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OPTICAL MATERIALS INVESTIGATION FOR
ULTRAVIOLET DOWN-CONVERTERS

J. G. Gwaltieri, et al

Army Electronics Command
Fort Monmouth, New Jersey

October 1972

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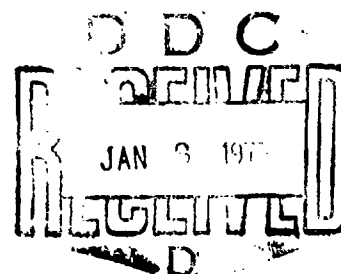
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This report covers the first four months effort on the development of an ultraviolet down-converter to selected visible spectral regions. It consists mainly of knowledge drawn from literature searches, discussions with personnel in-house and in private industry, recent solid state laser spectroscopic research experience, and some preliminary spectroscopic data. Possible solid state down-converter materials, their transmission regions and methods of production for crystalline materials are discussed. The spectroscopic properties of rare earth ions which are especially suited for radiation conversion via visible fluorescence are reviewed. Finally, the performance properties of some of these materials are summarized and recommendations for further research are given.			

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OPTICAL MATERIALS INVESTIGATION
FOR ULTRAVIOLET DOWN-CONVERTERS

by

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October 1972

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OPTICAL MATERIALS INVESTIGATION FOR ULTRAVIOLET DOWN-CONVERTERS

INTRODUCTION

High intensity AC arc discharge lamps, consisting of mercury vapor under fairly high pressure (1-5 atm) are being used as illumination sources for military application. In order to achieve desired color and efficiency, metal additives are combined with mercury to produce richer arc emission spectra in a desired color region. In some cases, it is desirable to further enhance the color of these lamps. One way to accomplish this is to down-convert what would ordinarily be undesirable emission from the lamp, such as ultraviolet radiation, to the visible region. The conversion would occur in a solid fluorescent material which would act as an external active window in the spectral region of interest.

The general desirable properties of such an optical element are:

1. That it require no power to operate, other than the absorption of the undesirable UV radiant power.
2. That its specular transmittance be at least 90%. If the original beam is highly scattered within the window, the source will become a Lambertian radiator. This is undesirable since it will act as a far field attenuator for large beam angles, as is evident in Figure 1.
3. The cost of the active window must not be prohibitive.

Three wavelength regions, blue (near $4500\text{\AA}$), green (near $5400\text{\AA}$), and orange-red ($6100\text{\AA}$), are effective in changing the perceived chromaticity of an object or a light source. In other words, the eye uses these wavelengths most effectively in sampling all incoming light. Color discrimination is improved by elimination of wavelengths near $5000\text{\AA}$ and $5800\text{\AA}$, and color discrimination per watt input at the eye is maximized. For this reason, the three color regions mentioned above are of interest here.

The spectral distribution of a pure Hg arc (see Figure 2) in the visible is a high density of lines combined with a broad continuum, peaking in the neighborhood of $4500\text{-}5000\text{\AA}$. Also, there are many intense lines in the UV, the principal ones are located at 3650 and $3663\text{\AA}$, and these are lines to be used in the downconversion process. These arc lamps operate at quite high temperatures ($650\text{-}700$ degC) and are usually enveloped by glass or quartz whose temperature approaches $300\text{-}400$ degC. These heat sources must be considered in designing any fluorescent window for use with these lamps.

Another reason for searching for efficient ultraviolet down-converters to the visible is to improve the efficiency of laser pumping. Ultraviolet emission from typical flashlamps used as excitation sources for laser materials is wasted, and may be detrimental. For example, there may be inordinate heating due to host absorption and subsequent phonon generation. Also, photochemical dissociation and excited state absorption may occur, and this could degrade laser performance. If a filter material could be found which down-converts the UV radiation to more useful spectral regions, an increase in pumping efficiency would be achieved.

CONVERTER MATERIALS

Three classes of materials are under consideration. Namely, crystals, plastics, and glasses. Liquids² have also been considered for use as light converters, but will not be considered here. Generally, these materials should not absorb in the spectral region 0.25 - 6 μm . The reasons for this are:

1. The UV must penetrate in order to optically pump the fluorescence with high efficiency.
2. Visible radiation must not be attenuated since then the light output from the arc lamp would be decreased.
3. The infrared should not be absorbed since then the window would be heated and the fluorescence could be quenched.

