## No. 48 TRANSMITTANCES OF SOME OPTICAL MATERIALS FOR USE BETWEEN 1900 AND 3400 Å *

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ABSTRACT

Transmittance curves for samples of materials useful as polarizing prisms, windows, cements, and transmission filters between 1900 and 3400 Å are given. New filters were devised which, when used with ASCOP 541F solar-blind detectors, have effective wavelengths of 2640 and 2830 and bandwidths of 225 and 200 Å, respectively. A cell for liquid filters is described.

### *I. Introduction*

A program of photometric and polarimetric studies of stars and planets was recently described in *Applied Optics*.[1] A special effort was made to find filters having high transmission and narrow bandwidths (about 250 Å), usable on faint objects in the spectral range 1900–3400 Å. Below 3400 Å there are no commercially available glass filters and few interference filters having high transmission with narrow bandwidths. The problems of filters and cements are discussed in Sections II–IV. Interference filters are discussed further in Section V, and inorganic and organic filter solutions in Sections IV and VI, respectively.

In this paper, transmission values are given in terms of percentage transmittance $T_\lambda = 100\ I/I_0$, where $I_0$ and $I$ are the intensities of light incident on and emerging from the sample. Fresnel reflection losses are therefore included in $T_\lambda$. The tracings were done on the Cary 14 recording spectrophotometer. The zero indicated on the Cary 14 is taken here to mean transmission $\leqslant 0.05\%$.

### *II. Glasses and Crystalline Solids*

Figures 1 and 2 give transmissions of the solid materials, usable as polarizing prisms (curves 4, 5, 6), windows, and short-wavelength cutoff filters between 1860 and 4800 Å, listed in Table I. The dashed curves are reproductions of published curves.[2,3] The solid lines represent new tracings. All of the materials transmit at wavelengths longer than 4800Å.

TABLE I

SOLIDS AND GLASSES TRACED IN FIGURES 1 AND 2

| Curve number | Description | Thickness (mm) | Remarks |
|---|---|---|---|
| 1 | Suprasil disk | 1.59 | Ref. 4 |
| 2 | Optosil plate | 1.59 | Ref. 4 |
| 3 | Optosil plate | 6.35 | Ref. 4 |
| 4 | ADP | 23.0 | Light ⊥ to opt. axis |
| 5 | ADP | 43.5 | Light approx. ⊥ to opt. axis |
| 6 | Calcite | 11 | Light approx. 45° to opt. axis |
| 7 | Schott BG-24 | 1.0 | Ref. 3, but approximately corrected for reflection loss assuming constant *n* |
| 8 | Schott UG-5 | 1 | |
| 9 | Schott UG-11 | 1 | |
| 10 | Glass microscope slide | 1.3 | Braun #48299 |
| 11 | Kel-F 81 | 1 | |
| 12 | Plexiglas | 6.35 | |
| 13 | Four Suprasil disks | 4 × 1.59 | Water coupled |
| 14 | Corning C.S. 9-54 | 0.50 | 1/4 stock thick |
| 15 | Corning C.S. 9-54 | 2 | Ref. 2 |
| 16 | Corning C.S. 7-54 | 3.0 | |
| 17 | Corning C.S. 9-53 | 0.50 | 1/4 stock thick |
| 18 | Corning C.S. 9-53 | 2 | Ref. 2 |
| 19 | Schott UG-2 | 2 | |

Curve 6 is of a sample of calcite chosen for clarity instead of crystalline perfection. The low maximum of transmission indicates a poor surface polish. The sharp drop near 2100 Å, however, is typical of calcite. Calcite is birefringent, is unaffected by uv radiation, is insoluble in water, and withstands reasonably well sudden temperature changes. Another birefringent crystal with high transmission to shorter wavelengths is ammonium dihydrogen phosphate (ADP), shown in curves

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4 and 5. The samples are from the Clevite Corporation in Cleveland, Ohio. ADP, however, is water soluble and fractures easily under thermal shock. Polishing of an optical surface must be done with alcohol, for example, and on a soft surface since ADP scratches easily. Irradiation with the 2537 Å line for 1 hr with 500 $\mu W/cm^2$ produced no decrease in the transmission of this material. ADP is reported (by the Clevite Corporation) to be stable to 140°C and has a maximum safe operating temperature of 125°C.

