

FLUORESCENCE OBSERVATIONS OF LDEF EXPOSED MATERIALS AS AN INDICATOR OF INDUCED MATERIAL REACTIONS

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INTRODUCTION

Observations and measurements of induced changes in the fluorescent emission of materials exposed to the space environment on the Long Duration Exposure Facility (LDEF) have revealed systematic patterns of material-dependent behavior. These results have been supplemented by inspection of similar materials exposed on previous space shuttle missions and in laboratory testing. The space environmental factors affecting the fluorescence of exposed materials have been found to include (but are not necessarily limited to) solar ultraviolet (UV) radiation, atomic oxygen (AO), thermal vacuum exposure, and synergistic combinations of these factors. Observed changes in material fluorescent behavior include stimulation, quenching, and spectral band shifts of emission. For example, the intrinsic yellow fluorescence of zinc oxide pigmented thermal control coatings undergoes quenching as a result of exposure, while visible coloration is stimulated in the fluorescent emission of several polyurethane coating materials. The changes in fluorescent behavior of these materials are shown to be a revealing indicator of induced material reactions as a result of space environmental exposure.

BACKGROUND

Light emission processes excluding thermal and electrical excitation are generally referred to as luminescence. Photoluminescence is further defined as UV radiation-stimulated light emission from chemical compounds. Fluorescence, as usually defined and as used in this paper, is then photoluminescence distinguished by the lifetime of excited states (usually on the order of 10 nanoseconds). Quenching usually refers to the deactivation of a molecule from an excited electronic state without the emission of a photon. Suppression of emission by absorption of another compound in the excited material (the "inner filter effect"), though not strictly "quenching," has the same effect (ref. 1). In this paper, "quenching" refers to the decreased fluorescent emission of materials as a result of exposure.

Most of the observations and measurements of fluorescence in the materials of interest were made with flight specimens from two LDEF experiments. These two experiments were A0171 (Solar Array Materials LDEF Experiment) and A0034 (Atomic Oxygen Stimulated Outgassing). Experiment A0171 was mounted on row 8 of the LDEF, oriented 38° from the leading edge RAM velocity vector (ref. 2). This passive full-tray experiment included six plates of solar array and solar concentrator component elements and candidate materials, including thermal control coatings, protective coatings, composites, metals, and polymeric materials (Figure 1). Experiment A0171 materials were exposed to an AO fluence of $6.93E21$ atoms/cm² and approximately 10,000 equivalent Sun hours (esh) of solar radiation. Experiment A0034 included two separate modules of thermal control coatings and collector mirrors, one mounted on the leading edge (row 9) and one mounted on

the trailing edge (row 3). The coatings were housed at the base of aperture-limited compartments in the modules with adjacent witness mirrors to provide an assessment of the role of AO in the outgassing of thermal control coatings (ref. 3). In the compartmentalized design of experiment A0034, coatings that were exposed to the combined space environment under open apertures were duplicated for exposure under sealed ("covered") apertures and apertures sealed with UV-grade optical windows (Figure 2). Thermal control coatings in the modules of experiment A0034 were exposed to an AO fluence of $8.72E21$ atoms/cm² on the leading edge and $1.32E17$ atoms/cm² on the trailing edge. As a result of the aperture limited exposure of the coatings on the leading and the trailing edges, the exposure to solar radiation was considerably limited and is estimated to be less than 2,000 esh.

Since the 5.8-year life in low-Earth orbit (LEO) of the LDEF mission exceeded all previous space exposure doses for retrieved material specimens, it was considered relevant to review the fluorescent behavior of similar materials from shorter space missions. For this purpose, stored specimens of materials from AO experiments flown on STS 5, 8, and 17 (41-G) (references 4,5) were reexamined under black light illumination and the spectral fluorescence measured. Materials of the STS-5 AO experiment were exposed to an estimated fluence of $1.0E20$ atoms/cm², while materials of the other two experiments accumulated fluences of $3.5E20$ and $3.0E20$ atoms/cm², respectively. The exposure to solar UV radiation during these relatively short duration missions (nominal seven days) was inherently limited. The comparison of induced fluorescence behavior in material specimens of these early shuttle flights to LDEF mission results is constrained by the unknown changes induced by years of laboratory environment storage; however, recent results from laboratory testing and more recent flight experiments (STS-46) indicate that the general nature of these results are valid for comparison. Other observations and measurements of some of the materials to be discussed in this paper have been previously published based on materials exposed in LDEF experiments S0069 and AO114 (ref. 6).

Fluorescence in the materials to be discussed is readily visible with conventional black-light illumination. These types of observations are useful indicators of induced changes in emission coloration as a result of exposure, and the observations generally confirm and even supplement the more detailed information obtainable from instrumented spectral fluorescence measurements. Fluorescence observations reported in this paper were obtained using both "long wave" and "short wave" illumination sources. The "long wave" source is a Dart Products B100A "Blak-Ray," providing approximately 7 mW/cm² irradiance at 15 cm, at a peak wavelength of 365 nm (320 to 380 nm). This filtered mercury emission source produces bright, rich colors in the NIST yellow and blue fluorescence standards (SRM 1931) stimulated by wavelengths in excess of 340 nm, while producing no visible color in the green standard (excitation wavelength 280 nm) and only a very dim glow in the orange (235 nm excitation). All four of the NIST standards fluoresced brightly under "short wave" excitation, using a Spectrolin B-14F 15 W lamp with $1,100$ mW/cm² irradiance at 30 cm (peak wavelength 254 nm). For purposes of comparison to the detailed spectral fluorescence data to be discussed, a summary of the visible observations with these black light sources is provided in Table 1.

Measurements of fluorescence in the materials of interest were obtained using an SLM Aminco SPF-500C dual monochromator spectrofluorometer (see Table 2 for specification). Narrow-band filters at the exit slit of the excitation source and excitation wavelength-absorbing glass at the entrance aperture of the emission port were used to exclude extraneous light. For most of the materials examined to date, a narrow-band excitation of 260-nm wavelength was used. The results indicate a (nearly invisible) UV band of fluorescent emission for most of the materials and, for many of these materials, one or more bands of visible emission. These results are summarized in tabular form for most of the materials discussed in this paper in Table 3. It is necessary to state that the "assignment" of a peak wavelength to fluorescent bands of often very broad wavelength width is a

highly speculative endeavor and is only attempted here to provide a measure of comparison for different environmental exposures and materials. The spectral fluorescence curves presented in this paper include the results of “short wave” black light observations for comparison.

