_o intensity of light incident on sample

n index of refraction

r fraction of incident light lost through reflection at a glass-air interface

t temperature

x path length of light beam through sample, cm

α absorption coefficient, cm^{-1}

λ wavelength, $m\mu$

APPARATUS DESIGN AND PERFORMANCE

Optical System

The optical components of the spectrophotometer used (a modified Beckman DK-IA) for the measurements are shown schematically in figure 1. The source energy is mechanically chopped at 480 cps before entering the sample chamber, and sensed at the detectors by a narrow band pass ac amplifier; thus, any furnace radiation which falls on the photodetector produces a steady signal and is not amplified to affect the recorded signal.

In standard usage, the instrument illuminates the sample with monochromatic light, and the detectors are close to the sample chamber. In this configuration, the furnace irradiance was high enough to drive the detectors into nonlinear operation. The light path was reversed, as seen in figure 1, and the source energy was dispersed after passing through the sample. Thus, the uncollimated furnace irradiance underwent large inverse-square losses in the monochromator, and only a very small fraction of the remaining radiation was passed by the prism. Satisfactory operation was obtained: the performance of the system was the same whether the furnace was at 22° or 982° C.

In all other respects, the operation was that of a standard double-beam instrument, with the source beam split into reference and sample beams in the sample chamber. The data were presented on a strip-chart recording whose abscissa was wavelength, and whose ordinate was the ratio of energy transmitted in the sample beam to that transmitted in the reference beam at each wavelength.

The useful spectral range of the instrument with furnace installed was 170 to 3500 millimicrons. The spectral resolution varied from the order of 5 millimicrons at 2500 millimicrons to the order of 0.01 millimicron at 250 millimicrons. Reproducibility of I/I_0 for the same sample was within 1/2 percent. Long-time wavelength reproducibility (of the order of weeks) was within ±10 millimicrons. The time required to take all the data on a single sample was some 5 hours; however, short-time wavelength reproducibility is determined by the operator's ability to reset the positions of the strip-chart recording and wavelength scroll at the starting positions. This reproducibility was within ±2 millimicrons in the infrared and ±0.2 millimicron in the ultraviolet.

Furnace Design and Performance

The furnace was designed to fit within the available spectrophotometer sample chamber without modification of the chamber. The furnace (fig. 2) consists of an inner metal liner, heating element, insulating wall, and water-cooled outer casing.

One end of the furnace is removable; the cold wall, insulation, heating element, and sample container slide out as a unit for sample insertion. The assembled furnace is held shut by two pins in the floor of the sample compartment, which slide through small metal tabs welded to either end of the furnace. These also serve to position the furnace apertures accurately with respect to the sample and reference light beams.

The liner box, open on one end, and the sample container, which slides into it, were fabricated of 0.065-inch 304 stainless steel. The sample container will accommodate a 1/2-inch-square by 1-inch-long sample in both the sample and reference sides. After being flame sprayed with a 0.010-inch-thick insulating coat of aluminum oxide, these sides were wrapped with the heater element, as shown in figure 3. A nickel-chrome alloy resistant to corrosion in a reducing atmosphere was used for the heater in the form of 0.032-inch wire. The heater assemblies were then overcoated with aluminum oxide, which mechanically bonded the heater wires in place. All surfaces of the sample container were flame sprayed with chromium sesquioxide; its high emissivity minimizes internal optical reflection.

Blocks of insulating firebrick, which has extremely low thermal conductivity ((1.2 Btu/sq ft)/(in.)(hr)(F^{O}) at 482^{O} C), were cut from a single firebrick and cemented onto the liner and the endplate of the sample container with a refractory cement.

The water-cooled outer casing was fabricated from strips of oval cross-section copper tubing, 0.625- by 0.170- by 0.040-inch wall, edge welded and series connected to form a box. The end loops were edge welded around a 1/8-inch-thick copper plate, through which ports for the light beams were cut. The top of the box was formed of a 1/8-inch copper plate covered by more of the flat tubing. The heater components and cold wall are shown prior to assembly in figure 4.

Finally, the insulated liner assembly was cemented into the outer casing; the insulated sample container was cemented and bolted with a single No. 0-80 machine screw to the water-cooled end plate, and the heaters were wired in series, as shown in figure 5.

The heating rate of the empty furnace under full-power startup is illustrated in figure 6. Approximately 25 minutes are required to achieve maximum usable temperature. Figure 7 is a plot of the power requirement as a function of temperature. At 982^{O} C the furnace draws 7 amperes at 36 volts (252 W). The double thickness of sample container and inner liner walls distributes the heat very well: at 927^{O} C the maximum nonuniformity in temperatures across the internal surface is approximately 8^{O} as measured by radiation pyrometer sightings. At water flow of 1 liter per minute, the coolant undergoes only 3.2^{O} temperature rise at 871^{O} C furnace temperature. To check the correspondence between furnace and sample temperature, one 10-millimeter sample was drilled to admit a small thermocouple. The temperature at the center of the sample was within 2.8^{O} C of the furnace temperature 5 minutes after the furnace reached a stable temperature of 871^{O} C.

Furnace Power Supply

A power supply was constructed in which the main furnace current was hand set by one variable transformer, and an increment of control power was added to the output by an indicating pyrometer controller. A Chromel-Alumel thermocouple was the signal source for the pyrometer; a second thermocouple from the furnace was used for generating strip-chart recordings and calibrating the pyrometer. The thermocouples were twisted together and were placed in between the inner walls of the sample container to sense the average temperature of these walls. Control power was added to the output of the main power variable transformer by placing the secondary winding of a filament transformer in series with the main power outlet; the primary winding of the filament transformer was connected to the output of a second variable transformer by the relay in the pyrometer controller. Adding a small control power increment to the main power input yielded good temperature regulation: the furnace temperature is stabilized about 10 minutes after power is applied with a final oscillation of less than 1^{o} at 982^{o} C. A schematic view of the circuitry is shown in figure 8.

SAMPLES AND SAMPLE PREPARATION

Sample Supply and Preparation

Fused silica samples were obtained in two optical grades from each of two vendors as follows:

Code 7940, UV grade; Corning Glass Works
Code 7940, Optical grade; Corning Glass Works
Suprasil I grade; Engelhard Industries
Suprasil II grade; Engelhard Industries

A total of 42 samples were obtained, nominally 1-, 2-, 4-, 10-, and 20-millimeter-thick by 1/2-inch-square cross section. An effort to obtain a broad sampling of average-quality fused silica as actually commercially available was pursued by buying the samples in two orders, some 6 months apart, and by specifying that half the samples within a single order were to be from one ingot and the others from a second ingot. After receipt of all the samples, they were optically refinished by a commercial optical shop to a flatness across the face of 1/4 wavelength of sodium light; the faces were parallel to within 2 wavelengths.