Curves 2 and 3 (Fig. 1) show the commonly used variety of fused silica known as Optosil.* It has the advantages of being an amorphous, easily workable, and thermal-shockproof material. However, Optosil has the disadvantages of an absorption band at 2380 Å due to SiO, decreasing transmission below 2200 Å, and bluish-green fluorescence to uv radiation.[4] All these disadvantages are overcome in Suprasil.* Suprasil is a synthetic fused silica produced under oxidizing conditions. It is produced in two varieties, Suprasil I and II, the first having a greater degree of homogeneity than the second. Curves 1 and 13 show the transmissions of Suprasil I disks. Suprasil I and II are little affected by uv, x rays, alpha, beta, and gamma rays.[4] Further information is given in the Englehard Industries, Inc., booklet.

Kel-F 81,† shown in curve 11, is a resilient plastic which is reported to be chemically inert and not affected in a temperature range from −240 to +200°C.

### *III. Optical Contacting Agents*

The principal requirements of a bonding agent are proper refractive index, stability to uv radiation, and usability over a wide temperature range. The commonly used cements such as Canada balsam (curves 7 and 8 of Fig. 3) are not usable below 3000 Å even in very pure form, therefore new materials had to be found. These are shown in Figs. 3 and 4. Cellulose caprate‡ satisfies these requirements better than Canada balsam and is usable to shorter wavelengths. There are two varieties of this cement: curve 15 in Fig. 4 shows the transmission of the plasticized form[5] and curve 14 that of the unplasticized. Field has done extensive studies on cellulose caprate, and he reports that the plasticized form has a refractive index of 1.493 and has excellent temperature and uv stability. Canada balsam darkens under exposure to a 400-W S-1 mercury-vapor lamp at 20 cm after 200 hr, whereas cellulose caprate, in both forms, remains unaffected.[5]

Dow Corning's Sylgard compounds No. 182 (curve 5) and No. 51 (curve 4) have the particular advantages

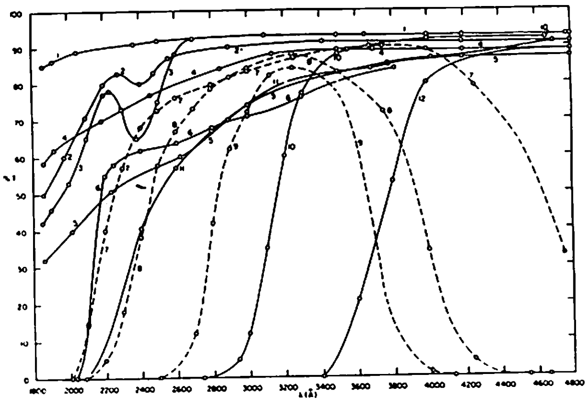

Fig. 1. Transmittances of glasses and crystalline solids. See Fig. 2 and Table I.

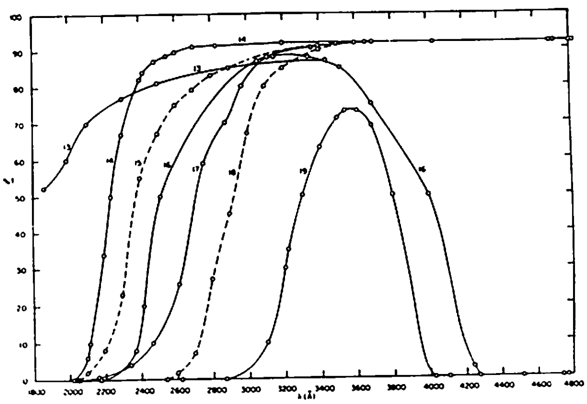

Fig. 2. Transmittances of glasses and crystalline solids. See Fig. 1 and Table I.