Crystals

Considering all three classes, crystals seem to satisfy the above criteria best. Examples are LiF (transmission range 0.12 - 9.0 μm) and CaF_2 (transmission range 0.13 - 12.0 μm). There are many techniques for producing crystalline materials. The methods considered here are: (1) pressure sintering, (2) Bridgman method, (3) controlled profile, (4) substrate sputtering, (5) solution growth, and (6) flame fusion.

Eastman Kodak has pioneered work on hot-pressed infrared materials. Their IRTRAN-1 through 6 are produced by this method. IRTRAN-3 or polycrystalline CaF_2 can be pressed up to 5 inches in diameter. Optically, hot pressed polycrystalline CaF_2 is equal to the single crystal material except for a slight haze or scatter. This is due almost wholly to submicron voids between grains in polycrystalline materials. However, at 0.5 μm the specular transmittance is still 91%. Kodak has a patent position on hot-pressing and their price for hot-pressed CaF_2 is very high. Their patents will be examined to determine a less expensive alternative method. Y_2O_3 may also be prepared by sintering as reported by General Electric. They prepared a mixture of Y_2O_3 (90%) and ThO_2 (10%) or ZrO_2 (10%) which they called YTTROLOX. This material is³ transparent from 0.25 to 9.5 μm with no absorption bands. ECOM has facilities for experimental studies in hot pressing and sintering of these compounds.

Isomet has supplied a large flat single crystal of CaF_2 : Eu^{2+} (0.2%) grown by the Bridgman method. This crystal was polished but unfortunately, the sample cleaved into four pieces during the polishing procedure. We can experiment with the smaller pieces since they are still quite large (1x1 inch). The Bridgman method is not as expensive as most other production methods, which would make this method attractive for CaF_2 if the single crystal material did not have such an easy cleavage. By contrast, the hot-pressed polycrystalline CaF_2 is much stronger (modulus of rupture is 5300 psi at 25°C).

Tyco laboratories has had success in growing Al_2O_3 in many shapes and sizes by the controlled profile method. There is no reason why this method could not be used to grow other materials such as CaF_2 .

Other methods such as RF sputtering, flame fusion, and solution growth, are speculative and will depend on the actual material and shape required. However, using any of these methods, one can, in principle, produce crystals of any desired shape and size.

Plastics

Among the common plastics, polyethylene and polystyrene are representative of many different plastics. Except for narrow bands, where only a small amount of energy is absorbed, and for broader bands in some materials, the transmission in the IR is relatively good. For thicker samples, the regions of small absorption deepen rapidly and widen considerably, and the absorption becomes so great that the material may no longer be satisfactory for the intended use. However, the thickness required in one plastic sample to absorb and convert UV to blue radiation was about 2 mm. Whether this is already too thick has not yet been determined. Plastics have an advantage in that they are tough and may be formed into any size and shape easily and inexpensively. Their disadvantage is that they cannot be heated much over 200°C.

The most common base materials for fluorescent plastics are polyethylene, polystyrene, and polypropylene. A consideration of their properties and those of other potential fluorescent plastics follows in Table I.

Table I. Properties of some common plastics.

Material	Yield Stress	Max. Continuous Service Temp.	Clarity	Color
Polyethylene (high density)	5000 psi	200 degC	translucent	colorless
polystyrene	9500 psi	82 degC	transparent	colorless
polypropylene	4900 psi	N.A.	slightly hazy	slightly yellow
polytetrafluorethylene	2000 psi	260 degC	translucent	colorless
polytrifluorochlorethylene	4200 psi	200 degC	transparent	colorless
polyvinyl chloride (rigid)	N.A.	74 degC	transparent	colorless
polyvinyl chloride (non rigid)	N.A.	105 degC	transparent	colorless

The first two compounds may be made fluorescent in any part of the visible spectrum. Their fluorescence is relatively bright and their spectral distribution is broadband.