The samples were washed in detergent and distilled water, washed in acetone, rinsed in distilled water, dried, and stored in sealed vials. Subsequently they were handled

only by tweezers whose tips were frequently recoated with collodion to assure their cleanliness.

Sample Selection

Room-temperature absorption-coefficient data were taken on all the samples with an air reference as described in the appendix, using the manufacturers' data on index of refraction. Very little data scatter was apparent in the infrared absorption bands, but considerable scatter from sample to sample was noted at the ultraviolet absorption edge within a given optical grade. Figure 9 illustrates the ultraviolet data so measured along with absorption coefficients computed from the manufacturers' data. No grouping or correlation according to specimen thickness was found in the data.

It should be emphasized that the sample preparation was the same for each sample. Further, the transmission data were taken in rapid succession, which virtually eliminated instrument drift as a possible explanation of the transmission variation; reproducibility of the data curves over a period of 1 hour is within the width of the pen trace. Finally, the sample cleaning procedure was repeated after data at elevated temperature were taken, and the room-temperature data were taken in a different spectrophotometer; these data duplicated the original data. Thus it may be concluded that this is an effect intrinsic to the samples themselves.

Because of the large variations in absorption coefficient, even the relatively large sample set was not statistically significant for the ultraviolet absorption edge. At 180 millimicrons the standard deviation of absorption coefficient was greater than the mean absorption coefficient. Nonetheless, the mean properties are taken to represent an approximation of those of fused silica as presently commercially available.

Figures 9(c) and (d) show that Suprasil I and Suprasil II are essentially the same in the ultraviolet region; they were also the same in the infrared region. Because the optical properties were therefore assumed to remain similar at elevated temperatures, data at elevated temperatures were taken only for Corning UV and Optical grades and for Suprasil I.

The greatest divergence in transmission seems to occur at about 180 millimicrons. Some actual transmission curves for "identical" samples (same optical grade and thickness) are illustrated in figure 10. The transmission data tend to blend at wavelengths longer and shorter than 180 millimicrons. From the data of figure 9, a thin sample and two thicker samples were chosen from each of the three sample sets on the basis of their having absorption coefficients lying close to the mean of the set at 180 millimicron.

Methods of Calculation and Procedure

The thin sample (thickness x_1) was placed in the reference beam in the furnace, and the thicker sample (thickness x_2) was placed in the sample beam in the furnace. In this way, reflection losses were cancelled, and

$$I/I_o = e^{-\alpha(x_2-x_1)}$$

More than just bypassing the labor of subtracting the reflection loss from each point on the curve, this technique avoids the necessity of knowing the index of refraction as a function of temperature and wavelength.

Prior to each session of data taking, the spectrophotometer was warmed up for 1 hour for stabilization. The instrument was continuously purged with pure nitrogen (less than 20 ppm oxygen, 2 ppm water vapor) to prevent contamination of the optics and to minimize light absorption in the instrument itself.

First of all, 0-percent and 100-percent transmission reference lines were run on the chart; then the samples were placed in the furnace and room-temperature transmission data were taken. The furnace was brought up to 260° C, allowed to stabilize for about 30 minutes, and a second transmission curve taken. In succession, data at 538°, 760°, and 982° C were taken. The furnace was then allowed to cool, and a new sample combination was placed in it.

The raw data were a family of transmission curves, as illustrated in figure 11. The transmission read out on the strip chart was then converted to the absorption coefficient, that is,

$$\alpha = -\frac{\ln I/I_o}{x_2 - x_1}$$

RESULTS AND DISCUSSION

The data from the three sample sets are presented in figures 12, 13, and 14. For each sample set, separate data curves are shown for the ultraviolet absorption edge, the weak absorption band at 1.4 microns, and the overlapping absorption bands at 2.2 and 2.75 microns. No data are shown for regions where the absorption coefficient is less than 0.01 centimeter^{-1}.

The 2.75-micron absorption band is generally attributed to the presence of water in the SiO_2 structure, in particular, to O-H vibration. The 2.2-micron absorption band is a combination of frequencies from O-H vibration and Si-OH deformation (ref. 6). Although the differences between Corning grades are slight, the samples may be arranged in the

order of increasing absorption at the water band as follows: Corning UV, Corning Optical, and Suprasil. The Suprasil (fig. 14(c)) contains approximately three times the concentration of water as the Corning fused silica (figs. 12(c) and 13(c)).

The 1.4-micron band is most likely the first harmonic of the 2.75-micron O-H band; again the Corning grades (figs. 12(b) and 13(b)) are almost indistinguishable, but the Suprasil (fig. 14(b)) shows greater absorption.

The ultraviolet cutoff is here defined as that wavelength at which the absorption coefficient reaches 10 centimeter^{-1} (for this value, a 3 mm window would absorb 95 percent of the incident light). This cutoff wavelength is plotted as a function of temperature in figure 15. The cutoff wavelength is essentially the same for Corning UV and Suprasil at a given temperature. The cutoff moves in a fairly smooth but nonlinear fashion toward longer wavelengths with increasing temperature. The slope $d\lambda/dt$ changes around 538^{o} C, becoming larger at higher temperatures.

The samples may be arranged in the order of increasing ultraviolet absorption (figs. 12(a), 13(a), 14(a)) as Suprasil, Corning UV grade, and Corning Optical grade. This order is particularly apparent at wavelengths longer than the cutoff; the samples are almost indistinguishable at high absorption coefficients, as suggested.

The absorption peak at 1.4 microns moves toward the infrared with increasing temperature; it passes through a maximum in absorption around 260^{o} C. This movement of the absorption peak is illustrated in figure 16(a). The movement amounts to about 10 millimicrons for Corning UV and Suprasil (not shown) and 14 millimicrons for Corning Optical grade.

The 2.2-micron peak exhibits the same wavelength motion (some 17 millimicrons from 22^{o} to 982^{o} C) but passes through a minimum in absorption between 260^{o} and 538^{o} C as seen in figure 16(b).

The water band has quite a peculiar behavior (fig. 16(c)). From 2746 millimicrons at room temperature, it moves about 5 to 6 millimicrons toward the ultraviolet at 260^{o} C, then moves toward the infrared a total of about 12 millimicrons at 982^{o} C. It shows a monotonic decrease in absorption with increasing temperature.

The wings of all the absorption bands increase in absorption with increasing temperature because of the band broadening. This amounts to roughly a 300 percent increase in the fraction of incident light absorbed in the wings of the 2.2- and 2.75-micron bands. No attempt is made here to propose a model in interpretation of these data, except to note that the red shift of absorption peaks (ref. 4), red shift of ultraviolet cutoff wavelength (ref. 7), and the general increase in absorption between absorption bands (ref. 4) with increasing temperature reflect general trends found in other inorganic solids by other investigations.