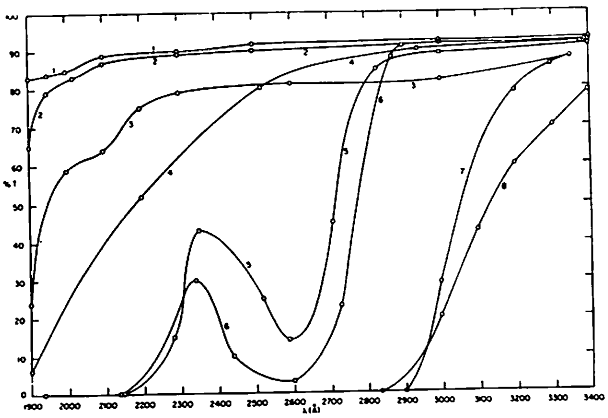

Fig. 3. Transmittances of contacting materials. See Fig. 4 and Table II.

of being unaffected over a large temperature range: −65 to +200°C for No. 182, and −65 to +100°C for No. 51.[6] They also have low chemical reactivity and are stable to uv radiation. These coupling agents do not set to a hard bond but retain the resilience of thick gels.

The most promising contacting agent found was a dimethylpolysiloxane compound sold by Dow Corning

* Manufactured by Englehard Industries, Inc.

† Made by the Minnesota Mining and Manufacturing Company.

‡ Available from Lyon Research Laboratories, Woodbridge, Conn.

TABLE 2
BONDING MATERIALS TRACED IN FIGURES 3 AND 4

| Curve number | Description[a] | Thickness (mm) |
|---|---|---|
| 1 | Water | ~0.025 |
| 2 | Glycerol silicate | ~0.025 |
| 3 | Amphenol silicone compound #53-307 | ~0.025 |
| 4 | Dow Corning Sylgard compound 51 | 0.089 |
| 5 | Sylgard compound 182 | 0.089 |
| 6 | Dow Corning silicone grease C-2-0057 | ~0.025 |
| 7 | Canada balsam—Fisher purified | 0.025 |
| 8 | Canada balsam—Jaegers | 0.025 |
| 9 | Glycerine | 0.025 |
| 10 | Dow Corning 200-Fluid (26 July 1962) | 0.025 |
| 11 | Item 10 (9 Aug. 1962) | 0.025 |
| 12 | Above after 1 hr radiation with 448 $\mu W/cm^2$ of 2537 Å (9 Aug. 1962) | 0.025 |
| 13 | Above after 5 months on shelf (9 Jan. 1963) | 0.025 |
| 14 | Cellulose caprate unplasticized | 0.002 |
| 15 | Cellulose caprate plasticized, ref. 6 | 0.002 |

[a] 1, 2, 3, 9, 10, 11, 12, 13 between 1.6-mm Suprasil disks, remainder between 1.6-mm Optosil disks.

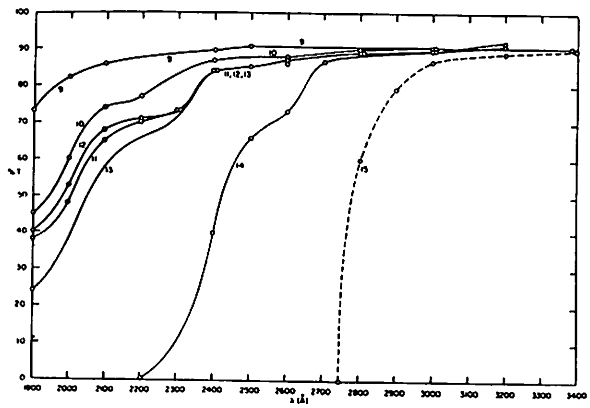

Fig. 4. Transmittances of contacting materials. See Fig. 3 and Table II.

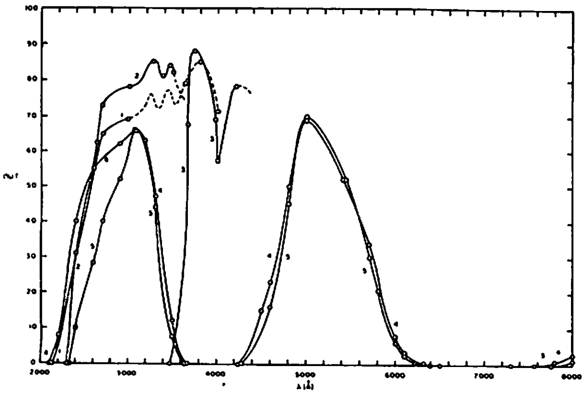

Fig. 5. Transmittances of 1-cm aqueous solutions of inorganic colored compounds. See Fig. 6 and Table III.