Glasses

Most optical glasses transmit in the infrared only to about $2.7 \mu\text{m}$, then a fairly strong absorption sets in; beyond about $4 \mu\text{m}$ glasses have no appreciable transmission. On the other end of the spectrum, glasses cut off in the $0.3\text{--}0.4 \mu\text{m}$ region. However, glass is usually inexpensive and may be cast into any size or shape.

FLUORESCENT IONS IN CRYSTALS AND GLASSES

$\text{Eu}^{2+}:\text{CaF}_2$ (Blue region)

Europium is known to appear in compounds often as a divalent ion. This is because rare earth ions are more stable if they can attain either the unfilled f^7 , half filled f^7 , or completely filled f^{14} electronic configuration. Eu^{3+} is an f^6 electronic configuration, while Eu^{2+} is an f^7 . In some crystals, the divalent state of Europium is easier to incorporate than the trivalent state. The $\text{Eu}^{2+}:\text{CaF}_2$ spectrum consists of broad bands with some sharp lines below $4000 \text{ \AA}$ in absorption (see Figure 3), and strong continuous fluorescence in the blue violet near $4060 \text{ \AA}$. A fluorescence line at $24,206 \text{ cm}^{-1}$ of Eu^{2+} in CaF_2 was assigned to a transition from an upper state belonging to the f^6d configuration. Thus, it is a d-f Laporte allowed electronic transition. For this reason, $\text{Eu}^{2+}:\text{CaF}_2$ is a bright fluorescent crystalline material for the blue region of the spectrum (see Figure 4). In addition, it appears that all UV absorption and down-conversion to the blue takes place within 1-2 mm of the surface in 0.1 - 0.2 atomic % Eu concentration in CaF_2 . The temperature dependence of the $4325 \text{ \AA}$ emission of 0.2% $\text{Eu}^{2+}:\text{CaF}_2$ was studied from room temperature to 194 degC . The results are plotted in Figure 5. The intensity is still half its original value at 90 degC , but 1/10 at 160 degC , and 1/100 at 200 degC . It seems from this performance that a window of this material must not be heated to more than 100 degC . There is little brightness diminution up to this temperature.

$\text{Eu}^{2+}:\text{CaF}_2$ has already been considered for use as an active pumping filter for lasers.³ The device would consist of a jacket or cladding and be placed between the flashlamp pumping source and the laser material.

Eu^{3+} in Y_2O_3 and Y(P,V)O_4 (orange-red region)

Eu^{3+} introduces its own characteristic emission and absorption lines into the spectrum of Y_2O_3 . These lines represent transitions between 4f electron levels. In addition to sharp lines, strong, broad absorption in the UV and occasional broad emission bands are observed. The origin of the broad bands has not been well established. They may represent 4f to 5d transitions or charge transfer bands. For our purposes, the important point is that the total absorption in the broad bands is much greater than that in the sharp lines and the efficiency of excitation of the fluorescence is correspondingly greater. The broad excitation band is also well placed (centered at $2600 \text{ \AA}$) relative to the emission of a mercury discharge lamp. The red emission is centered at

6113Å. The principal emission is a $^5D_0 \rightarrow ^7F_2$ transition. It has been reported that the fluorescent efficiency of $\text{Eu}^{3+}:\text{Y}_2\text{O}_3$ remains high even at elevated temperature⁴. The fluorescent output under high pressure mercury excitation actually increases with temperature up to 650 degC as shown in Figure 6. However, in our preliminary studies, we have found that the 6113Å fluorescence intensity diminishes with increasing temperature under either of the principal Hg lamp excitations located at 3650 or 2537Å.

Another phosphor $\text{Eu}^{3+}:\text{Y}(\text{P,V})\text{O}_4$ has strong emission lines at 6150 and 6190Å which actually become more intense with increasing temperature, peaking between 400-500°C. This may be due to shifting of the excitation bands for these lines toward the intense 3650Å mercury output.

$(\text{UO}_2)^{2+}$: glass and $(\text{UO}_2)^2$: LiF (green region) and Cu_2O : glass (blue region).