CONCLUDING REMARKS

Spectral absorption coefficients of fused silica were measured over the wavelength range from 0.17 to 3.5 microns at five temperatures ranging from 22° to 982° C. A set of 42 samples was used to furnish a "typical" or representative sample set. Initial transmissivity measurements at 22° C showed large variations in absorption coefficient from sample to sample. The samples chosen for measurement at evaluated temperatures were those that exhibited absorption coefficients lying close to the mean absorption coefficient of a number of samples with the same optical grade and manufacturer, at a wavelength of 180 millimicrons.

It has been shown that the transmissivity of fused silica shows a general decrease with increasing temperature. The ultraviolet cutoff wavelength shows a shift toward the infrared of approximately 30 millimicrons between 22° and 982° C. The absorption peak at 1.4 microns moves toward the infrared while going through a maximum in absorption; the peak at 2.2 microns moves toward the infrared while going through a minimum; and the peak at 2.75 microns moves first toward the ultraviolet, then toward the infrared while monotonically decreasing in absorption. The incident light absorbed increases by a factor of 2 to 4 in other regions of the infrared spectrum between 22° and 982° C.

The observed effects are reversible; identical data are reproduced whether the data are taken in steps of increasing or decreasing temperature.

Around 260° to 538° C, various trends in the data show abrupt changes, such as the slope of figures 15 or 16(a). It is difficult to correlate this temperature range with any known temperature-dependent effects other than the α-β phase transformations of the crystalline silica minerals (quartz, cristobalite, and tridymite). The phase transition from α to β quartz occurs rapidly and reversibly at 573° C, and the α-β cristobolite transition occurs rapidly and reversibly at 200° to 275° C. This allows the suggestion that the abrupt and reversible changes in the temperature range of 260° to 538° C may be due to these reversible transitions.

The source of the crystalline quartz and cristobolite in the fused silica is unknown. The rate of formation of cristobolite by devitrification of fused silica is very slow, even at 982° C. Also the formation of crystalline quartz from cristobolite is even more sluggish, if not impossible, by thermal means alone. However, small amounts of fluxes such as calcium oxide or potassium oxide in the fused quartz will considerably increase the devitrification rate to cristobolite and possibly crystalline quartz. Since the specimens are commercial fused quartz, it is possible that they contain these fluxing impurities. It is also possible that the crystalline quartz and cristobolite were originally present in the fused quartz specimens.

The observed data in the infrared are in general agreement with the data of Douglas and Gagosz (ref. 5). The general red shift of absorption peaks, while not explicitly noted

by Douglas and Gagosz, is detectable in the data they present. At the ultraviolet cutoff, the room-temperature absorption which they report as well as that at an elevated temperature is somewhat less than the absorption reported here when Corning 7940 UV grade is compared with their samples called "Corning 7940." For instance, their mean absorption at 180 millimicrons is one-fourth of the mean of samples in the present experiment and one-tenth that indicated by the manufacturer's data (Corning 7940 data from ref. 5 are included in fig. 16(b)). Douglas and Gagosz, however, do not report having attempted to select samples with "average" properties. The internal spread in the ultraviolet absorption edge data reported in reference 5 is of the same order as the difference between those and the data here presented.

Finally, general trends have been noted, such as the thermal effects on ultraviolet cutoff or absorption-peak wavelength, but the absolute value of the absorption coefficient varies with manufacturer, optical grade, and ingot number. If a window or lens for a certain experiment is to undergo temperature changes in the course of the experiment, that particular window or lens should be calibrated in a spectrophotometer.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 27, 1965.

APPENDIX - METHOD OF CALCULATING ABSORPTION COEFFICIENT FOR A SINGLE SAMPLE

The light transmitted I through a sample with parallel and flat faces is a fraction of the incident light I_o given by I/I_o = (fraction lost through absorption)(fraction lost if interference effects are ignored through reflection).

The fraction lost through absorption for monochromatic light is of the form $e^{-\alpha x}$ where α is the absorption coefficient (cm^{-1}) at a particular wavelength, and x is the sample thickness. The effect of multiple reflection in the sample may be evaluated by first noting that the reflection loss r at a glass-air interface is given by

$$r = \left(\frac{n - 1}{n + 1}\right)^2$$

From figure 17 it is seen that $I_oA(1 - r)$ is transmitted through the first face and $I_oA(1 - r)^2$ through the second face (where $A = e^{-\alpha x}$). The total transmitted intensity may be summed:

$$I = I_oA(1 - r)^2\left[A^or^o + A^2r^2 + \ldots + (A^2r^2)^n + \ldots\right]$$

The terms in the brackets are an expansion of the form $(1 - x)^{-1}$; thus

$$I = I_oA(1 - r)^2(1 - A^2r^2)^{-1}$$

or

$$\frac{I}{I_o} = \frac{A(1 - r)^2}{1 - A^2r^2}$$

To evaluate the contribution of the denominator, assume that A = 1 (no absorption) and that n = 2 (fused silica does not exceed 1.6 in the wavelength range concerned). The value of r is then 0.111 and $A^2r^2 = 0.012$. Since n approaches 1.6 only where strong ultraviolet absorption occurs, the denominator is essentially 1, and multiple reflections are ignored. The reflection loss is taken to be just $(1 - r)^2$, and $I/I_o = (1 - r)^2e^{-\alpha x}$, from which

$$-\alpha = \frac{\ln(I/I_o) - \ln(1 - r)^2}{x}$$

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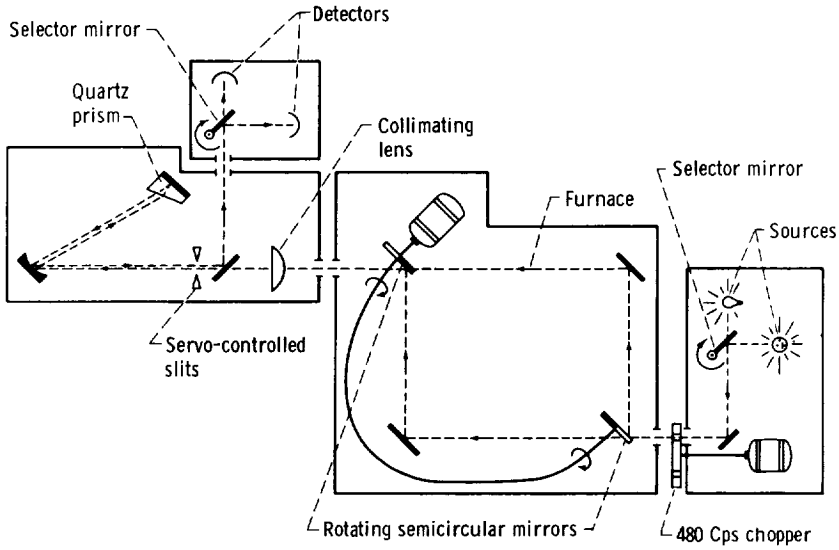

Figure 1. - Optical schematic of spectrophotometer.