as their 200-Fluid (curve 10 of Fig. 4). This fluid is used in the high-viscosity form (1,000,000 centistoke) to reduce flow rate, and the change of viscosity is very small between −40 and +200°C. It is reported to be moisture repellant, chemically inert, insoluble in alcohol (but soluble in hydrocarbon solvents), and fairly stable to uv radiation. It will not react chemically with quartz or ADP, and no fluorescence is expected by Dow Corning.[7] The refractive index[7] at room temperature at 4047 Å is 1.4169. The extent of solarization and aging effects can be seen by comparison of curve 10, made just after assembly (26 July 1962), with curve 11 (9 August 1962), and curve 12 after a 1-hr exposure to an irradiance of 500 $\mu W/cm^2$ with 2537 A radiation. Curve 13 made five months later (9 January 1963) shows the aging.

Glycerol silicate (curve 2, Fig. 3) and glycerine (curve 9) show excellent transparency, but both react with ADP.

### *IV. Inorganic Filter Solutions*

Inorganic compounds have been used in conjunction with commercially available filters[2,3,8] to give high transmission, wide bandwidth filters for the visible region.[9] A brief survey was made of aqueous solutions of the common salts of a few transition metals since these metals are known to be selectively absorbing in the visible (see Table III). Figures 5 and 6 suggest

TABLE 3
INORGANIC AQUEOUS SOLUTIONS[a] TRACED IN FIGURES 5 AND 6

| Curve number | Description | Concentration (g/l) |
|---|---|---|
| 1 | $MnSO_4 \cdot H_2O$ | 500 |
| 2 | $MnCl_2$ | 500 |
| 3 | $Mn(NO_3)_2 \cdot 6H_2O$ | 210 (50% sat.) |
| 4 | $NiSO_4 \cdot 6H_2O$ | 500 |
| 5 | $NiCl_2$ | 500 |
| 6 | $CoSO_4 \cdot 7H_2O$ | 500 |
| 7 | $CoCl_2$ | 500 |
| 8 | $CuSO_4 \cdot 5H_2O$ | 250 (80% sat.) |
| 9 | $CuSO_4 \cdot 5H_2O$ | 320 (sat.) |
| 10 | $Fe_2(SO_4)_3$ | 03.0 |

[a] 1-cm thickness, 1 day old, dissolved and traced at room temperature, analytical-reagent grades.

that the transmission bands are determined primarily by the metallic ion. The manganese salts (curves 1, 2, 3, Fig. 5) have many dips in transmission, of the order of 10%, down to 7500 Å. Chromium salts were found to be similar to cobalt in the red but also have transmission bands centering near 4800–4900 Å and 3300 Å. A tracing of a low concentration of an iron compound (curve 10) demonstrates how important it is to exclude this common contaminant from aqueous filter solutions. Chemicals of the highest purity and nonferrous mixing utensils should be used. It is suspected that the presence of iron is often responsible for aging effects.

Cupric sulfate has been used to eliminate red leaks from filters[10]; it shows no transmission to at least 2 $\mu$. When used with nickelous sulfate and Corning 9863, an excellent filter is obtained having a transmission peak near 3200 Å.[1]

To eliminate the disadvantages of a liquid filter, such as leakage and freezing, a sorbitol solution can be dyed with the absorbing substance to form a semisolid complex. This gives a relatively permanent filter which is amorphous and thus has no polarizing properties. Sorbitol is usable between −70 and +70°C but is subject to aging (decrease in transmission) at short wavelengths due to the presence of absorbing materials such as iron, and therefore must be highly purified. Directions for easy purification of sorbitol, and for the construction of a filter offering 20% transmission at 2650 Å with a 300-Å bandwidth, are given by Dunkleman and Field.[11] Sorbitol must be sterilized to prevent the growth of mold, and it is limited to compounds soluble in water. One centimeter of clear sorbitol purified by the above method showed a transmittance of 85% at 3100 Å, 67% at 2700 Å (absorption due to a remaining impurity), 73% at 2500 Å, and 5% at 2300 Å.