RESULTS

Changes in material fluorescence as a result of space environmental exposure were revealed as one or more of the following effects:

1. Quenching of emission
2. Stimulated emission (new or altered color)
3. Emission wavelength shift
4. Enriched emission (same color).

Induced quenching of the UV and visible fluorescent emission bands of S13G-LO paint (Figure 3) from LDEF experiment A0171 is characteristic of all of the investigated zinc-oxide pigmented thermal control coatings (S13G, S13G-LO, Z93, and NS43C). The level of quenching was found to be proportional to the degree of exposure to the space environment. Fluorescent quenching of Z93 (Figure 4) resulted from exposure on the leading edge of the LDEF, with relatively high levels of AO and solar UV radiation, and from exposure on the trailing edge, with a similar level of UV irradiance and orders of magnitude less AO (the small feature of the fluorescence curves for Z93, peaked at 780 nm, is considered to be third-order reflected source light). Exposure of these coatings under sealed UV-grade quartz windows on the LDEF modules resulted in similar quenching, though diminished in magnitude. Further testing and analysis is required to understand the apparent quenching of the Z93 coating exposed under a metal cover (“closed”) during flight (Figure 5). Experiment A0034 coatings exposed in this manner experienced the flight thermal vacuum environment in the absence of AO and UV radiation. Quenching of a similar nature has been found for S13G-LO coatings exposed on limited duration space shuttle missions (Figure 6). For these shuttle missions, the level of solar UV irradiance is estimated to have been less than 100 ESH. Low levels (400 esh or less) of laboratory UV irradiation of these type coatings have not resulted in detectable quenching of fluorescence. In other laboratory testing, AO exposure alone resulted in quenching of the UV and visible fluorescent emission of the white conductive zinc-oxide pigmented paint NS43C (Figure 7). This exposure was done in the MSFC Drift-Tube system, providing approximately $1E21$ atoms/cm² of thermal energy AO at ambient temperature in the absence of UV radiation. The emission peaked at 780 nm in Figure 7 is third-order reflected source light.

Stimulated emission can be clearly seen (Figure 8) in the response to exposure of bulk RTV 511 resin material from A0171, in addition to quenching of the intrinsic UV fluorescence band and alteration of the intrinsic “red” emission. Direct (“open”) exposure of Chemglaze A276™ on the leading edge of the LDEF (experiment A0034) resulted in apparent total erosion of the polyurethane binder from the titanium dioxide pigment and effectively total quenching of fluorescence (Figure 9). Adjacent A276™ material exposed to UV radiation under a sealed quartz window was visibly darkened, resulting in a stimulated yellow/orange fluorescent glow under black light. Laboratory testing using near UV sources (mercury-xenon arc lamps) at one “UV Sun” intensity level showed that this stimulated fluorescence results from less than 100 esh irradiance. A final example of laboratory exposed material (Figure 10) is provided summarizing the effects of thermal energy AO, near-UV

irradiation, and material temperature on the fluorescence of a candidate silicone seal material S383. The stimulated emission following these various types of exposure is visible as a yellowish glow under black light illumination. Testing further indicated that exposure to AO alone did not perceptibly change the intrinsic fluorescence of this material. Exposure to UV radiation, whether alone or following AO exposure, was done in vacuum to a level of approximately 1,700 esh. These tests revealed a visible fluorescent glow under black light following only a very few hours of UV irradiation, similar in effect to testing results with the Chemglaze polyurethane coatings; comparison of the fluorescence spectra of control S383 to control Chemglaze Z306™ (Figure 19) indicates similar structure at the short and long wavelength ends of the spectra. From Figure 10, it can be seen that the effect of elevated temperature on materials exposed sequentially to AO and UV radiation was in reducing the intensity of stimulated fluorescence compared to ambient temperature test sequencing. This is presumably a result of enhanced reactivity of the S383 to AO at the elevated temperature. In contrast, elevated temperature exposure to UV radiation alone of the silicone S383 material resulted in enhanced stimulation of fluorescent glow (Figure 10).

A definitive example of an emission wavelength shift is provided in Figure 11 for A0171 PEEK resin material. An emission wavelength shift is considered to be distinguished from stimulated emission by the degree of stimulation of significantly new or altered coloration in the black light illuminated glow.

Enriched emission as a result of space exposure on PMR neat resin (Figure 12) from A0171 is the rarest effect revealed in the examined materials. This effect in the PMR neat resin is accompanied by quenching of emission in the UV bands.

DISCUSSION

Measurements of spectral fluorescence distributions in solid materials are subject to variability resulting from ambient laboratory changes in temperature and humidity. These effects were found most pronounced in the zinc oxide-pigmented thermal control coating, as the differences in the measurements on the same flight sample (S13G-LO) on two different dates show (Figure 12). Significant differences are found in the fluorescence distributions of another S13G-LO flight sample when artificially heated compared to ambient environment measurements (Figure 14). Differences were also found for samples stored in desiccators prior to measurement (Figure 15). Measurements of the fluorescence distributions of materials investigated for this paper were all completed, for each set of samples including controls, in a timely sequence equalizing the laboratory environment following desiccator conditioning. In addition, frequent reference to measurements of the NIST fluorescence standards provided confidence in the wavelengths of identified spectral features and provided a means of normalizing intensity levels.

Two exceptions were found to the general classification of environmentally induced fluorescent behavior based on material commonalty. These exceptions are in specimens of YB-71 (Illinois Institute of Technology Research Institute's zinc orthotitanate) and Lord Chemical's Chemglaze Z306™.

Visible observations under black-light illumination of fluorescent emission from unexposed or exposed YB-71 coatings reveal only varying shades of reddish-purple coloration. Two distinct bands of emission on opposite ends of the visible spectrum are revealed in fluorescence measurements of unexposed YB-71 coatings (Figures 16 and 17). In general agreement with the observations, for LDEF leading edge A0034 coatings of YB-71, the effects of "open" exposure to the combined space

environment are complex (Figure 16); the principal emission band at approximately 420 nm is decreased in intensity with (poorly resolved) evidence of associated stimulated emission bands. The reddish emission (peaked at 710 nm) is relatively unchanged as a result of the exposure. LDEF trailing edge exposure (Figure 17), with four orders of magnitude less AO for “open” samples, also resulted in decreased emission (quenching) of the 420-nm band, although the “new” bands of resolved emission are significantly different. Exposure of YB-71 under a UV-grade quartz window (“UV only”) on the trailing edge resulted in a more intense band of spectrally shifted emission than “open” exposure that appears similar in resolved structure. Flight exposure of YB-71 under a metal cover (“vacuum only”) also resulted in quenching and a somewhat different resolved emission band pattern.

