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NWSC/CR/RDTR-29

FLARES CONTAINING CALCIUM NITRATE AS AN OXIDIZER

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Naval Weapons Support Center
Applied Sciences Department
Crane, Indiana 47522

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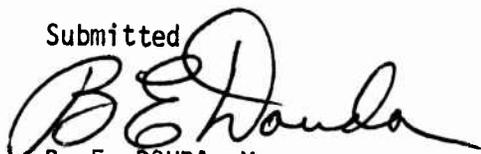
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FINAL REPORT for Period 1 July 1975 to 1 June 1976

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Prepared for
COMMANDER
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WASHINGTON, D. C. 20361

Submitted

A handwritten signature in cursive script, appearing to read "B. E. Douda". The signature is written in dark ink and is positioned to the right of the word "Submitted".

B. E. DOUDA, Manager
Chemical Sciences Branch
Pyrotechnic Division
Applied Sciences Department

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Flares composed of magnesium, sodium nitrate, calcium nitrate, and boron in varying ratios were prepared and tested. Luminous efficiencies, spectral power curves, and color data are presented for various formulas. Although no improvements in luminous efficiency were observed, significantly higher luminous intensity was observed in some cases when magnesium-calcium nitrate-sodium nitrate were compared with standard magnesium-sodium nitrate formulas. The dominant wavelength and excitation purity were essentially the same in all cases except when pure calcium nitrate-magnesium formulas were used. A detailed analysis of the visible spectra from magnesium-calcium nitrate and magnesium-calcium nitrate-boron flares is given.

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PREFACE

The authors would like to thank Mr. Forrest Burton for his assistance in making the candlepower measurements. The authors would also like to thank Mrs. Sondra Williams for typing the report and assuring that it was in the proper format.

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INTRODUCTION

During the past few years attempts have been made to produce illuminating flares whose spectral distributions were such that the resulting illumination was "white light". While many of the compositions tested produced white light, the luminous efficiencies were generally an order of magnitude less than the standard magnesium-sodium nitrate illuminating flare.¹ Preliminary results have been reported which indicate that when anhydrous calcium nitrate was partially substituted for sodium nitrate in a standard magnesium-sodium nitrate formula there was no loss in efficiency.² Similar results were obtained when calcium nitrate was substituted completely for sodium nitrate.³ No attempt was made to optimize the formula or to measure the color of the emitted light. Since calcium-oxygen species, e.g. CaO, CaOH and Ca₂O₂, emit strongly in the blue, green and red regions of the spectrum, a flare with calcium nitrate as part of the oxidizer might provide a means of producing high intensity white light.^{4,5}

¹H. A. Webster and C. W. Gilliam, *Spectral Characteristics of Flares Containing Sodium Iodate as an Oxidizer*, RDTR No. 276, Naval Ammunition Depot, Crane, Indiana (April 1974). Available DDC-AD 782510.

²J. E. Tanner, C. E. Dinerman, and H. A. Webster, *Effect of Barium and Calcium on Illuminating Flare Performance*, RDTR No. 281, Naval Ammunition Depot, Crane, Indiana (September 1974). Available DDC-AD 923559.

³D. Hart and H. J. Eppig, *Long Range Research on Pyrotechnics: Burning Characteristics of Binary Mixtures*, Technical Report No. 1669, Picatinny Arsenal, Dover, New Jersey (October 1947).

⁴R. W. B. Pearse and A. G. Gaydon, *The Identification of Molecular Spectra, Third Edition* (Chapman and Hall, Ltd, London, 1965).

⁵R. M. Blunt, *Evaluation of Processes Occurring in Pyrotechnic Flames*, RDTR No. 91, Naval Ammunition Depot, Crane, Indiana (March 1967). Available DDC-AD 655820.

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The purpose of this report is to describe the experiments which were performed to test this hypothesis. In addition, an analysis of the visible emission spectra obtained will be presented.

EXPERIMENTAL*

Flare Compositions

Based on the earlier results, the eight groups of flares listed in Table 1 were made. Flare groups WL29-WL32 were designed to test the effect of adding boron to the compositions containing calcium nitrate. Flare groups WL33-WL36 were designed to test the effect of adding calcium nitrate to a standard magnesium-sodium nitrate composition and to find the optimum calcium nitrate/sodium nitrate composition.

Except for the calcium nitrate, all the materials used in the manufacture of these flares were standard MIL-SPEC ingredients. The magnesium used was JAN-M-382, granulation 17. Barium nitrate was MIL-B-162B. The sodium nitrate was MIL-S-00322A. All solid materials were dried prior to use. The calcium nitrate was obtained in the hydrated form, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$. The hydrated material was oven-dried under vacuum at 150°C for 24 hours. This procedure completely removes the waters of hydration without decomposing the calcium nitrate. The anhydrous calcium nitrate was then ground and sieved at -30 mesh under an inert nitrogen atmosphere.

The epoxy resin used for the binder was a mixture of Dow resins**: DER 321, 61.2 percent; DER 732, 26.3 percent and CS 3482.1, a polyamine hardener, 12.5 percent. This is a standard Mk 45 binder composition.

After mixing, the compositions were pressed into paper tubes 5.5 cm in diameter. Approximately 300 grams of composition were used in flare groups WL29-WL32 and 450 grams in groups WL33-WL36 for each candle. The flare compositions were consolidated at $7.03 \times 10^6 \text{ kg/m}^2$ (10000 psi) pressing pressure.

*In order to specify procedures adequately, it has been necessary occasionally to identify commercial materials and equipment in this report. In no case does such identification imply recommendation, endorsement, or criticism by the Navy, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

**These names are trademarks of the Dow Chemical Company, Midland, Michigan.

TABLE 1. FLARE FORMULAS AND EXPERIMENTAL RESULTS

| | WL29 | WL30 | WL31 | WL32 | WL33 | WL34 | WL35 | WL36 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| Mg | 45 | 35 | 35 | 45 | 58 | 58 | 58 | 58 |
| B | 10 | 15 | 15 | 5 | -- | -- | -- | -- |
| NaNO ₃ | -- | -- | 5 | -- | 37 | 25 | 18 | -- |
| Ba(NO ₃) ₂ | -- | -- | -- | 15 | -- | -- | -- | -- |
| Ca(NO ₃) ₂ | 40 | 45 | 40 | 30 | -- | 12 | 19 | 37 |
| Epoxy | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Candlepower (kcd) | 311 | 269 | 351 | 166 | 222 | 265 | 299 | 210 |
| Efficiency (cd-sec/g) | 39200 | 33170 | 39850 | 21500 | 36800 | 33600 | 40500 | 31000 |
| Burn Time (sec) | 24.0 | 37.0 | 33.5 | 39.0 | 70.0 | 52.0 | 60.0 | 60.0 |
| x | 0.543 | 0.556 | 0.553 | 0.523 | 0.516 | 0.537 | 0.554 | 0.580 |
| y | 0.423 | 0.427 | 0.429 | 0.444 | 0.433 | 0.429 | 0.425 | 0.395 |
| dom λ (nm) | 589 | 589 | 588 | 585 | 856 | 587 | 589 | 594 |
| Purity | 91 | 96 | 96 | 91 | 86 | 91 | 94 | 94 |