Most Uranyl compounds exhibit green fluorescence, some fluorescing brightly. Most data indicate that the UO_2 ion is linear, having well known modes of vibration. In fact, the fluorescence of $(\text{UO}_2)^{2+}$ is expected to be vibration dependent, indicating that it may be affected by increasing temperature.⁵ This has been investigated in the case of $(\text{UO}_2)^{2+}$ in glass.⁶ The fluorescence falls off rather markedly with increasing temperature as displayed in Figure 7. By contrast, copper activated blue fluorescent glass has an increasing fluorescence with increasing temperature up to about 86 degC and then falls off very slowly with increasing temperature. It seems that if $(\text{UO}_2)^{2+}$ is to be considered at all, it will have to be embedded in a host which is transparent to infrared radiation, and has low frequency lattice vibrations. Both of these criteria would minimize competing non-radiative relaxation processes. A good candidate is $(\text{UO}_2)^{2+}$: LiF, which is pumped by ordinary room light as is the blue fluorescence of $\text{Eu}^{2+}:\text{CaF}_2$. This system retains almost 80% of its original intensity up to 300 degC. The curve of normalized intensity versus temperature for 3650Å excitation, shown in Figure 8, was obtained by measuring the area under the fluorescence spectra at each temperature and dividing by the room temperature area.

CONVERTER PROPERTIES

The minimum thickness required for a 3 x 5 inch window of hot pressed CaF_2 is calculated to be 5.16 mm. This figure was arrived at by using conventional strength of materials formulas found in engineering handbooks. A safety factor of 4 was also included in the calculation. The modulus of rupture is the only parameter needed to make the calculation. When it is available for a particular material, it will be used to obtain the calculated minimum thickness. When it is not available, one could resort to actual vibration testing using the facilities available for vibration analysis in ECOM.

It appears that most, if not all, UV radiation will be absorbed by the fluorescent ions in 1-2 mm of window thickness. However, the transmittance of all window materials will be studied in the ultraviolet region.

Preliminary spectroscopic measurements regarding the increase in radiant emission in the blue region to the blue mercury line at 4358Å using $\text{Eu}^{2+}:\text{CaF}_2$ been made. In the experiment, a low pressure mercury source was used.

The output of this lamp consisted of only sharp lines (see Figure 9), no continuum could be observed in the blue region. The window fluorescence increased the blue line output by about 64% (see Figure 10). We expect that when a high pressure mercury source is used which has a blue continuum the increase in the blue region will diminish. This will be investigated when we are set up to handle the higher pressure AC mercury arc lamps.

A corresponding increase in the green region using $(\text{UO}_2)^{2+}$: LiF is shown in Figure 11.

To evaluate materials as efficient down-converters, the following analysis may be used:⁷ A lamp (ℓ) emits $dP = D_\ell(\lambda) d\lambda$ watts of power in wave-length interval $d\lambda$ centered at λ , where $D_\ell(\lambda)$ is the wavelength distribution of the radiant power. A fraction $\Omega_{\ell c}$ of this power which is emitted into 4π steradians impinge on the converter (c). Another fraction $A_c(\lambda)$ are absorbed. $A_c(\lambda)$ is the absorbance of the converter and is dependent upon its thickness and concentration of active ion. If $T_c(\lambda)$ = the transmittance of the converter and $R_c(\lambda)$ = its reflectance, then $T_c(\lambda) + A_c(\lambda) + R_c(\lambda) = 1$.

The radiant power absorbed by the converter is

$$P_{ca} = \Omega_{\ell c} \int D_\ell(\lambda) A_c(\lambda) d\lambda.$$

A fraction η_c of this absorbed light flux will be down-converted by photon fluorescence and radiated into 4π steradians with wavelength distribution $D_c(\lambda)$. The total converted radiant power will be

$$P_c = \int D_c(\lambda) d\lambda = \eta_c P_{ca}$$

Each sample may be evaluated for η_c , the conversion quantum efficiency, and this will be related to the sample thickness, fluorescent ion concentration, and wavelength distribution of the lamp.