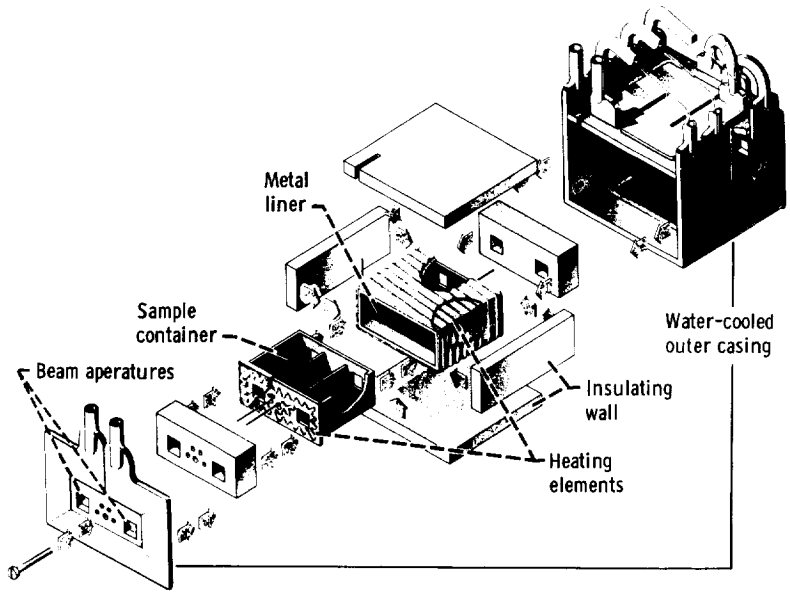

Figure 2. - Exploded view of furnace.

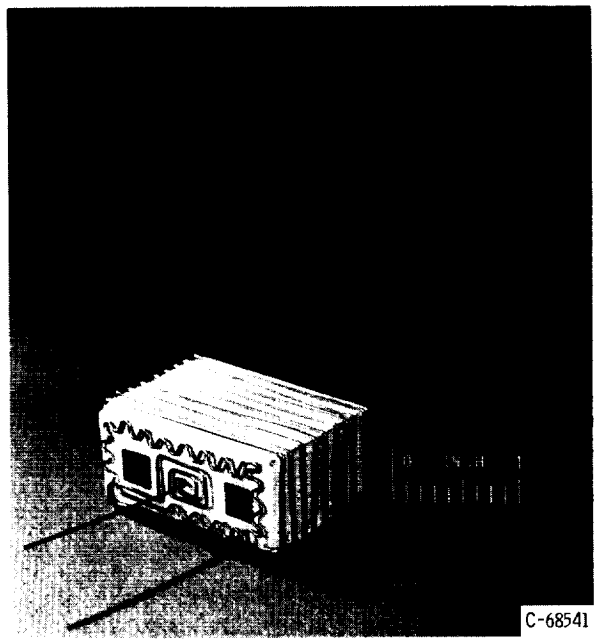

Figure 3. - Heating element wrapped on furnace liner.

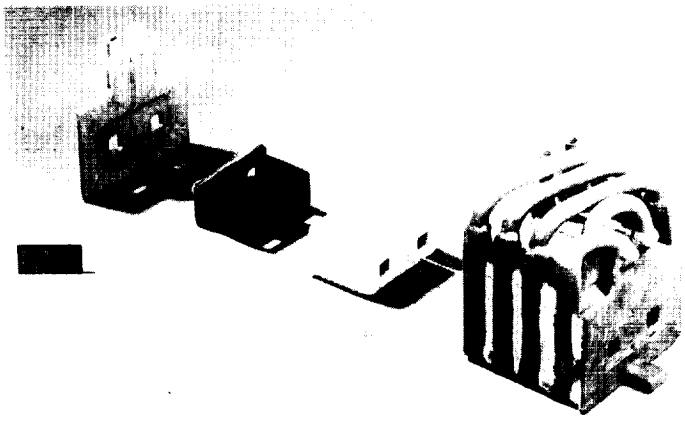

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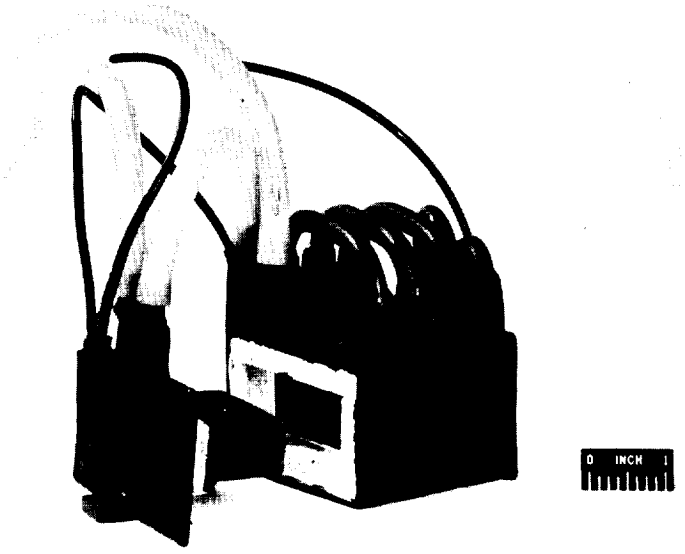
Figure 4. - Completed heated element and cold wall.



C-69105

Figure 5. - Assembled furnace.

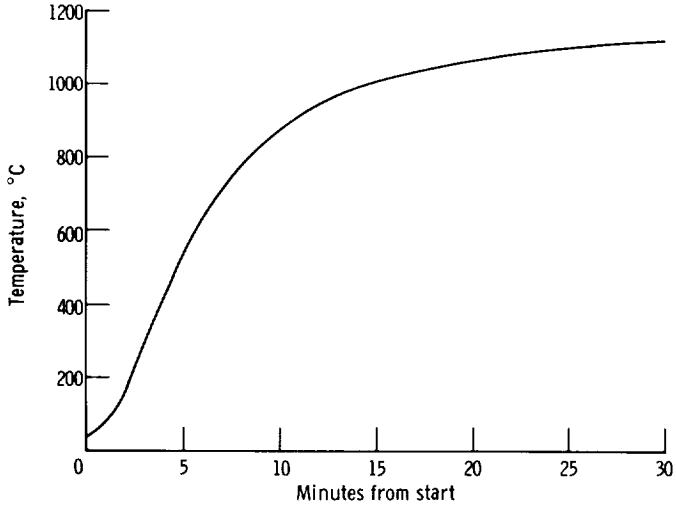


Figure 6. - Full-power startup; input, 8 amperes constant.