Experimental Details

The flares were burned face down at a distance of four meters from the spectrographs. No attempt was made to isolate any specific area in the flame. Visible spectra were taken in the region from 400-700 nm with a 1.5 m grating spectrograph. This spectrograph has a 450 line/mm grating and gives a dispersion of 15 Å/mm in the first order. Kodak Linagraph Shellburst[®] film* was used for recording the spectra. The spectra were scanned and digitized on an Optronics S-2000 densitometer and the film density was converted to radiant and luminous power readings using the techniques described previously.¹

Measurements of the candlepower were made with a silicon photodiode equipped with appropriate filters so that the filter plus detector response match the standard CIE eye response curve. The photodiode was calibrated prior to the experiments by measuring the known value of candlepower from an NBS traceable standard 1000 watt quartz-iodine lamp. The output signal of the photodiode was integrated with a precision integrator to obtain an average candlepower reading. Integration was started three seconds after ignition and stopped approximately three seconds before the flare burned out.

RESULTS

The results of this work are summarized in Table 1. This table gives the average values of candlepower, burn time, luminous efficiency, color coordinates, dominant wavelength, and purity for all flares tested. The values quoted are averages of 7-10 different flares. The average experimental error in candlepower and efficiency is ± 5 percent.

The values of the chromaticity coordinates, x and y , were calculated by applying the color-matching functions, \bar{x} , \bar{y} , and \bar{z} , to the radiant power spectra at 1.0 nm intervals and integrating the area under the curve to obtain the tristimulus values X , Y and Z . The chromaticity coordinates were then obtained using the expressions

$$x = \frac{X}{X+Y+Z}$$

*Linagraph Shellburst is a registered trademark of the Eastman Kodak Co., Rochester, New York.

and

$$y = \frac{Y}{X+Y+Z}$$

The values of dominant wavelength and purity are determined by the standard graphical method after plotting the chromaticity coordinates on a chromaticity diagram.⁶ The average experimental error in the values of x and y is ± 2 percent.

The relative radiant and luminous power spectra for each flare group are plotted in Figures 1-16. The luminous power spectrum is obtained by applying the CIE color-matching function, \bar{y} , to the radiant power spectrum. The area under the luminous power curve is then directly proportional to the candlepower. In order to show as much detail as possible, each spectrum is normalized to a value of one at its maximum. The spectra which are shown were selected as being representative of the flare group. The criteria used for this selection was to choose the spectrum which had chromaticity coordinates the same as the average value presented in Table 1. It should be pointed out that none of the spectra within a given flare group differed significantly from the ones presented.

DISCUSSION

General Observations

Substitution of $\text{Ca}(\text{NO}_3)_2$ for NaNO_3

The results of this work are in general agreement with previously reported work. A one hundred percent substitution of calcium nitrate for sodium nitrate reduces candlepower, burn time, and efficiency. The substitution of fifty percent calcium nitrate for sodium nitrate (flare group WL35) significantly increases the candlepower but the composition burns faster. The overall effect is a small increase in efficiency. The substitution of twenty-five percent calcium nitrate does not significantly affect the efficiency. Within the measurement limits of these experiments, there is no change in efficiency except when pure calcium nitrate is used as the oxidizer.

⁶G. Wyszecki and W. S. Stiles, *Color Science: Concepts and Methods, Quantitative Data and Formulas* (John Wiley and Sons, Inc., New York, 1967).

The dominant wavelength shifts to longer wavelength as calcium nitrate is substituted for sodium nitrate. The flares containing only calcium nitrate, WL36, have a dominant wavelength of 594 nm and are reddish orange in color while the sodium nitrate flares, WL33, have a dominant wavelength of 586 nm and are yellowish white. The flares containing mixtures of sodium nitrate and calcium nitrate have dominant wavelengths of 587 and 589 nm which fall between those of the flares containing only the pure oxidizer. There is no decrease in the excitation purity and these flares appear to be yellowish white and indistinguishable from the sodium nitrate flare.

Addition of Boron

The addition of boron at the ten and fifteen percent level in a flare containing only calcium nitrate as the oxidizer does not significantly change the efficiency. The boron-calcium nitrate-sodium nitrate flares, WL31, show significant increases in candlepower compared to standard illuminating flares while burning much faster. The overall effect is a slight increase in efficiency.

The substitution of barium nitrate for calcium nitrate in a boron-calcium nitrate flare greatly reduces the candlepower and the efficiency. The same effect is seen when barium nitrate is substituted for sodium nitrate in a standard illuminating flare. The intensity of barium oxide emission is remarkably less than that of most of the other molecular species.

The addition of boron to a calcium nitrate flare shifts the dominant wavelength to shorter wavelength as one would expect. The dominant wavelength of 588 nm obtained is the same as the dominant wavelength obtained when mixtures of calcium nitrate and sodium nitrate are used as oxidizers. The excitation purity of the boron-calcium nitrate flares is still very high and the flares appear to be yellowish white in color. The color is indistinguishable from the magnesium-sodium nitrate flares.

Analysis of the Spectra

The relative radiant and luminous power spectra for a standard magnesium-sodium flare, WL33, are shown in Figures 1 and 2. These spectra have been discussed and analyzed in

great detail in earlier reports.^{7,8} For this reason, no detailed discussion will be given. The prominent features are the MgO emission at 500.7 nm, the sodium doublets at 568 nm and 615 nm and the broadened resonance line emission centered at 590 nm.

The relative radiant and luminous power spectra for a magnesium-calcium nitrate flare, WL36, are shown in Figures 3 and 4. The emission features in these spectra are essentially all from molecular species. The emission at 500.7 nm is due to MgO. Emission from the CaO $B^1\Sigma \rightarrow X^1\Sigma$ is observed from 420 nm to 450 nm. This emission is much weaker than any other emission in the system and does not show in the normalized spectra. Spectra taken in the infrared region, 720 nm to 840 nm, show the CaO $A^1\Sigma \rightarrow X^1\Sigma$ system.