The ratio of the light output with the down-converter in place, to that when it is removed may be calculated as follows: The power incident at a receiver (r) will be a sum of contributions from the lamp and converter. The power absorbed by the receiver will be

$$P_{ra} = \Omega_{\ell r} \int D_\ell(\lambda) T_c(\lambda) A_r(\lambda) d\lambda + \Omega_{cr} \int D_c(\lambda) A_r(\lambda) d\lambda$$

When the down-converter is removed, the absorbed power will be

$$P_{\ell a} = \Omega_{\ell r} \int D_\ell(\lambda) A_r(\lambda) d\lambda$$

The enhancement ratio will be defined as $P_{ra}/P_{\ell a}$, and whenever $T_c(\lambda) = 1$, an overall gain due to downconversion may be defined as $(P_{ra}/P_{\ell a} - 1)$. To relate this to experiment, we note that if a source of P_λ radiant power of P_λ watts of wavelength λ in interval $d\lambda$ is incident on a photocathode, then the photocurrent will be

$$I = (e/hc) P_\lambda \cdot Q(\lambda) \cdot \lambda \text{ amps.}$$

With e = electronic charge and $Q(\lambda)$ = wavelength distribution of the photomultiplier quantum efficiency. Then the ratio of two current signals at the same wavelength, one with the down-converter in place and one when it is removed will be P_λ/P_λ' and will equal $P_{ra}(\lambda)/P_{ra}'(\lambda)$ when calculated at wavelength λ .

In the analysis, scattering and absorption due to overlap of the absorption band of the converter with its fluorescence band have been neglected.

SUMMARY

The most promising base material seems to be crystals. However, if a fluorescent phosphor could be found to have a reasonable quantum efficiency at high temperature, it would be of interest, since then a phosphor coated arc envelope could be employed. In this regard, the phosphor $\text{Eu}^{3+}:\text{Y}_2\text{O}_3$ is known to have a quantum efficiency of 0.5 at 475 degC for down-converting 2400Å radiation to principally 6113Å radiation.

An examination of the three spectral regions of interest for the most promising fluorescent materials reveals that: (1) In the blue region, $\text{Eu}^{2+}:\text{CaF}_2$ is the brightest emitter. However, this material will not down-convert efficiently above 100 degC. For this reason Cu_2O glass should be investigated because of its reported high temperature fluorescence. (2) In the green region, the brightest emitter is $(\text{UO}_2)^{2+}:\text{LiF}$. Its fluorescent output decreases slowly up to 300 degC, losing only 20% of its original intensity. In the orange-red region, Eu^{3+} is unquestionably the brightest emitter. However, the preparation of crystalline materials such as Y_2O_3 and $\text{Y}(\text{P,V})\text{O}_4$ remain a definite problem. Also, $\text{Eu}^{3+}:\text{Y}_2\text{O}_3$ only down-converts radiation in the 2300-2800Å region with high quantum efficiency. The fluorescent output of $\text{Eu}^{3+}:\text{Y}(\text{P,V})\text{O}_4$ increases in the orange-red region, under 3650Å excitation peaking around 400 degC. At 500 degC, the decrease is only 6%, but at higher temperatures the decrease is more pronounced.

RECOMMENDATIONS

Spectroscopic samples of crystals, glasses, and plastics from in-house and private industrial sources should be acquired, which satisfy transparency, temperature stability, and high fluorescent brightness criteria. Absorbance and fluorescence wavelength distributions should be determined. Fluorescence versus temperature measurements will also be required to evaluate proposed down-converter materials. The enhancement ratio of the radiant emission in the spectral regions of interest should be determined for all promising down-converters.

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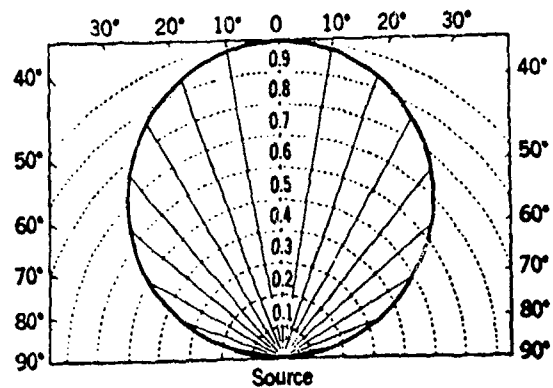


Figure 1. Polar diagram of radiant intensity when Lambert's law is obeyed.