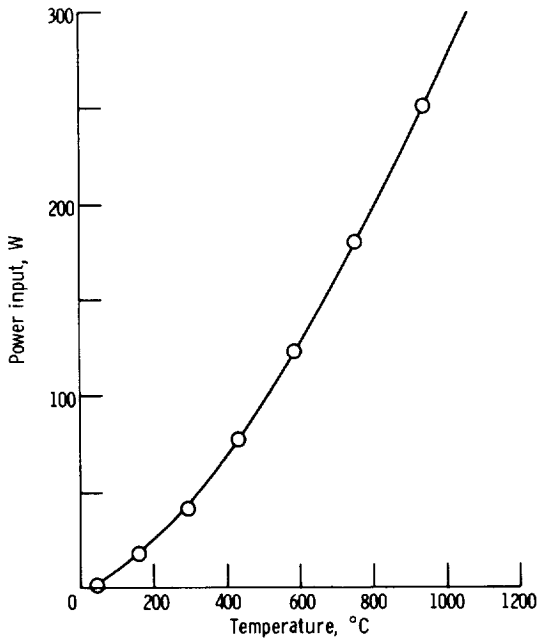
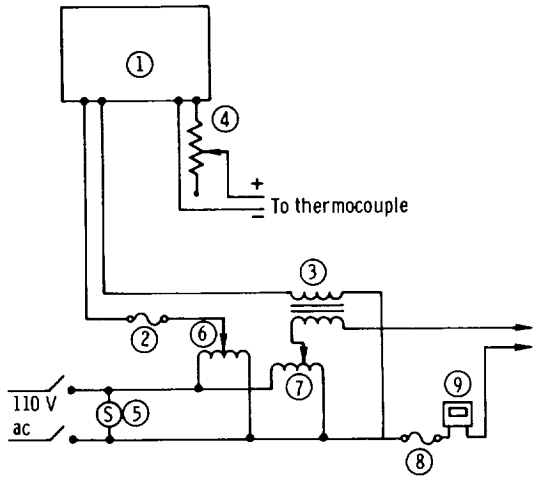


Figure 7. - Power requirement.



① pyrometer indicator-controller
② 3-A fuse
③ filament transformer
④ 100-ohm rheostat
⑤ Neon pilot light
⑥ 3.75-A variable transformer (control power)
⑦ 12.5-A variable transformer (main power)
⑧ 10-A fuse
⑨ 0-10-A ammeter

Figure 8. - Furnace power supply.

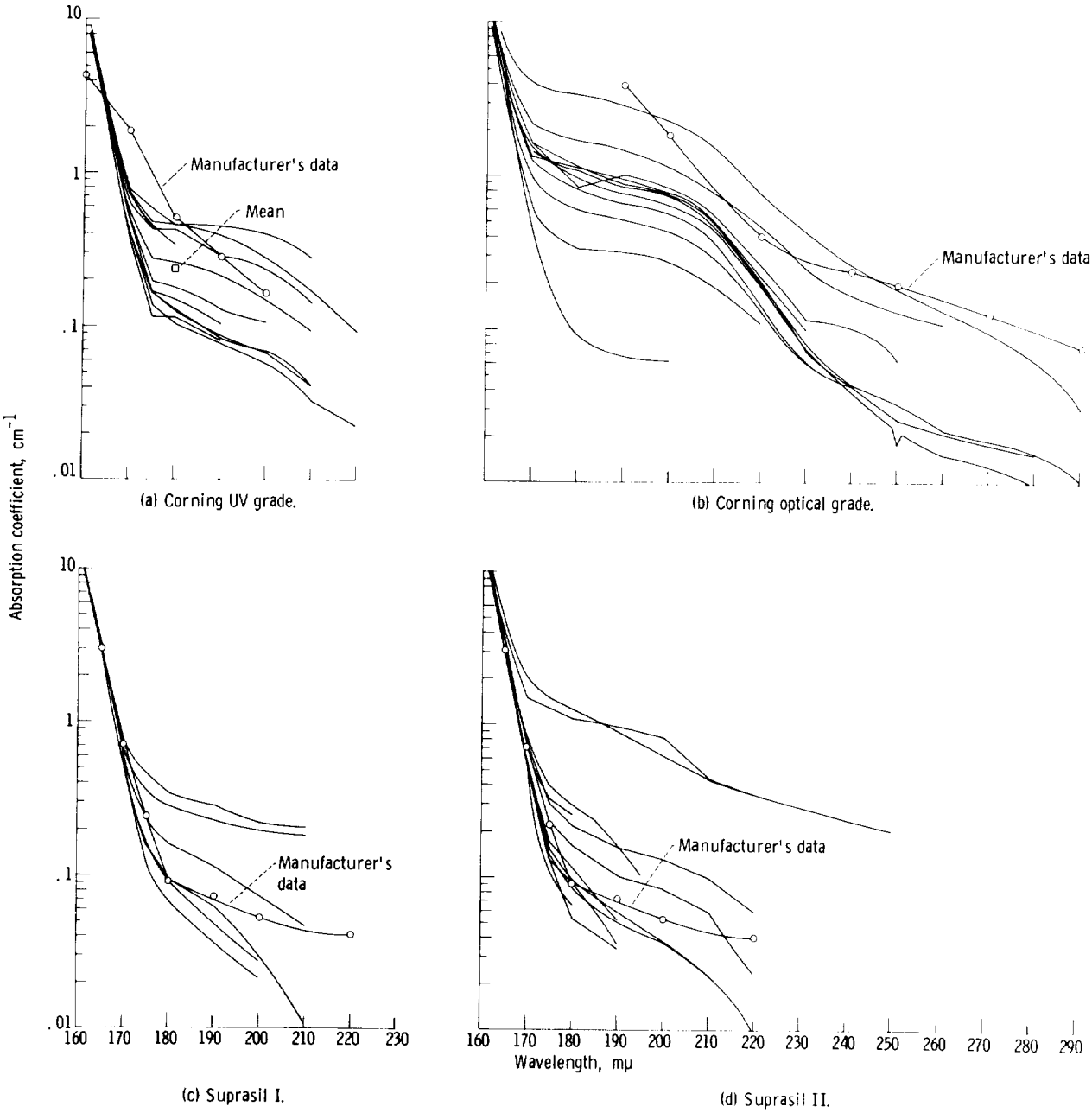


Figure 9. - Corning and Suprasil sample sets, room temperature.