The most prominent features in the calcium nitrate spectra are the band emissions at 555 nm, 573 nm, 604 nm, 623 nm and 642 nm. These emission features are due primarily to CaOH.⁴ The bands overlap another band system due to a diatomic calcium oxide species, Ca_2O_2 . The strong emission at 623 nm is the emission primarily responsible for the reddish orange color observed in the calcium nitrate flare. Atomic emission from calcium is observed at 423 nm, from sodium at 589 nm and from lithium at 671 nm.

Figures 5 and 6 and Figures 7 and 8 show the changes which result from the addition of sodium nitrate to a magnesium-calcium nitrate flare. In Figure 5, flare group WL35, the ratio of sodium nitrate to calcium nitrate is approximately one to one and the CaOH emission at 623 nm is still the overriding feature. The broadened sodium resonance lines centered at 589 nm are the emissions which shift the dominant wavelength to a shorter wavelength.

In Figure 7, flare group WL34, the ratio of sodium nitrate to calcium nitrate is two to one and the emissions from sodium at 589 nm contribute more than the CaOH emission

⁷ H. A. Webster, *Alkali Metal Emitters: I. Analysis of Visible Spectra*, NWSC/CR/RDTR-16, Naval Weapons Support Center, Crane, Indiana (1975). Available NTIS-AD-A019367.

⁸ B. E. Douda, *Radiative Transfer Model of a Pyrotechnic Flame*, RDTR No. 258, Naval Ammunition Depot, Crane, Indiana (1973). Available DDC-AD 769273.

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at 623 nm and the dominant wavelength again shifts to shorter wavelength. The absence of any emission in the blue and green regions of the spectrum accounts for the very high excitation purities which were observed.

The effect of adding boron to a magnesium-calcium nitrate flare is shown in Figures 9 and 10, flare group WL29, and Figures 11 and 12, flare group WL30. The emission features at 555 nm, 623 nm and 642 nm are again due to CaOH. Emission at 462 nm and 475 nm is very weak but present and is due to the $A^2\Pi \rightarrow X^2\Sigma$ BO α system.⁴ As in the case of CaO, these emissions are too weak to be shown on the normalized spectra.

Emission from a boron species is observed at 541 nm, 565 nm and 590 nm. These bands are possibly the "boric acid fluctuation bands" at 545 nm and 580 nm which are attributed to BO_2 overlapped by some BO bands.⁴ The discrepancy in wavelengths and the absence of some of the other fluctuation bands makes this assignment tentative at best. Spectra taken of flares composed of magnesium, barium nitrate, and boron do not appear to show these bands. This result could mean that the strong emissions at 565 nm and 590 nm are previously unreported emissions from a boron-calcium interaction.

Figures 13 and 14, flare group WL31, show the effect of adding a small amount of sodium nitrate, 5 percent by weight, to the magnesium-boron-calcium nitrate formula. Since the sodium concentration was low, the resonance lines at 589 nm are broadened less than in a standard magnesium-sodium nitrate flare. This behavior is in good agreement with the results predicted by the radiative transfer model.⁵ All the other calcium and boron oxide features are still present.

Figures 15 and 16 show the relative radiant and luminous power spectra of flare group WL32 in which barium nitrate was added to a magnesium-boron-calcium nitrate flare. In these spectra BaO emission is observed at 480 nm, 510 nm and 520 nm. The red BaO system is probably masked by the CaOH emission. It should be noted that while the boron concentration was only 5 percent by weight in this formula, the anomalous boron bands at 565 nm and 590 nm are still reasonably strong. This observation tends to substantiate a possible boron-calcium interaction. Even though there is now increased emission in the green spectral region, it is not strong enough to override the red emission and the excitation purity does not change.

CONCLUSIONS

The addition of calcium nitrate to a standard magnesium-sodium nitrate formula does not decrease the luminous efficiency significantly. The dominant wavelength shifts to longer wavelength and the excitation purity remains constant. Molecular emission from CaOH at 555 nm and 623 nm make up the bulk of the luminous output.

The addition of boron to a magnesium-calcium nitrate composition significantly increases the candlepower output but causes the flare to burn much faster. The overall result is a slight reduction in efficiency. Molecular emission from CaOH at 555 nm and 623 nm and molecular emission from a boron species, possibly BO_2 , at 565 nm and 590 nm make up the bulk of the emission. The assignment of the boron emissions at 565 nm and 590 nm to BO_2 is tentative since (1) these bands are not at the same wavelengths as those generally attributed to BO_2 and (2) these bands are not apparently present in flares containing only magnesium, boron and barium nitrate. It is possible that these bands are due to a calcium-boron interaction.

The excitation purity of magnesium-boron-calcium nitrate flares is high and the color is yellowish white. The substitution of small amounts of barium nitrate for calcium nitrate in these compositions significantly reduces both the candlepower and efficiency. The green BaO emission bands are present but weak and do not substantially change the excitation purity. None of the formulas tested produced white light.