## *V. Interference Filters*

Below 3000 Å, filter possibilities are scarce. Some interference filters are being produced for the region 2000 to 3000 Å by Barr and Stroud, Ltd. (Glasgow, Scotland), Thin Film Products, Inc., and Baird-Atomic, Inc. The transmission peaks are 30% at best, with bandwidths near 250 Å. A bad feature for astronomical purposes is the tailing-off, resulting in transmissions of the order of 1% for about 600 Å on each side of the half-transmission points. Schroeder has produced interference filters down to 1950 Å with 30% transmission and greatly suppressed tails.[12]

The principal problem with the interference filter is that of finding sufficiently transparent substrata. In order to have a reasonably high peak transmission, it is necessary to decrease either the number of alternating layers or the thickness of the metal films. This, in turn, increases the bandwidth and the undesired tail transmission. In principle, the tails, i.e., the transmission minima between interference orders, can never be zero. Other problems are the shift in the peak wavelength transmitted and the production of polarization when these filters are inclined to the optical axis. The effects are related to the angle of inclination.[13] However, the advantages of better stability to temperature and uv radiation make the interference filter preferable to other types of filters especially for use in spacecraft.

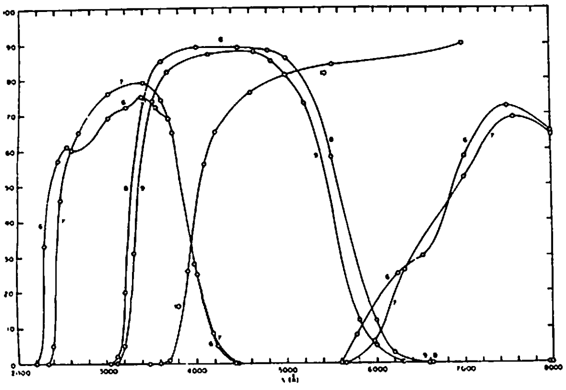

Fig. 6. Transmittances of 1-cm aqueous solutions of inorganic colored compounds. See Fig. 5 and Table III.

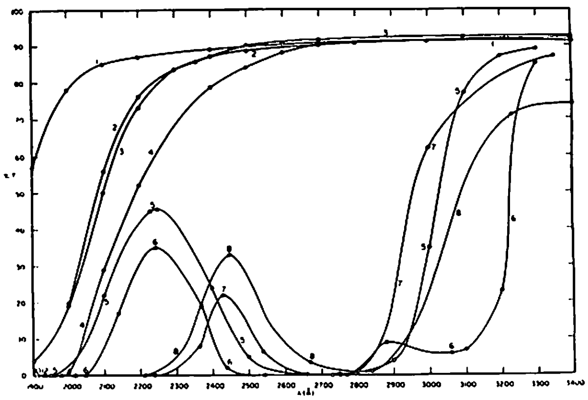

Fig. 7. Transmittances of 1-cm solutions of organic compounds for filters. See Fig. 8 and Table IV.

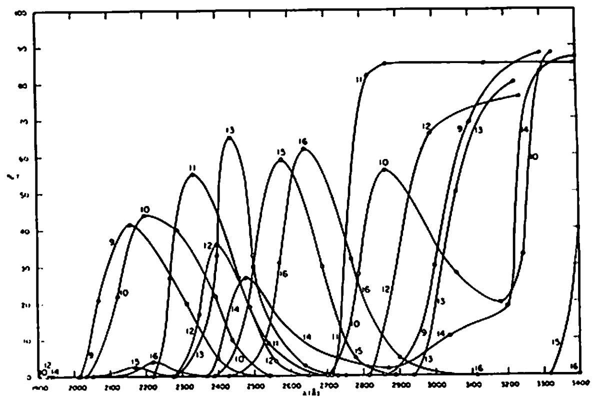

Fig. 8. Transmittances of 1-cm solutions of organic compounds for filters. See Fig. 7 and Table IV.

## *VI. Organic Filter Solutions*

Potential filter materials must satisfy the following criteria:

1. Permanence to uv radiation with time. Changes in absorption characteristics can be caused by photochemical reactions.

2. Long shelf-life. Dissolution of impurities can cause aging. There should be no reaction with the windows or cell materials.