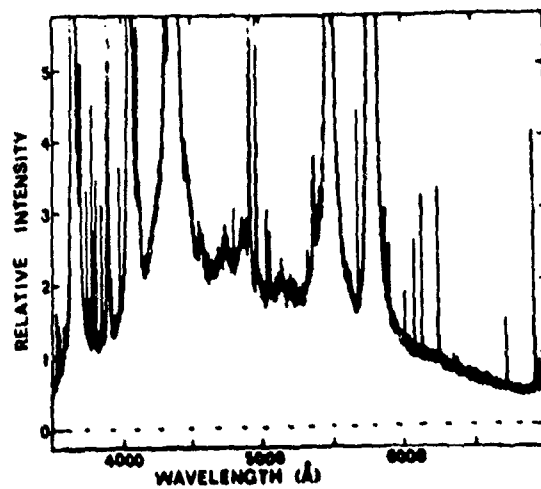


Figure 2. Continuum spectrum of a pure mercury arc.

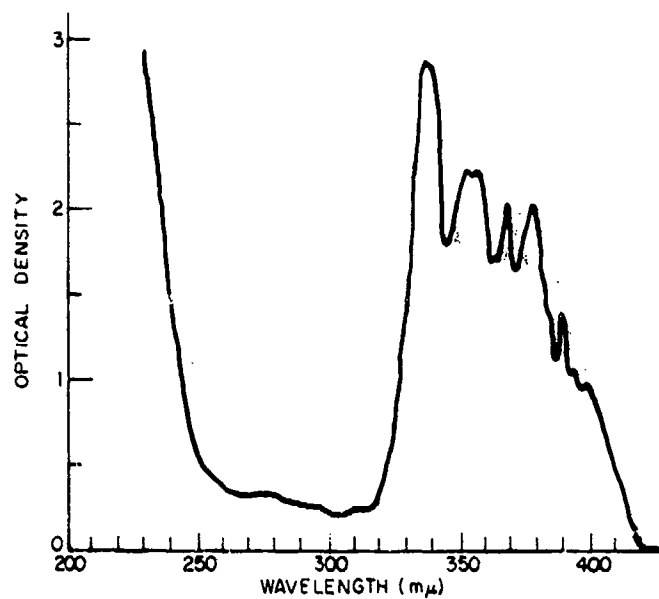


Figure 3. Absorption spectrum of 0.1% Eu^{2+} : CaF_2 .

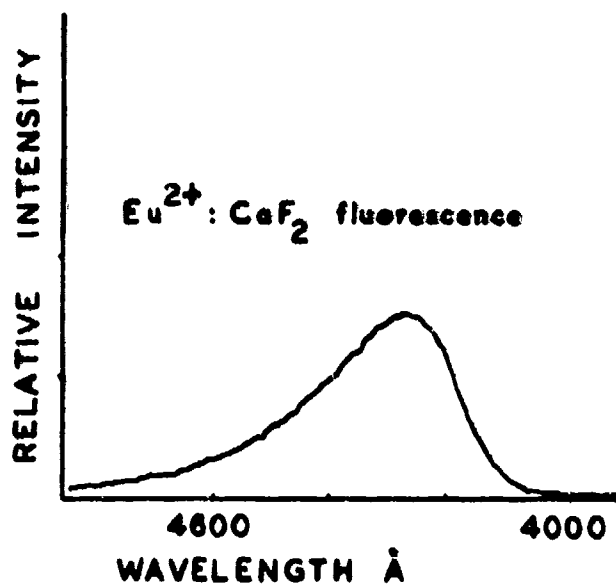


Figure 4. Fluorescence spectrum of 0.1% Eu^{2+} : CaF_2 .