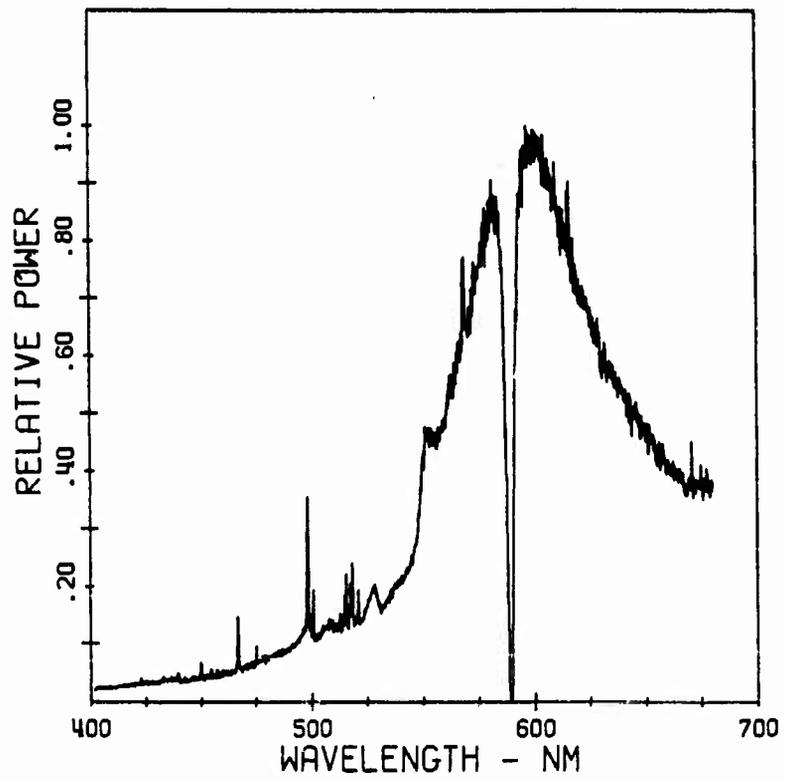


Figure 1. Radiant Power--Group WL33

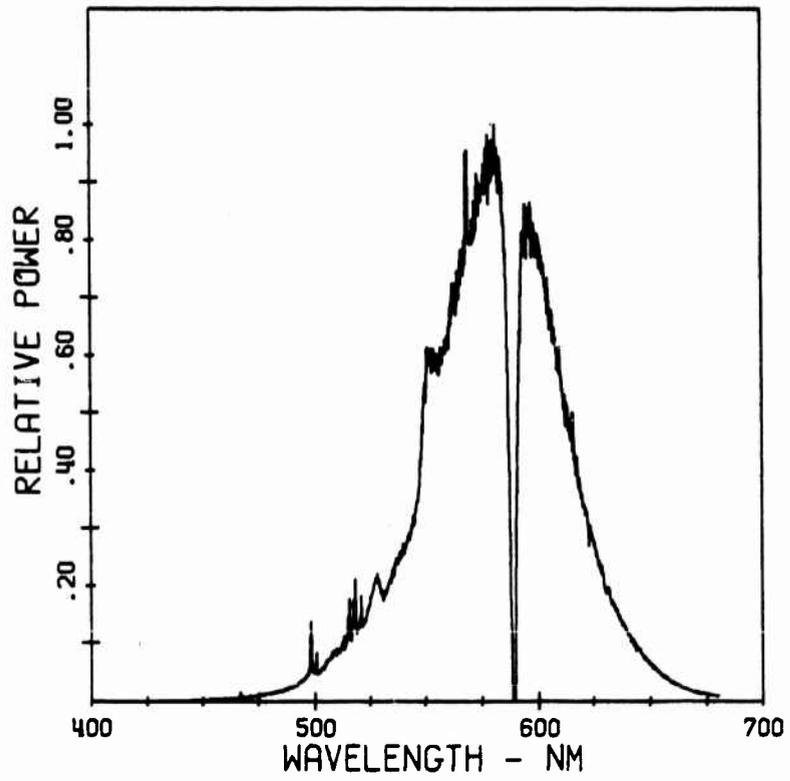


Figure 2. Luminous Power--Group WL33

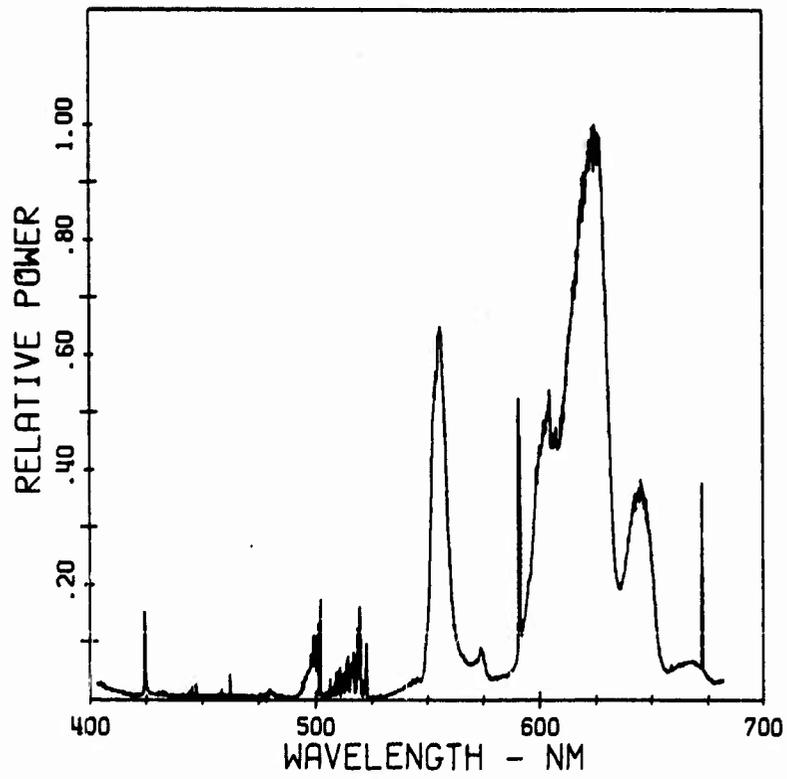


Figure 3. Radiant Power--Group WL36

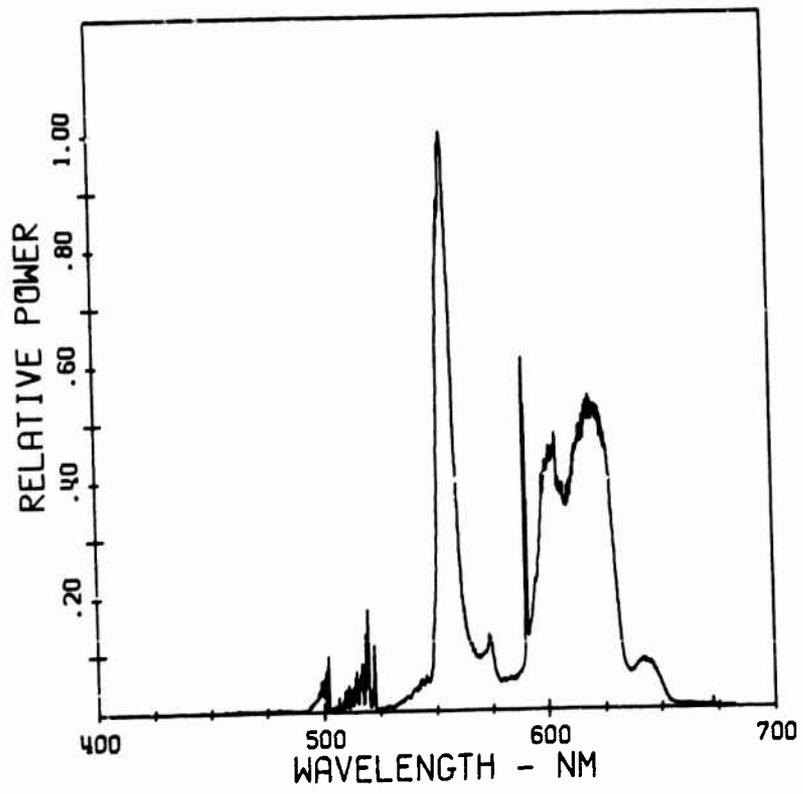