3. Reproducibility. Small fractions of solute are used and therefore must be weighed accurately.

4. Absence of fluorescence in the region of sensitivity of the detector.

5. Absence of optical rotation which is especially important in polarimetry.

6. Temperature stability: the absence of chemical reaction and shift in the absorption bands; no possibility of freezing or boiling in the temperature range of operation.

A search of the literature[14,15] relating to the selective absorption of organic compounds suggested possible filter components for use below 3000 Å. Since the compounds are almost exclusively insoluble in water, highly transparent organic solvents[16] had to be found which would not boil or freeze over a wide temperature range (−90 to +50°C). The transmittances of several organic compounds (1-cm path lengths), of the highest purity available (Eastman Kodak Co.), are presented in Figs. 7 and 8; descriptions are in Table IV. One centimeter of distilled water is shown (curve 1) as a comparison to the spectragrade solvents *n*-hexane (curve 2), *iso*-octane (curve 3), and methanol (curve 4). *Iso*-octane and *n*-hexane give almost identical transmissions with a given solute.

TABLE 4
ORGANIC SOLUTIONS TRACED IN FIGURES 7 AND 8

| Curve number | Description[a] | Concentration[b] |
|---|---|---|
| 1 | Water | Distilled |
| 2 | *n*-hexane | Pure |
| 3 | *Iso*-octane | Pure |
| 4 | Methanol | Pure |
| 5 | 2,4-pentanedione in meth. | 0.2 ml/l |
| 6 | 2-methylpyrazine in meth. | 0.13 ml/l |
| 7 | Durene in iso. | 0.57 ml/l |
| 8 | *p*-ethylphenol in meth. | 1. g/l |
| 9 | 2,4-pentanedione in iso. | 0.2 ml/l |
| 10 | 2-methylpyrazine in iso. | 0.13 ml/l |
| 11 | *p*-cymene in iso. | 0.4 ml/l |
| 12 | *o*-ethylphenol in meth. | 0.45 ml/l |
| 13 | 1-methylnapthalene in iso. | 0.66 ml/l |
| 14 | α-napthol in iso. | 3.67 g/l |
| 15 | *p*-dimethylaminobenzaldehyde (PDAB) in iso. | 0.49 g/l |
| 16 | PDAB in meth. | 0.49 g/l |

[a] iso. = iso-octane, meth. = methanol.
[b] 1-cm thickness.

Only criteria 1 and 6 are of major concern with inorganic (aqueous) solutions. In regards to organic solutions, criteria 1, 3, 4, and 5 concern the solute, while 2 is primarily determined by the solvent. Criterion 6 pertains to either the solute or the solvent or both.

The above tests have not yet been completed for all the materials shown in Figs. 7 and 8. *p*-dimethylaminobenzaldehyde (curve 16) passed all of the tests satisfactorily for the light intensity levels expected. PDAB, incidentally, was first suggested by Dunkleman and Field.[11] Figure 9 shows the response of filters constructed from two organic compounds. Both X and Y filters begin transmitting sharply at 3600 Å (10% at 3700 Å). Figure 10 shows these combined with ASCOP 541F solar-blind photomultipliers with Cs–Te cathodes and short-wavelength cutoff glasses. At 3700 Å the response of the Y combination is about $2 \times 10^{-6}$ A/W. Several variations are possible.

An approach now under investigation is that of "dyeing" transparent solids or highly viscous fluids with the organic compounds. There are good indications that the 200-Fluid, in 1,000,000-centistroke viscosity form, can be "dyed" to provide an amorphous filter. (See *Note added in proof* on page 366.)
The filter cannot leak from its cell since the viscous silicone fluid has a low coefficient of expansion over a large temperature range. A silicone-fluid filter was prepared by first dissolving the organic compound in *iso*-octane or *n*-hexane and then dissolving some silicone fluid in this solution. The hydrocarbon solvent was then removed by evaporation under low pressure, leaving the silicone fluid "dyed" with the organic compound.

The filter cell can now be eliminated by using the silicone-filter fluid between Suprasil disks with a proper spacer to give the desired amount of transmission. From Beer's law, the product of concentration and path thickness is constant for a given absorption with a given compound.