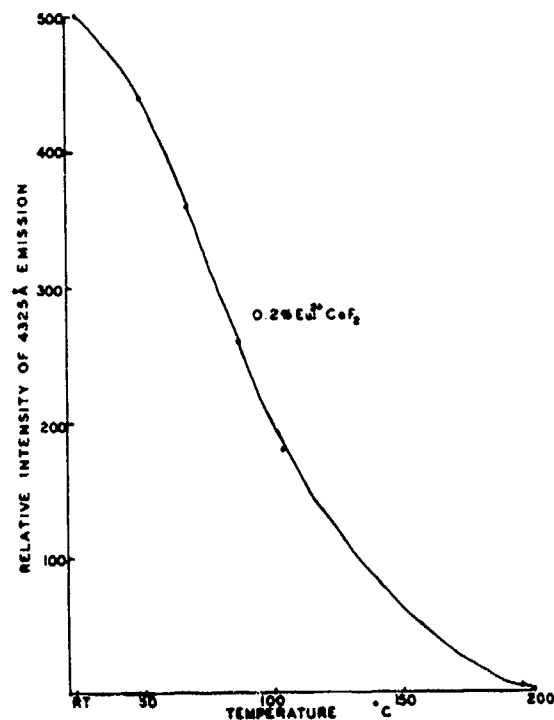


Figure 5. Temperature dependence of the intensity of the 4325 Å emission of 0.2% Eu^{2+} : CaF_2 .

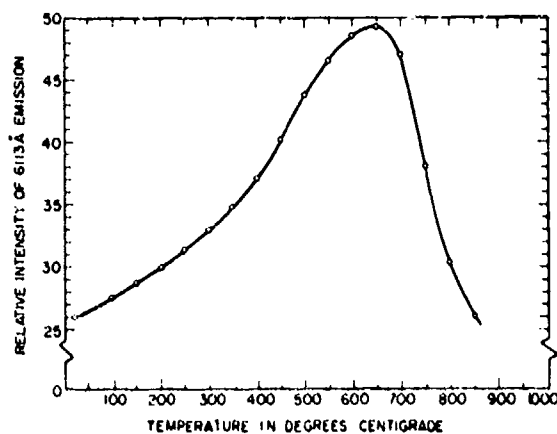


Figure 6. Intensity of the 6113 Å emission from 5% Eu^{3+} : Y_2O_3 under high pressure mercury excitation as a function of sample temperature.

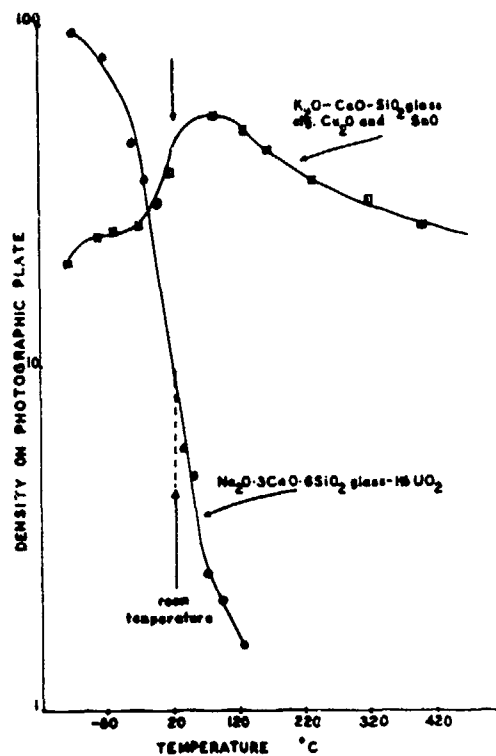


Figure 7. Temperature dependence of the fluorescence intensity of uranium and copper activated glasses.

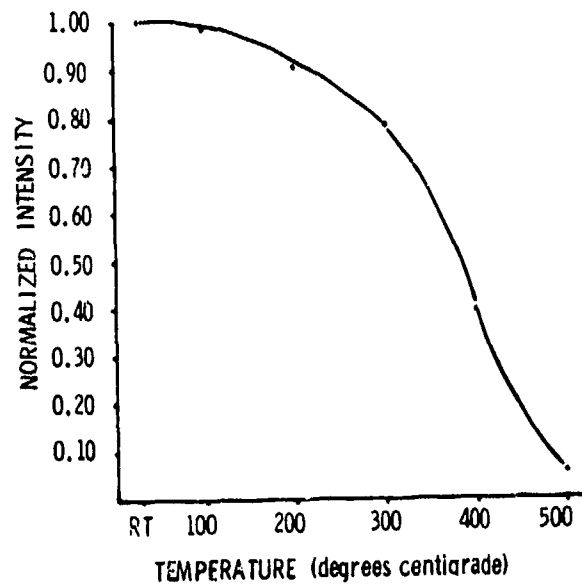


Figure 8. Temperature dependence of the normalized intensity of the fluorescence of $(\text{UO}_2)^{2+}$: LiF.