Figure 4. Luminous Power--Group WL36

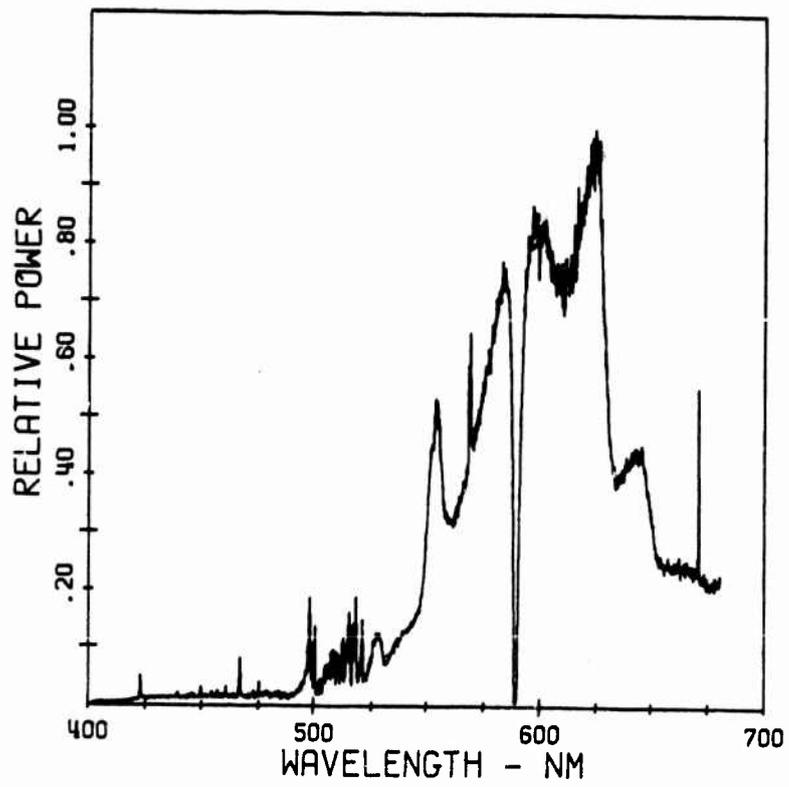


Figure 5. Radiant Power--Group WL35

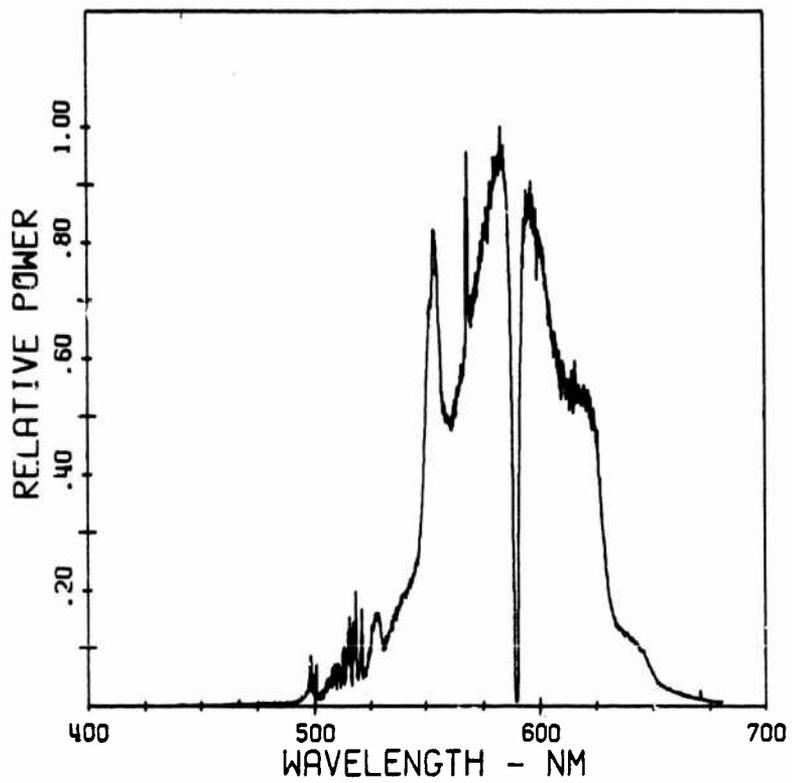


Figure 6. Luminous Power--Group WL35

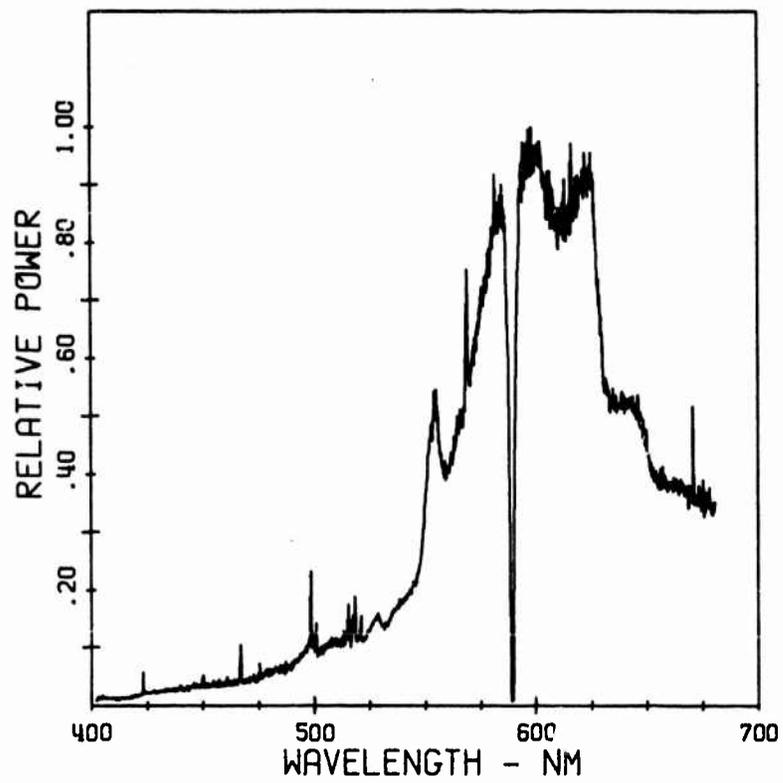