Exposure-induced changes in fluorescence of Chemglaze Z306™ coatings were found to be somewhat material-batch dependent, although laboratory testing as well as flight results does generally result in stimulated emission under black-light illumination. The effects of “open” exposure on LDEF experiment A0034 (leading edge) are apparently slightly spectrally shifted, enhanced emission intensity of all three of the intrinsic emission bands. Exposure restricted to solar UV radiation resulted in a new single broadband of stimulated emission, in agreement with black light observations. In contrast, exposure on STS-5 and STS-8 resulted in wavelength band dependent variations in quenching and stimulated, spectrally shifted emission (Figures 18 and 19). These data also indicate differently resolved patterns of emission in the unexposed control material. These differences in the control coatings of Z306™ are visibly apparent under black light illumination. Despite these differences in the fluorescence of flight exposed Z306™ coatings, there is a systematic pattern of decreasing emission wavelength correlated with increasing integrated flux (fluence) of AO (Table 4), based on the apparent middle band of emission. These results do not correlate with accumulated solar UV radiation exposure.

Extended exposure of organic materials to the intense sources required for spectrofluorometric investigations can lead to sample surface photodecomposition (ref. 1). This effect was most pronounced with the polyurethane binder coatings exposed to the spectrally dispersed exit beam of the SLM SPF-500C excitation monochromator. The photodecomposition following exposure was readily observed visually under black light illumination as a distinct image of the emission monochromator exit beam. Laboratory procedures optimizing the minimum beam intensity and minimizing the time allotted for measuring exposure eliminated detectable photodecomposition.

Optical measurements of fluorescent materials using conventional integrating spheres for hemispherical reflectance detection are subject to error at UV wavelengths. The photomultiplier detector, usually operating with the highest gain sensitivity at UV wavelengths, cannot distinguish surface-reflected light from excited fluorescent emission. This effect can lead to the variable fluorescent emission in the exposed materials being misleadingly interpreted as variations in diffuse reflectance at UV wavelengths in integrating spheres. This proved particularly significant for thermal control coatings of intrinsically low diffuse reflectance for wavelengths less than 400 nanometers. The effect was verified by measurements of diffuse reflectance of visibly fluorescing coatings in a Gier Dunkel integrating sphere with the photomultiplier detector, at excitation wavelengths, alternately covered and uncovered by a transparent (UV opaque) filter. Though clearly a measurable effect, testing appears to indicate that the effect is minimal on solar absorptance values.

CONCLUSIONS

Materials exposed to the space environment demonstrated changes in light emission characteristics. The primary environmental factors responsible for the detected changes in fluorescence have been attributed to exposure to AO and UV radiation. The fluorescent properties of materials considered highly resistant to conventional optical degradation in the space environment, such as Z93 and YB-71 coatings, were altered in degree and nature as much as the other materials that are highly susceptible to conventional induced optical degradation. These effects on thermal control coatings and a variety of polymeric materials exposed on the LDEF were also found in similar materials exposed on much shorter duration space shuttle missions.

Fluorescence is a revealing indicator of induced material reactions as a result of space environmental exposure. Detected changes in fluorescence were revealed as quenching, stimulated emission, enhanced emission, and spectral shifts in emission. Comparison of effects found in materials exposed on the leading and trailing edges of the LDEF, on shorter duration space shuttle missions, and in laboratory testing indicate that thermal vacuum exposure, AO impingement, and UV radiation, acting individually or in combination, are the causative factors in altered fluorescence. The fluorescent properties of these materials are generally more sensitive to exposure than more conventional characterization techniques, whether revealed by visible observations under black light illumination or detailed spectrofluorometric analysis.

ACKNOWLEDGMENTS

The authors appreciate the helpful discussions with Dr. Charles Neely of Auburn University.

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Table 1. Long wave and short wave black light visual observations.

Sample	Exposure	Color	L.W. UV	S.W. UV	Comments
STS-5 Z306	Control	Black	Dk. Brown	Mustard Brown	
	Exposed		Tan/Green	Yellow/Green	
STS-8 Z306	Control		Chocolate	Pea Green	
	Exposed		Lt. Pea Green	Lt'er Pea Green	
AO171-IV-44 Z306	Control		Chocolate	Pea Green	
	Exposed		Lt. Chocolate/Yellow	Brown	
AOO34 Z306	Control		Dk. Chocolate/Black	Pea Green	
	01-14 Window		Tan/Green	Brown	
	01-44 Open		Tan/Greater Green	Pea Green	
STS-5 S13GLO	Control	White	Bright Yellow	Bright Yellow	
	Exposed		Yellow	Paler Yellow	
AO171-IV-13 S13GLO	Control		Yellow	Bright Yellow	
	Exposed		Burnt Sienna/Brown	Burnt Sienna	Browned in exposed area
AOO34 S13GLO	Control		Yellow	Yellow	
	02-21 Open		Orange/Yellow	Orange/Yellow	
	02-15 Closed		Yellow	Brt. Yellow	
	02-12 Window		Yellow	Yellow	
	01-25 Open		Lt. Purple/Yellow	Lt. Purple/Yellow	
	01-11 Closed		Yellow	Yellow	
	01-12 Window		Yellow	Yellow	

Table 2. SLM Aminco SPF-500C spectrofluorometer specifications.

-
- Source: High Intensity 250W Xenon Arc Lamp
 - Dual Monochromator, 200 to 900 nm
 - Excitation Monochromator: 1200 line/mm holographic grating, maximized to 300 nm
 - Emission Monochromator: Classically ruled 1200 line/mm grating, blazed at 500 nm
 - Focal Length: 250 mm
 - Aperture: $f/4$
 - Resolution: 0.1nm
 - Bandwidths: 0.1, 0.2, 0.5, 1, 2, 2.5, 4, 5, 7.5, 10, 20 nm
 - Fluorescence Channel PMT: Hamamatsu R928P extended red response side window tube
 - Reference Channel PMT: Hamamatsu R777 or equivalent side window PMT
-

Table 3. Selected fluorescence peaks.