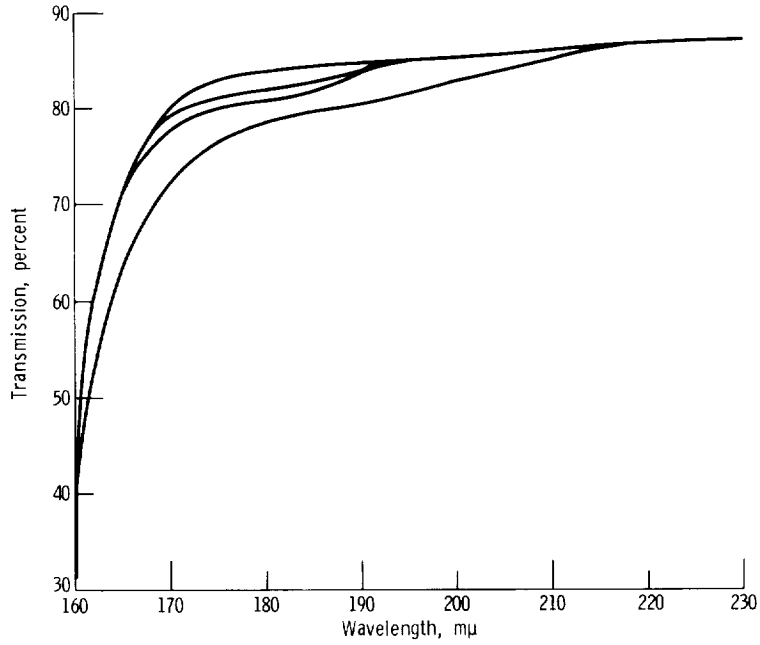


Figure 10. - Sample plots of transmission for "identical" samples.

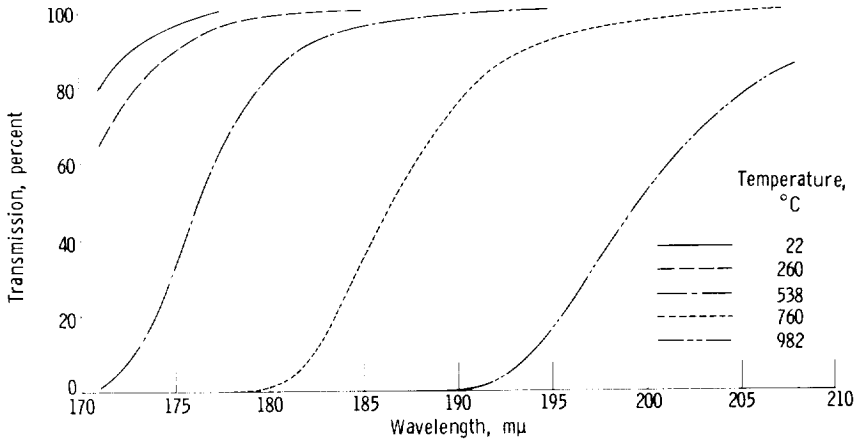
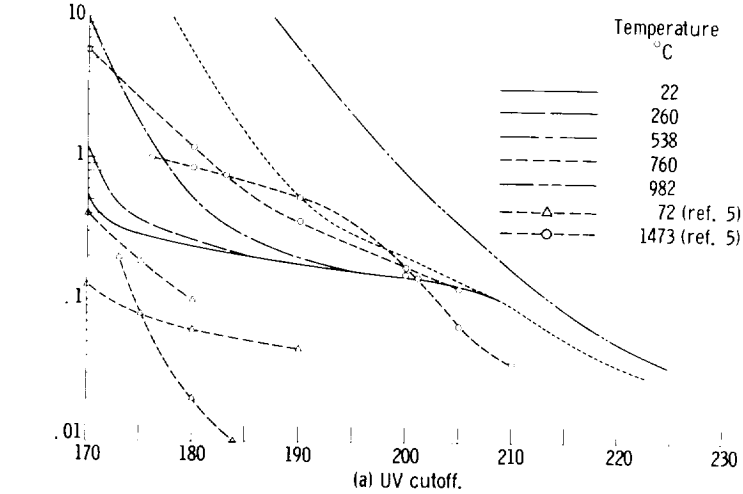
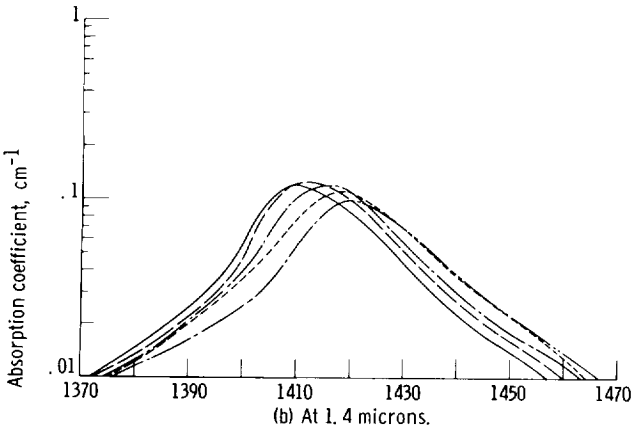


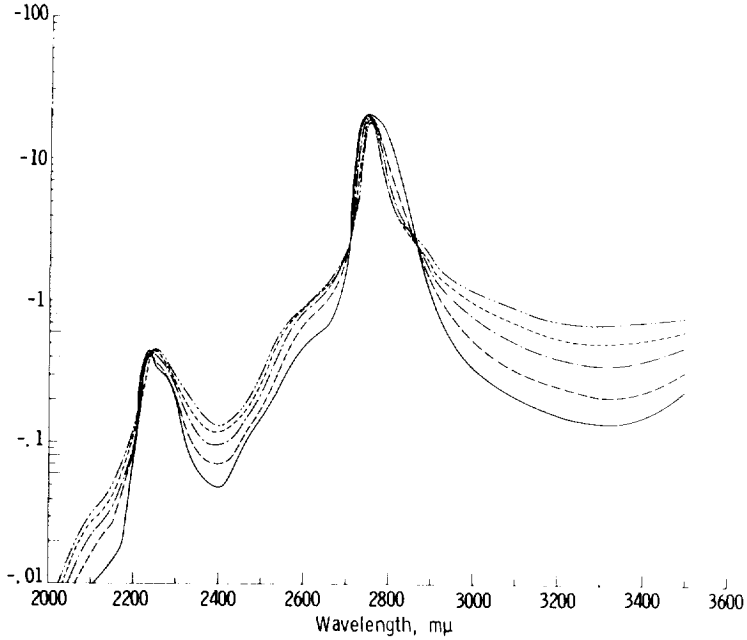
Figure 11. - Transmission of 9 millimeters of Corning UV grade.