Figure 7. Radiant Power--Group WL34

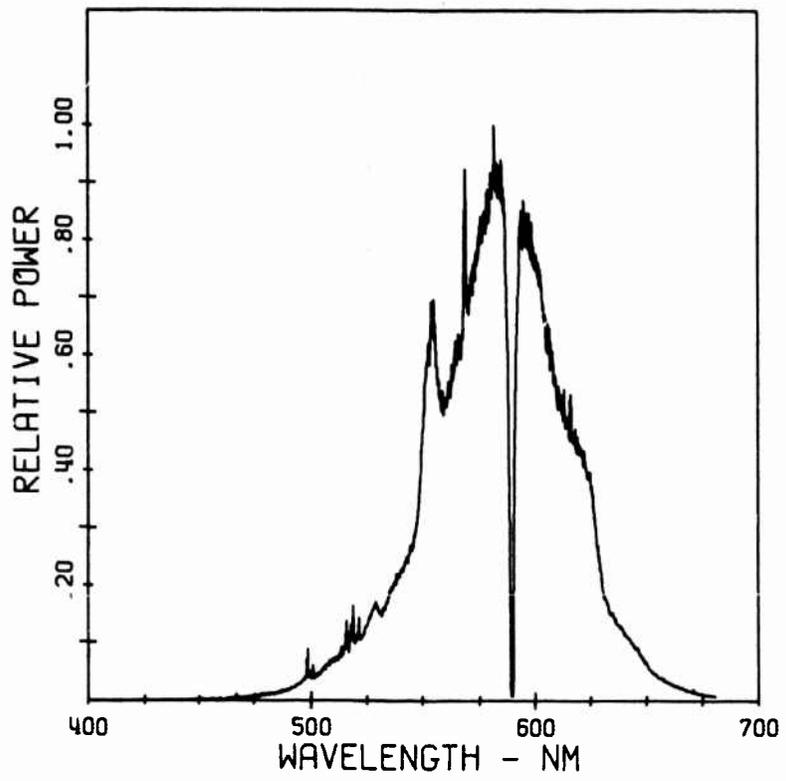


Figure 8. Luminous Power--Group WL34

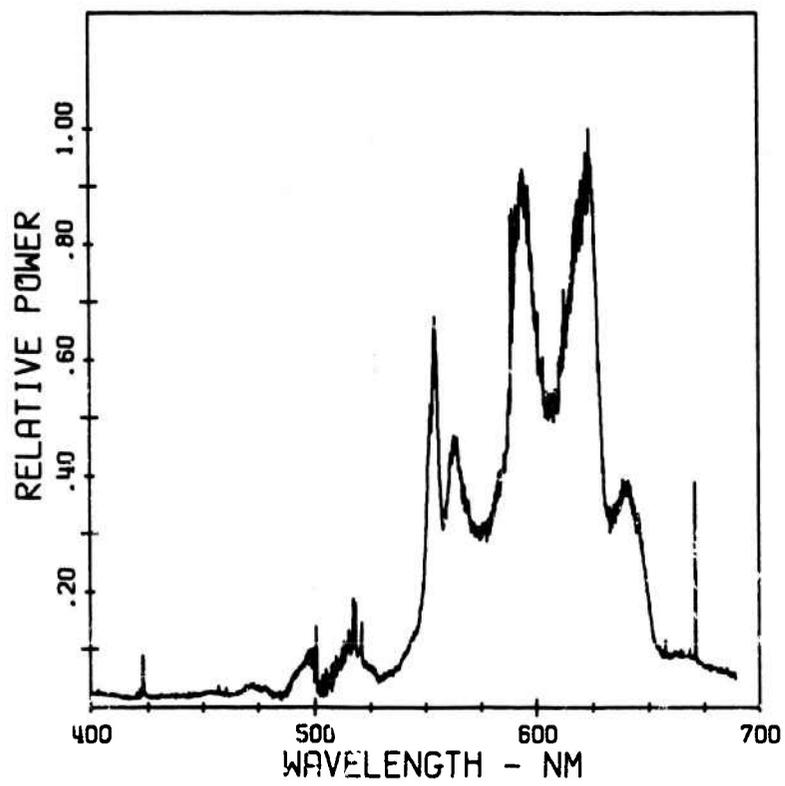


Figure 9. Radiant Power--Group WL29

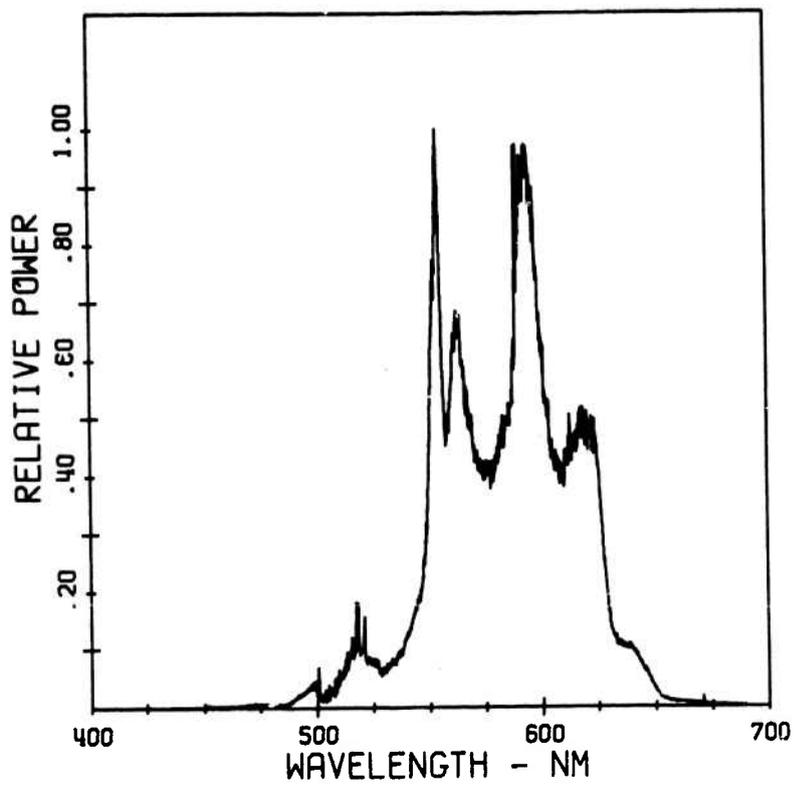


Figure 10. Luminous Power--Group WL29