Material	Peak (nm) \pm (nm)	Peak (nm) \pm (nm)	Peak (nm) \pm (nm)	Peak (nm) \pm (nm)
Z93	388 \pm 1		512 \pm 1	
S13G	385 \pm 2		515 \pm 2	
S13GLO - LE	384 \pm 2		512 \pm 2	
S13GLO - TE	382 \pm 1		526 \pm 3	
NS43C	384 \pm 2		516 \pm 2	
NS43C - AODTS Exposed	384 \pm 1		523 \pm 2	
A276	364 \pm 4	414 \pm 1	496 \pm 2	
A276 UV Only			560 \pm 2	
A276 AO + UV				
Z306 AOO34 Control	333 \pm 2	446 \pm 5		656 \pm 2
Z306 AOO34 UV Only			579 \pm 5	
Z306 AOO34 AO + UV	343 \pm 2	449 \pm 5		653 \pm 3
Z306 STS 5 Control	337 \pm 2	438 \pm 5	538 \pm 4	653 \pm 3
Z306 STS 5 Exposed	341 \pm 2		500 \pm 17	
Z306 STS 8 Control	333 \pm 2	446 \pm 5		656 \pm 2
Z306 STS 8 Exposed	336 \pm 2	469 \pm 5		
Z306 AO171 Control	333 \pm 2	446 \pm 5		654 \pm 3
Z306 AO171 Exposed	339 \pm 2	453 \pm 5		651 \pm 2
Z302 STS 5 Control	338 \pm 2	414 \pm 5		663 \pm 3
Z302 STS 5 Exposed	340 \pm 2 Shoulder	488 \pm 5		
Z302 STS 8 Control	339 \pm 1	447 \pm 4		662 \pm 2
Z302 STS8 Exposed	339 \pm 1	459 \pm 2		662 \pm 2
Z302 AO171 Unexp	337 \pm 2	457 \pm 2		656 \pm 2
Z302 AO171 Exp	340 \pm 2	495 \pm 10 (Wide)		647 \pm 5
Na Saly		419 \pm 1		
S383 EOIM-3 Control	338 \pm 2			663 \pm 2
S383 EOIM-3 Exp.60-38		468 \pm 5	588 \pm 2	
Viton EOIM-3 Control		470 \pm 15		
Viton EOIM-3 Exposed		455 \pm 2		
AO171 PEEK Control		466 \pm 5		
AO171 PEEK Exposed			512 \pm 1	
AO171 PMR Neat Con	396 \pm 3		515 \pm 2	
AO171 PMR Neat Exp			512 \pm 2	
AO171 RTV 511 Con	340 \pm 3		526 \pm 3	665 \pm 3
AO171 RTV511 Exp			548 \pm 3	

Table 4. AO exposure and wavelength shifts in exposed Z306™ fluorescence.

Spaceflight/Experiment	Peak λ (nm)*	AO Fluence (atoms/cm ²)
STS 5	516	9.99×10^{19}
STS 8	470	3.5×10^{20}
LDEF AO171, Row 8	452	6.93×10^{21}
LDEF AOO34, Row 9	439	8.72×10^{21}

*Estimated Peak Wavelength - Visible Emission

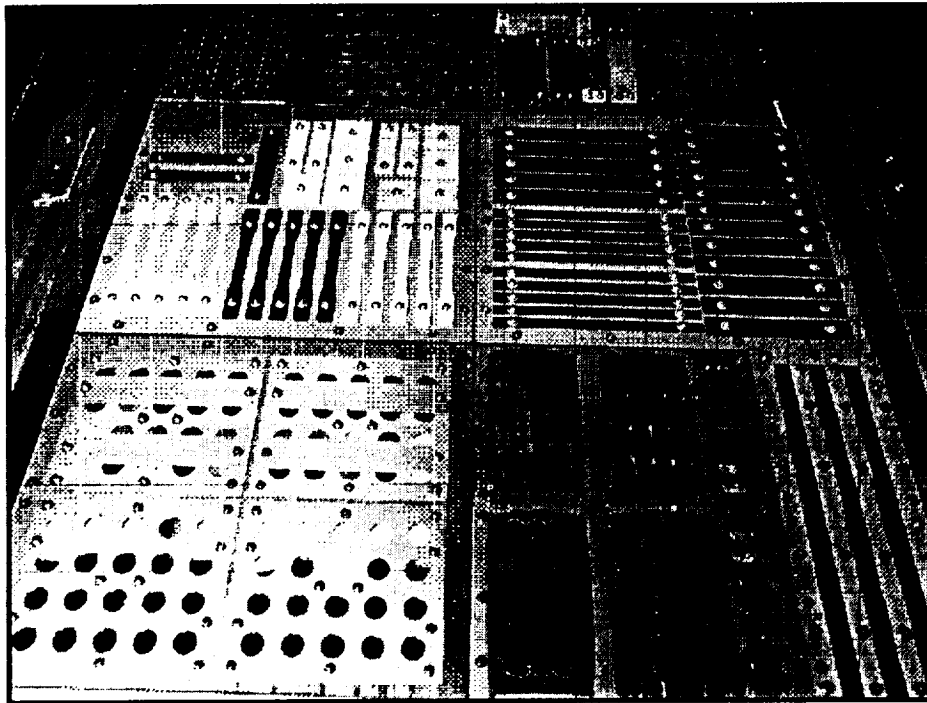


Figure 1. Solar array materials passive LDEF experiment AO171.



Figure 2. LDEF AO stimulated outgassing experiment AOO34.