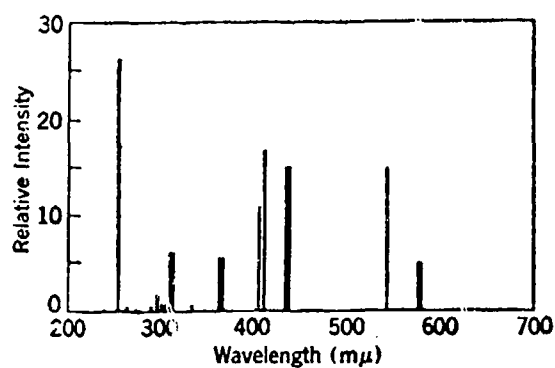


Figure 9. Relative intensity of lines emitted by a low pressure mercury vapor lamp.

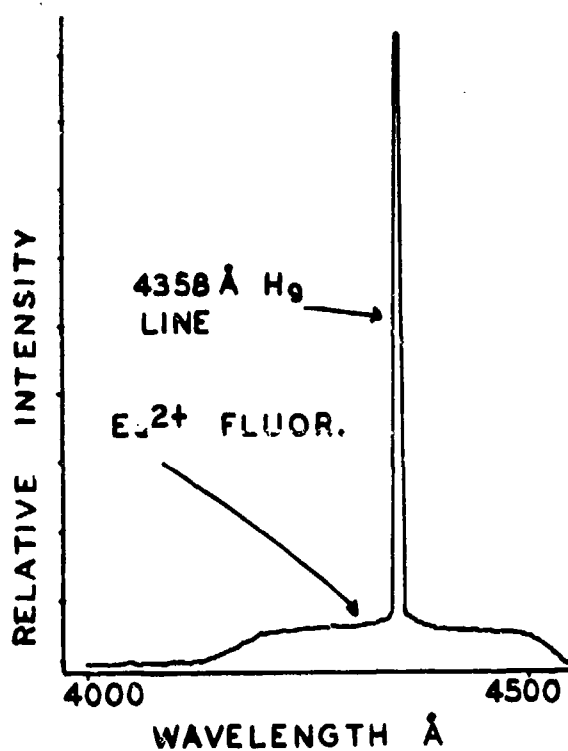


Figure 10. $\text{Eu}^{2+} : \text{CaF}_2$ fluorescent enhancement to the blue 4358Å mercury line.

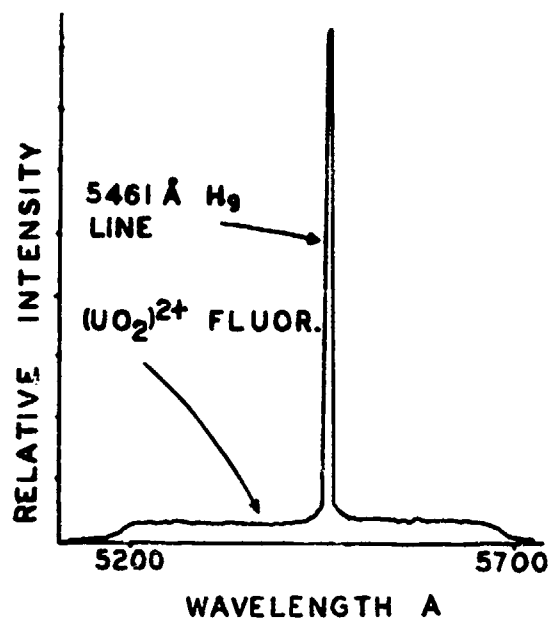


Figure 11. $(\text{UO}_2)^{2+}$; LiF fluorescent enhancement to the green 5461Å mercury line.