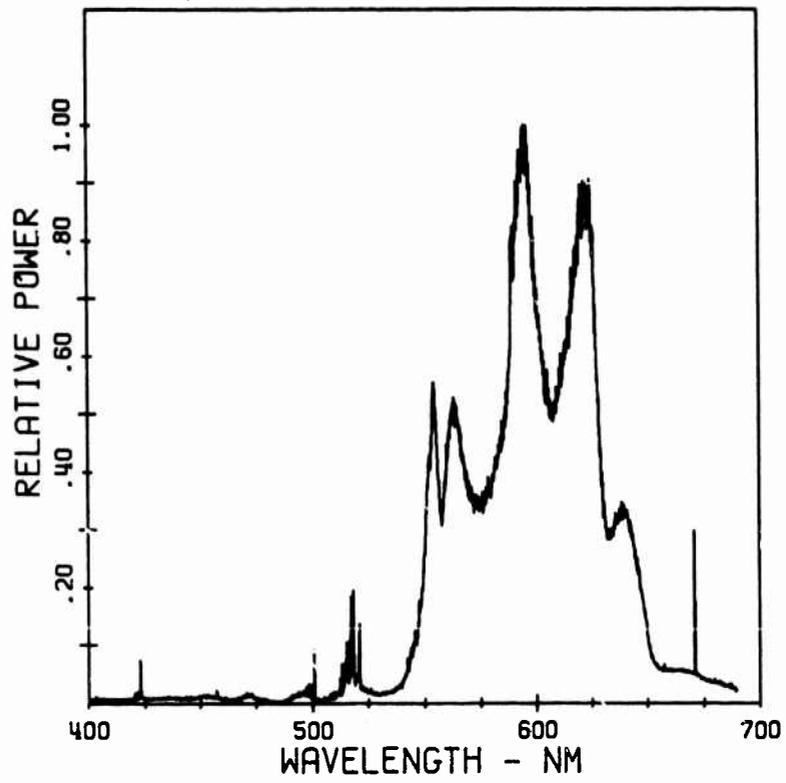


Figure 11. Radiant Power--Group WL30

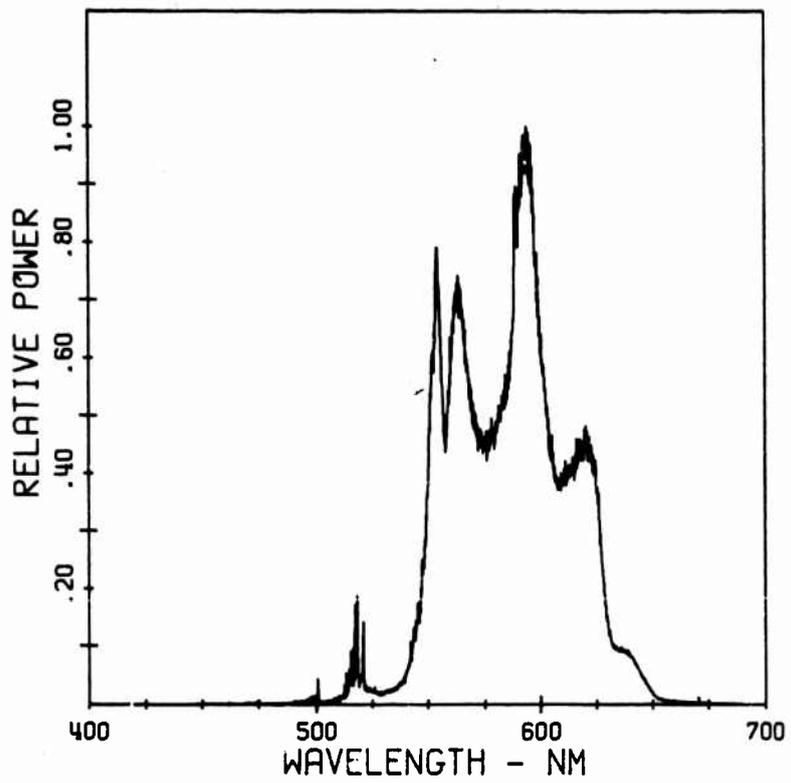


Figure 12. Luminous Power--Group WL30

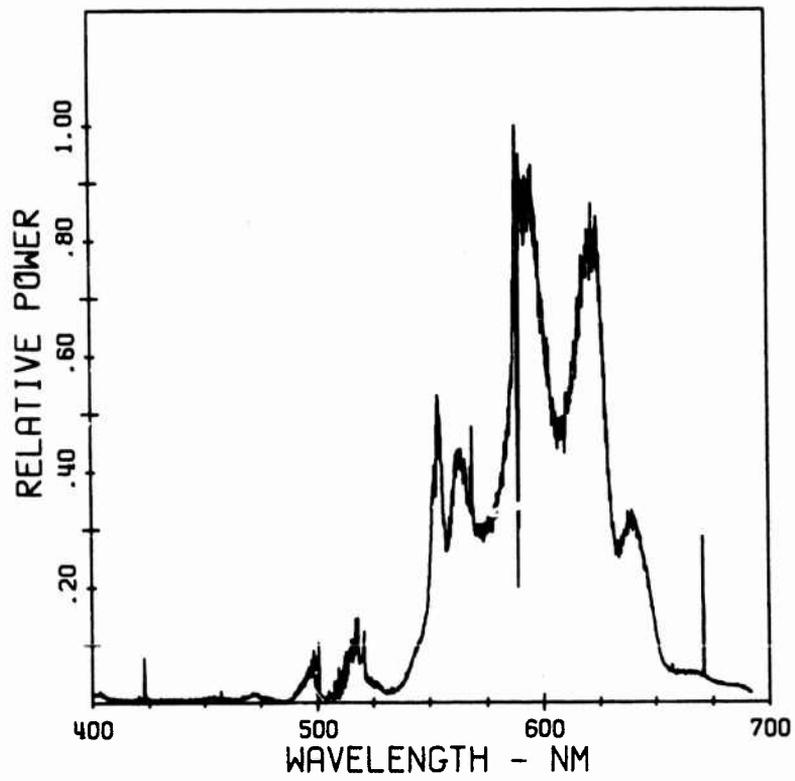


Figure 13. Radiant Power--Group WL31

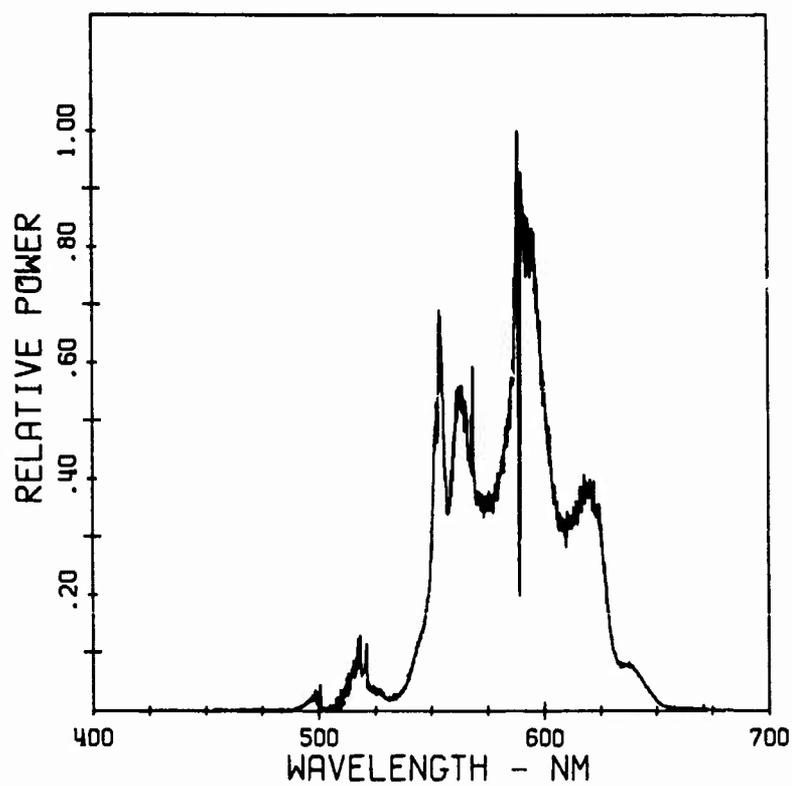