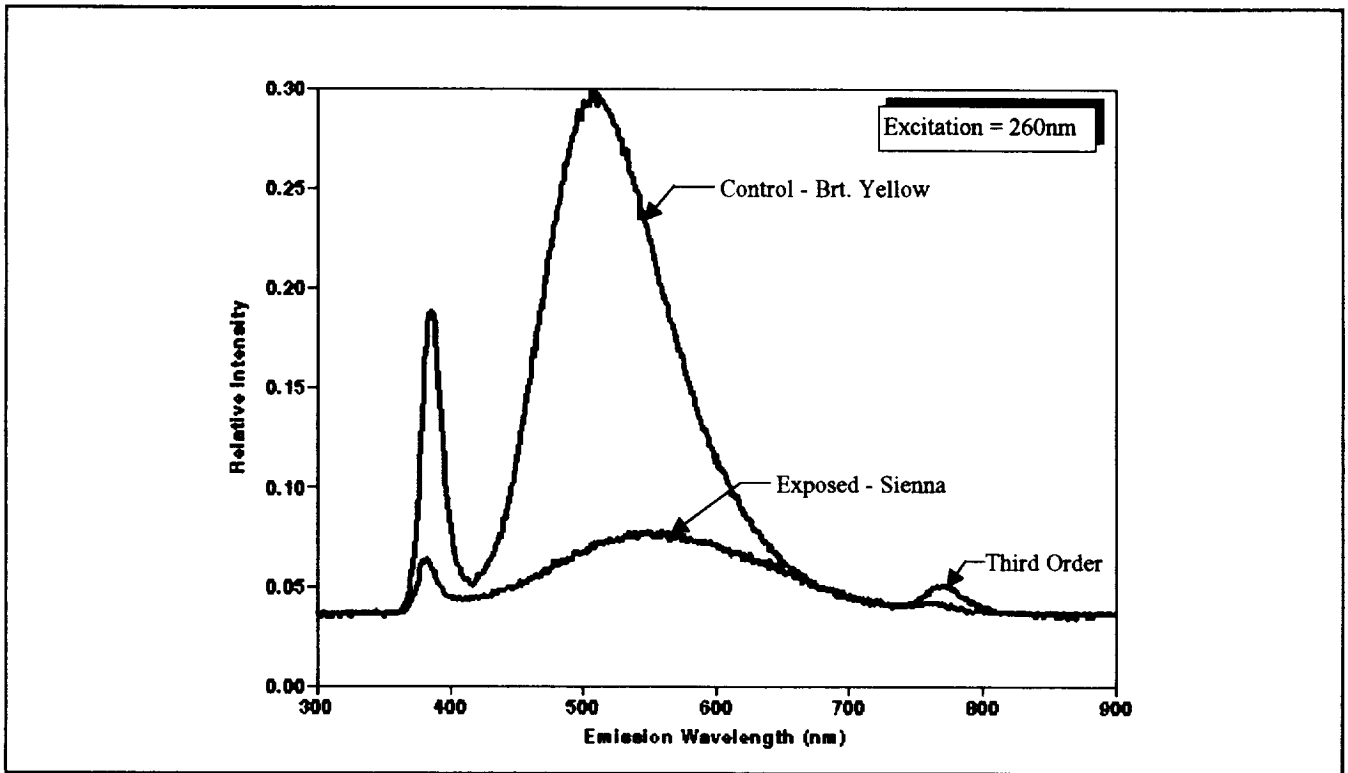


Figure 3. LDEF AO171 S13-GLO paint fluorescence curve.

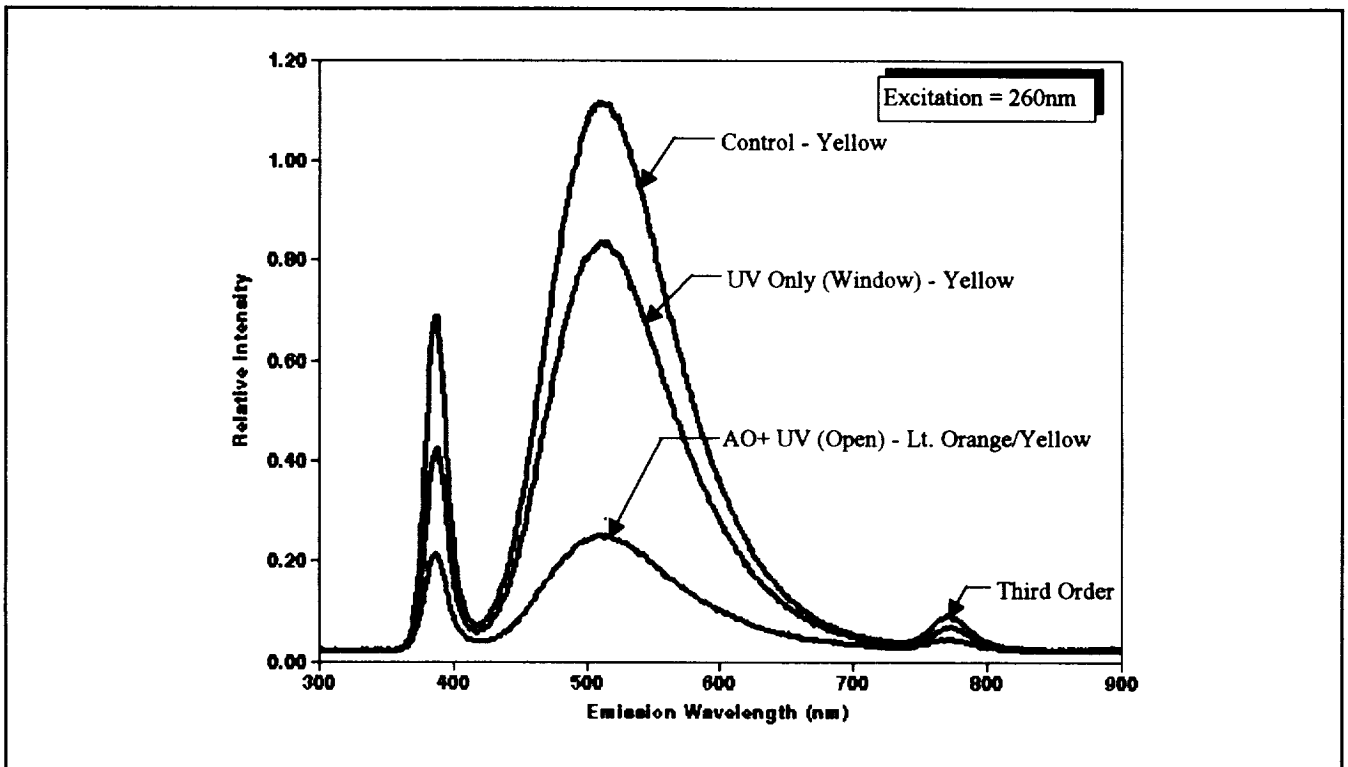


Figure 4. LDEF A0034 Z93 leading edge paints fluorescence curves.