(a) UV cutoff.

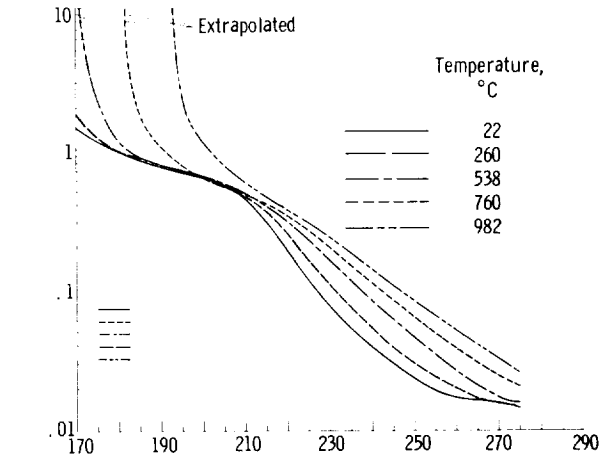


(b) At 1.4 microns.

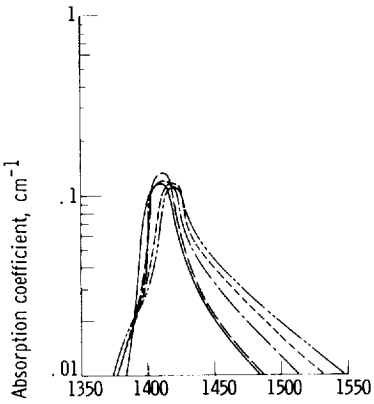


(c) Water band.

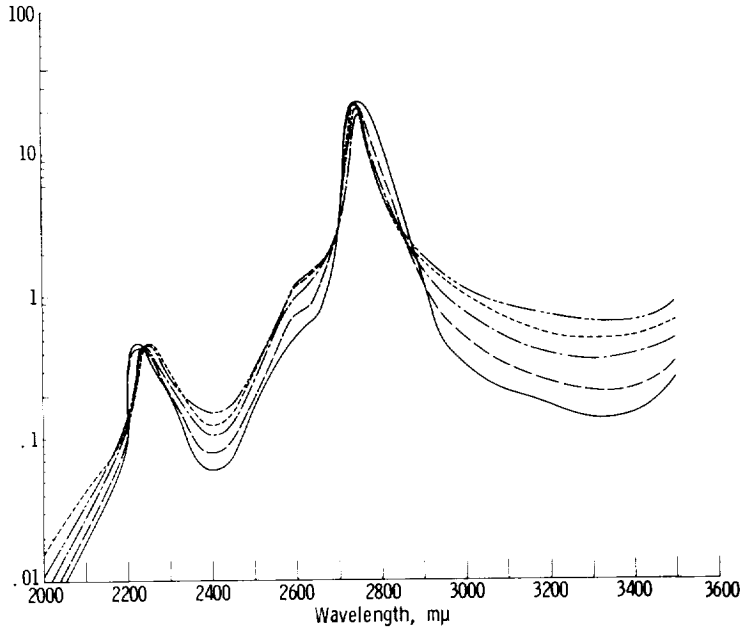
Figure 12. - Absorption spectrum, Corning UV grade.



(a) UV cutoff.

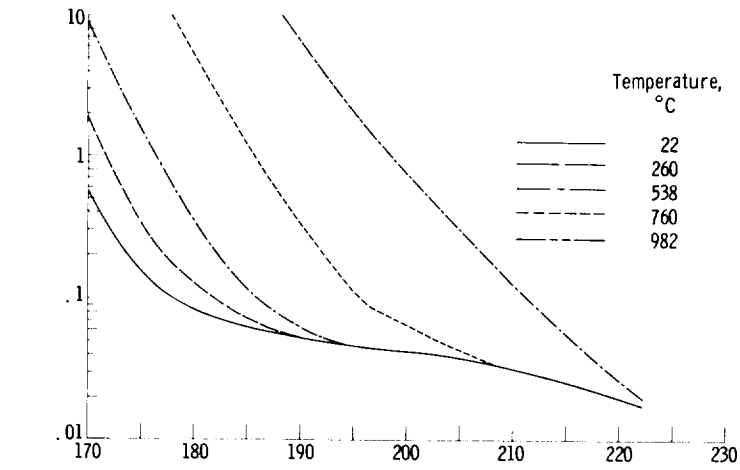


(b) At 1.4 microns.

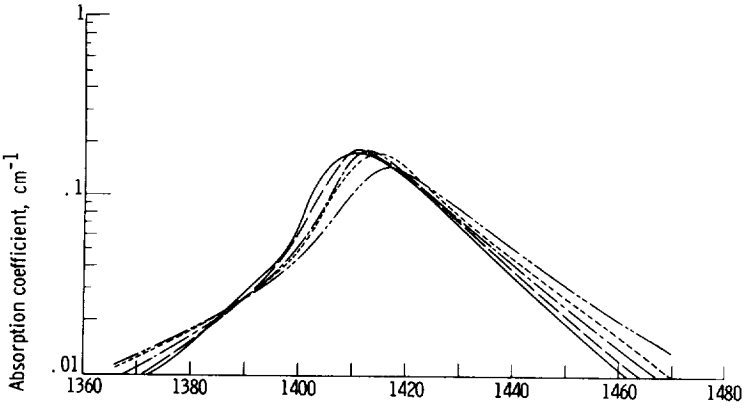


(c) Water band.

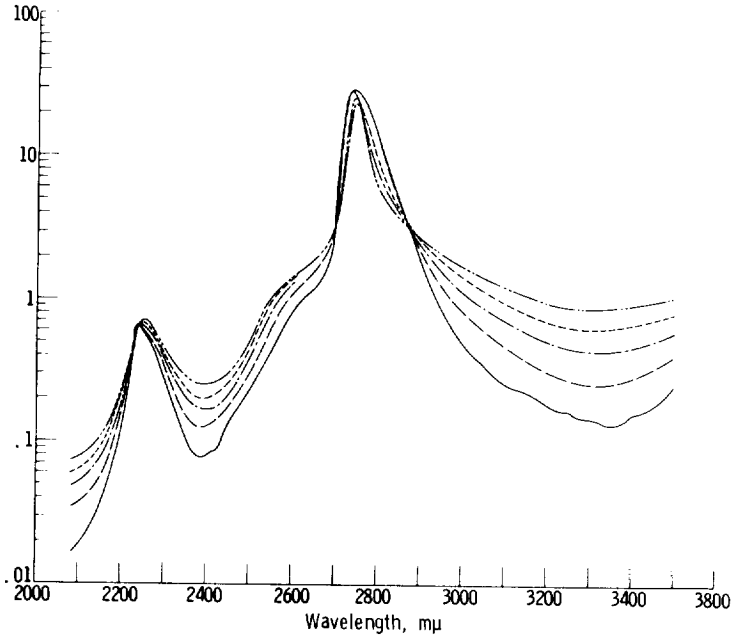
Figure 13. - Absorption spectrum, Corning optical grade.