Figure 14. Luminous Power--Group WL31

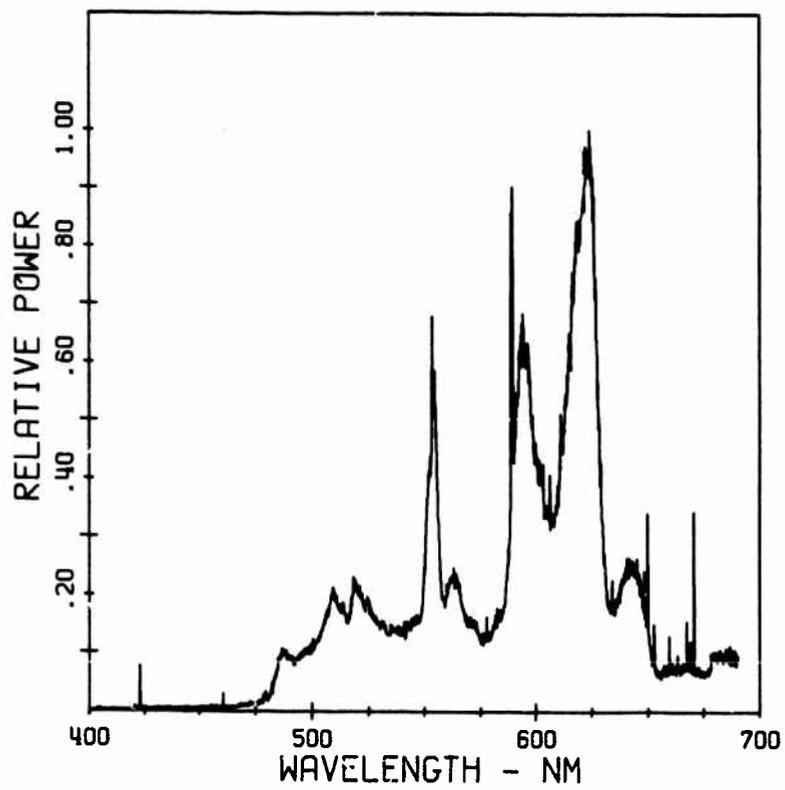


Figure 15. Radiant Power--Group WL32

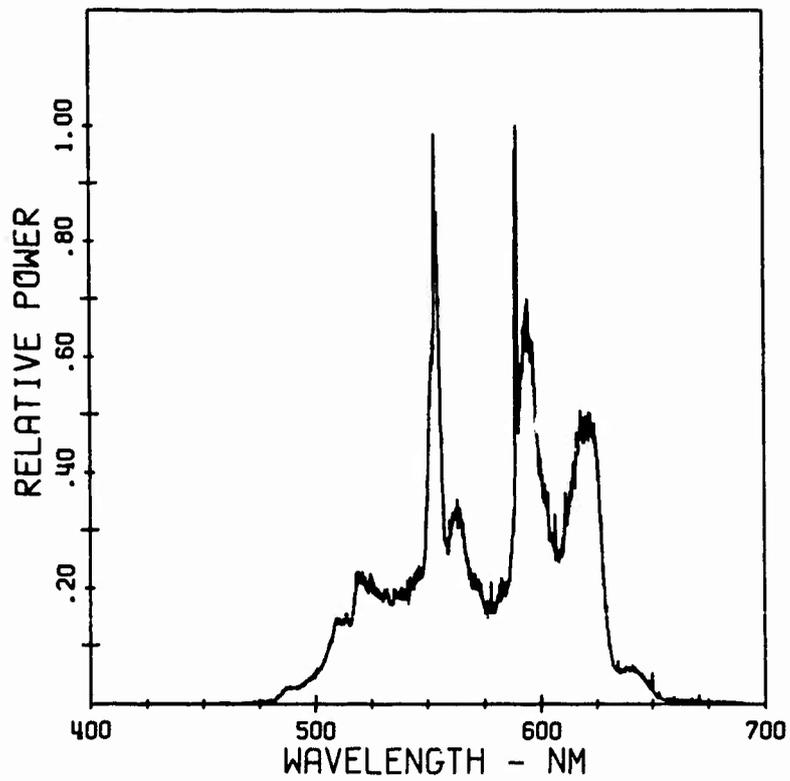


Figure 16. Luminous Power--Group WL32

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