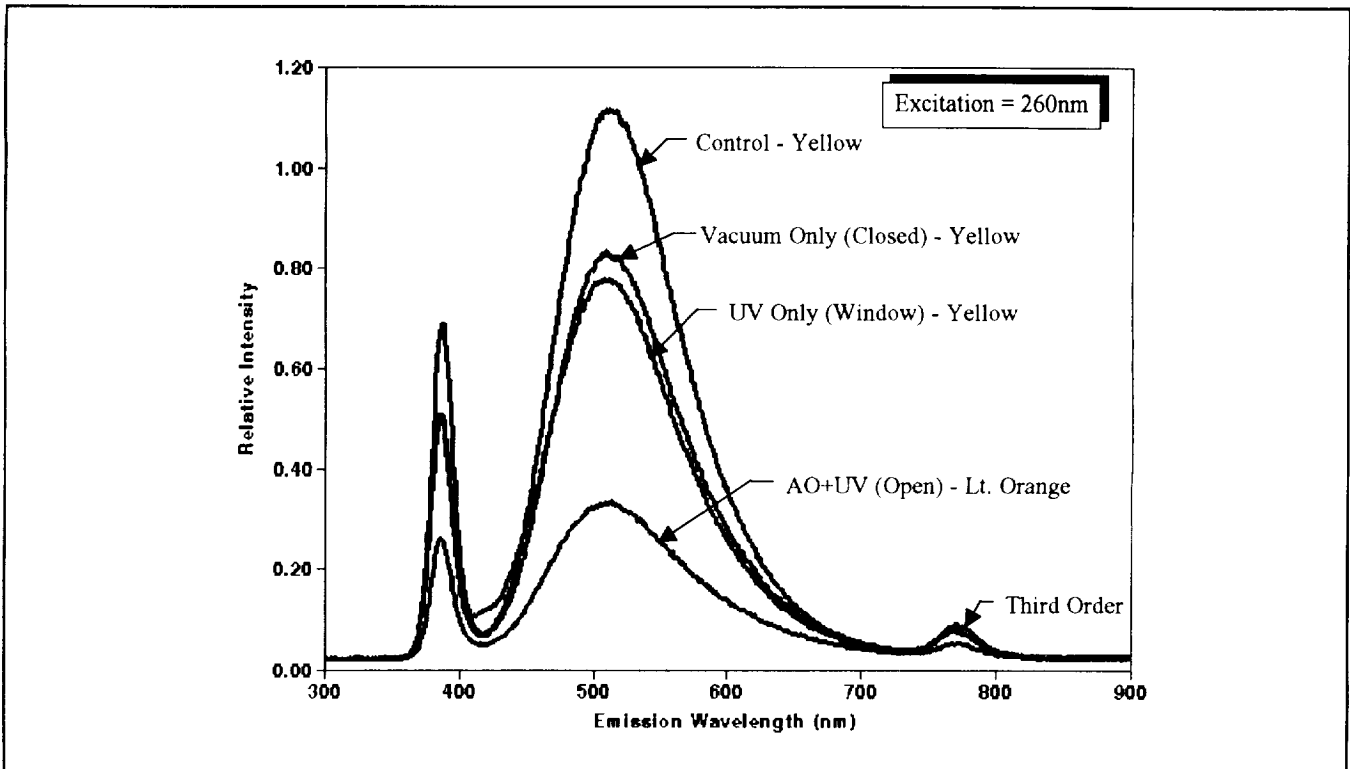


Figure 5. LDEF A0034 Z93 trailing edge paints fluorescence curves.

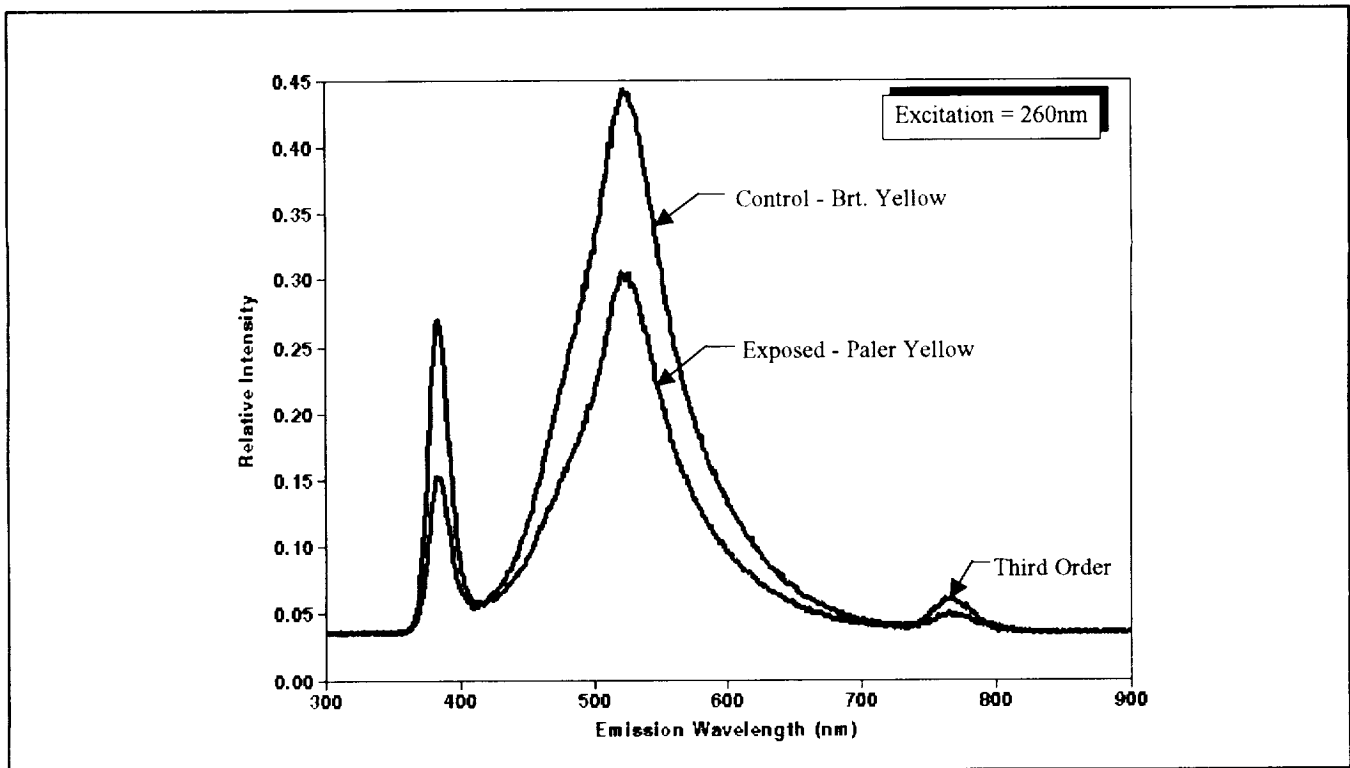


Figure 6. STS-5 S13-GLO paint fluorescence curve.