(a) UV cutoff.



(b) At 1.4 microns.



(c) Water band.

Figure 14. - Absorption spectrum, Suprasil.

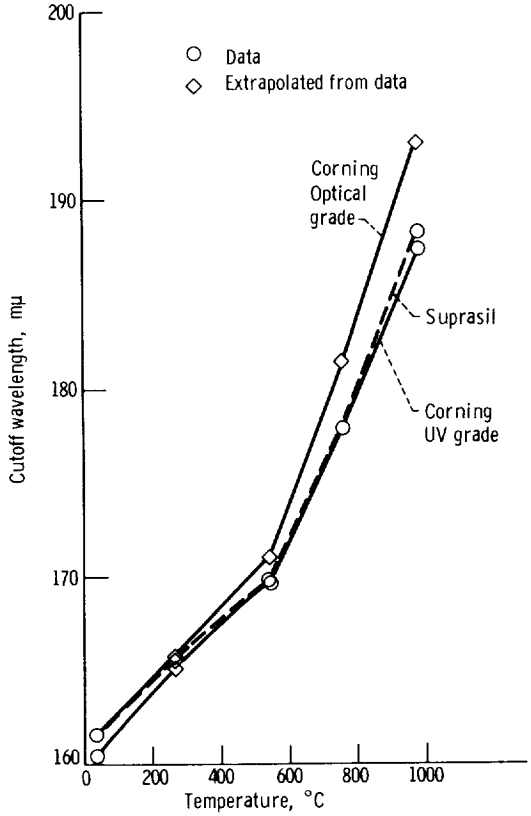


Figure 15. - Position of UV cutoff as a function of temperature: α = 10 centimeter^{-1}.

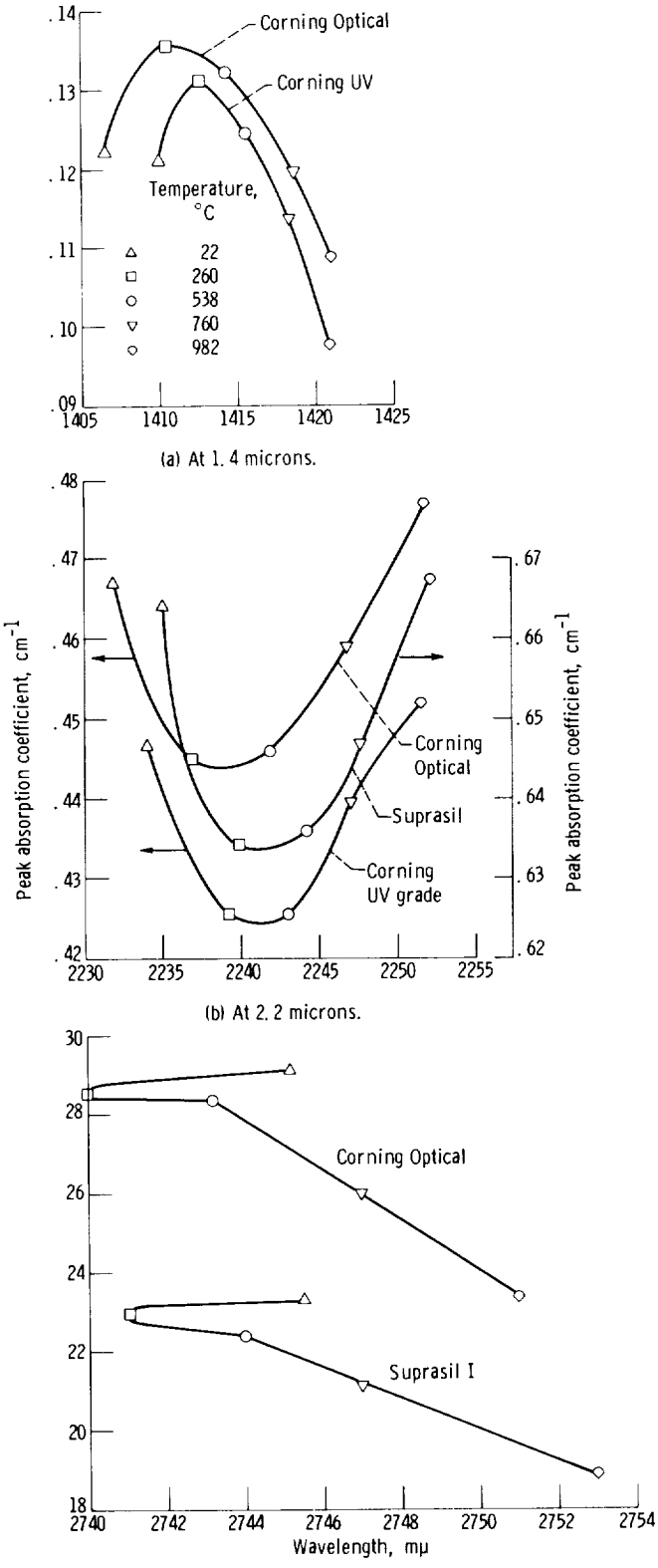


Figure 16. - Motion of absorption peaks.

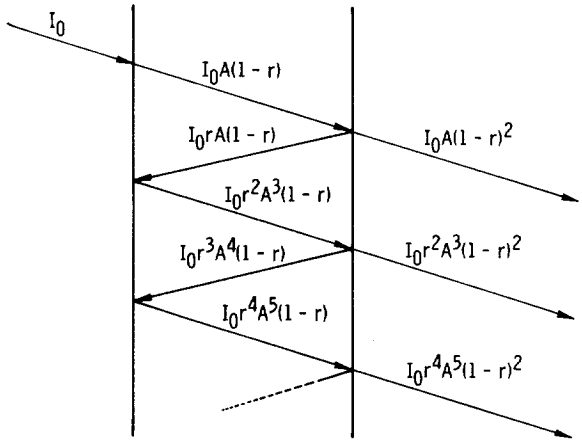


Figure 17. - Optical absorption and reflection within absorbing sample.