Since these organic filters begin transmitting again at longer wavelengths and are transparent throughout the visible, it is necessary to use solar-blind detectors. A number of solar-blind photomultipliers are commercially available. A method of reducing the sensitivity of photomultipliers at longer wavelengths has been suggested by Childs.[17]

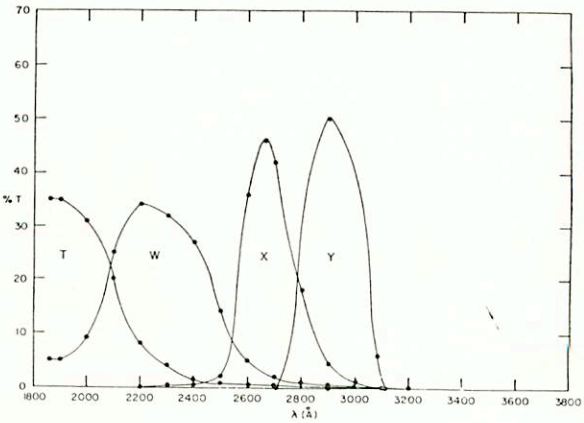


Fig. 9. Transmittances of uv interference filters: T and W, and organic filters: X, 1-cm material 16, Table IV, plus st. th. C9863; and Y, half-concentrations of materials 10 and 16, Table IV, plus st. th. C9863. X and Y begin transmitting again at 3600 Å.

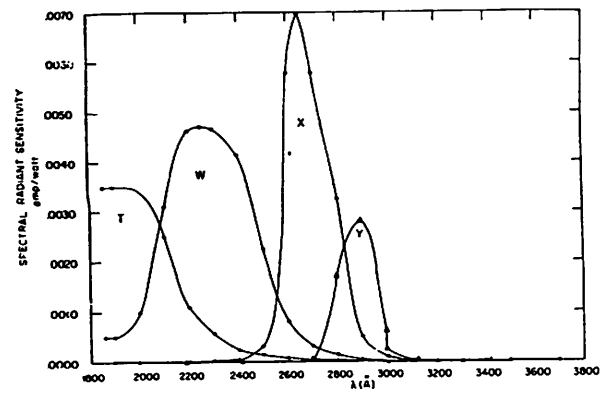


Fig. 10. Response of filters shown in Fig. 9 with ASCOP 541F solar-blind detector.

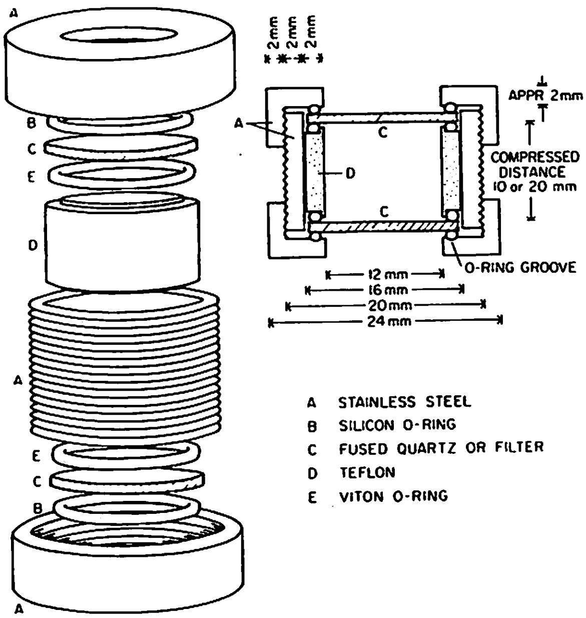


Fig. 11. Leak-proof liquid filter cell.

## *VII. Liquid Filter Cell*

In order to contain liquid filter solutions, a cell has to be leak-proof, rugged, and permanent over long periods of time and wide temperature variations. A design for such a cell is presented in Fig. 11, with suggested dimensions. The use of a Teflon spacer and proper O-ring seals provides a relatively leak-proof, noncontaminating cell. It was found that silicone O-rings give an excellent seal for alcoholic solutions. However, they swell up and cause leaks if used with *isó*-octane or *n*-hexane; Viton O-rings are best for these solvents. Such a cell did not develop bubbles for some three weeks.

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*Note added in proof.* Silicone-fluid filters of good stability to temperature, low pressure, and aging have been made. Their transmissions differ only slightly from the *iso*-octane solution traces shown.

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