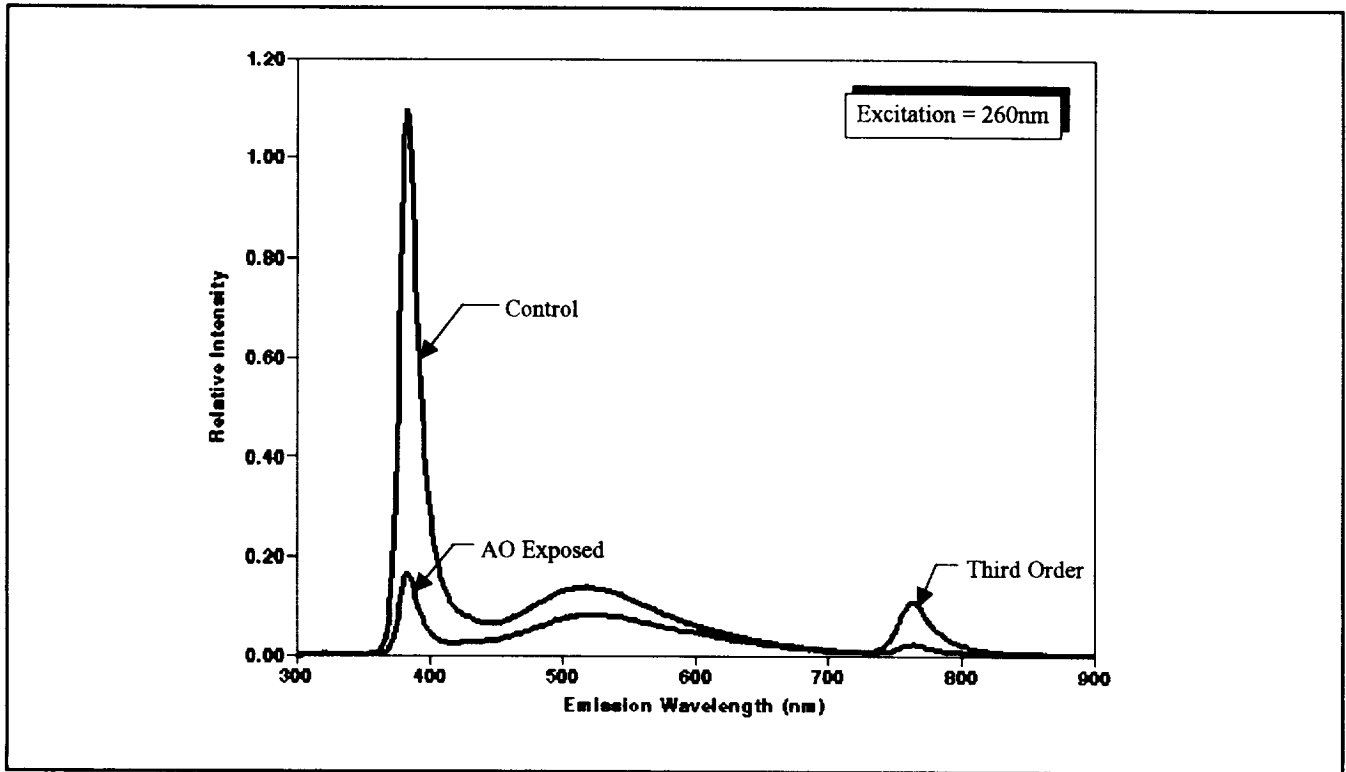


Figure 7. Fluorescence curve for NS43C paint exposed in the MSFC AODTS AO system.

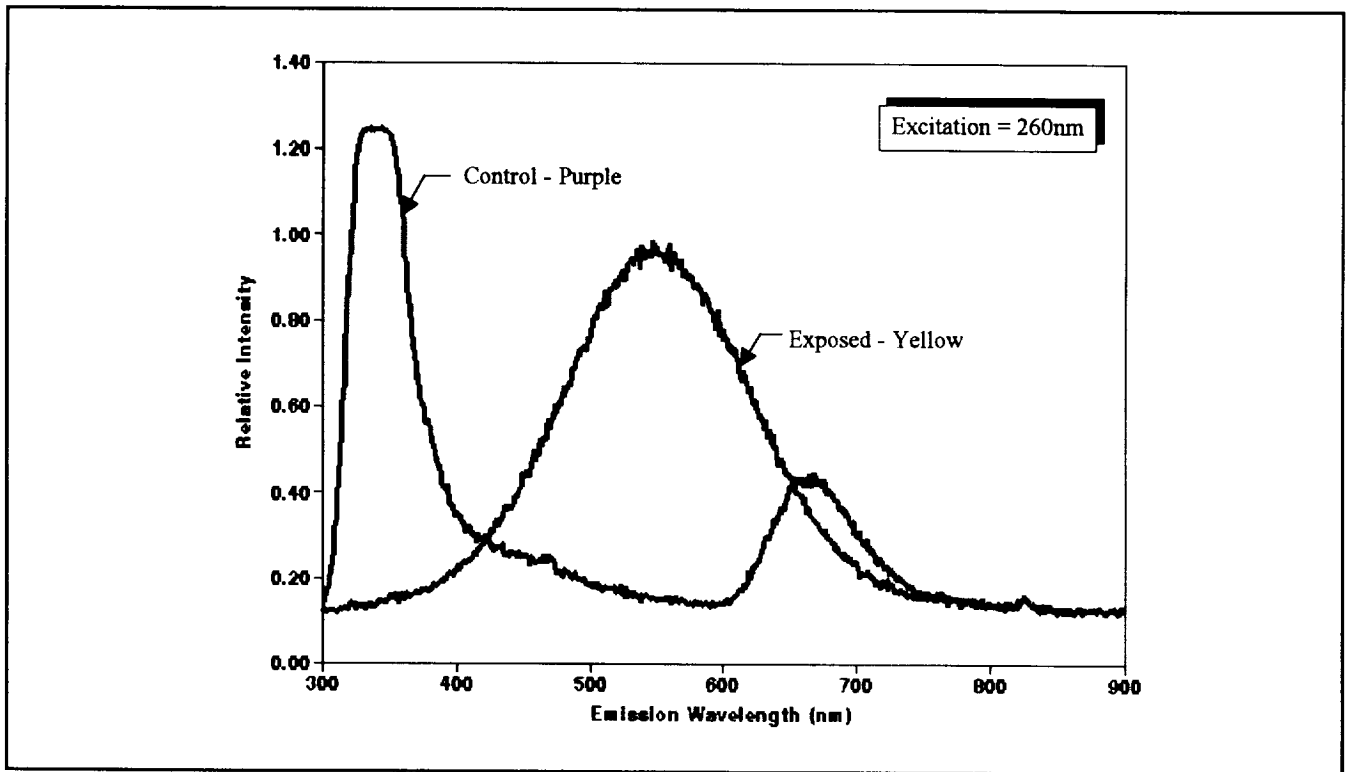


Figure 8. LDEF A0171 RTV 511 fluorescence curve.

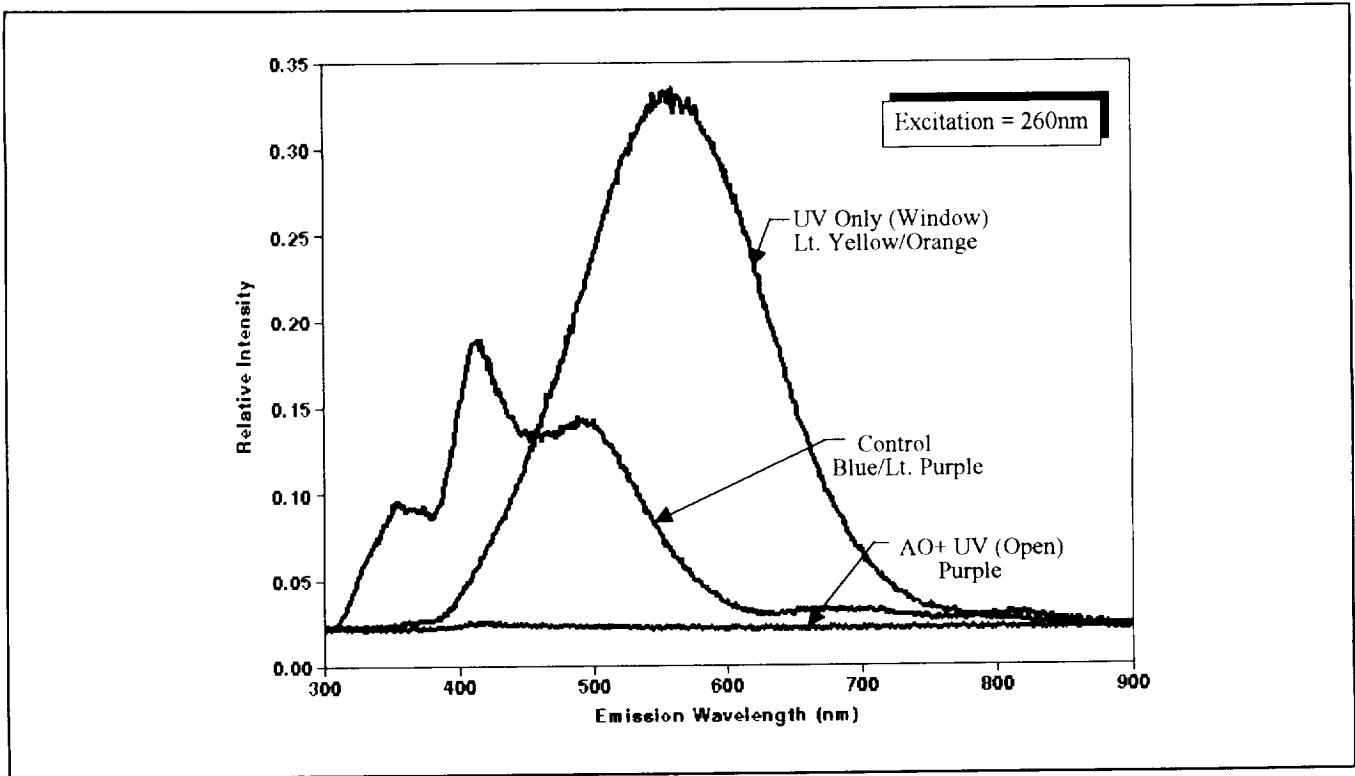


Figure 9. LDEF A0034 Chemglaze A276™ leading edge paint fluorescence curves.

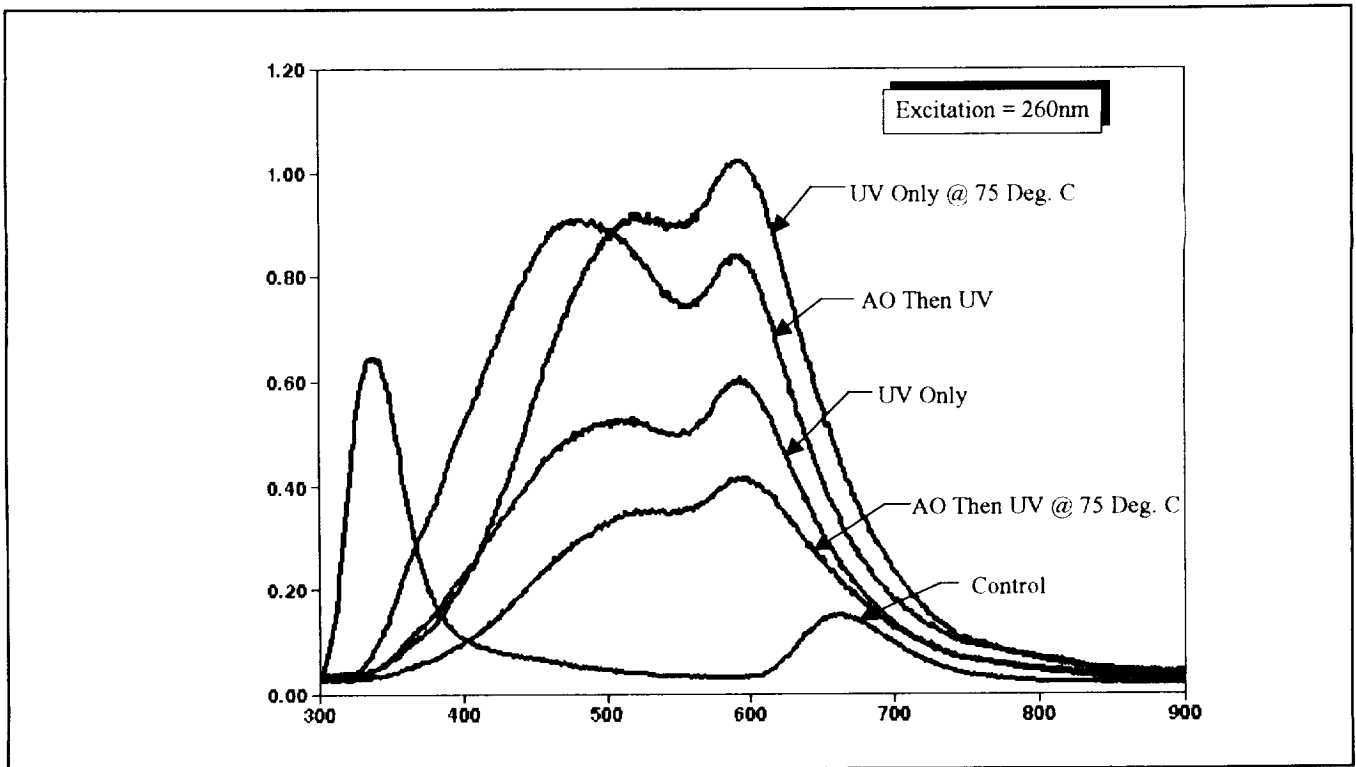


Figure 10. Fluorescence of laboratory AO and UV exposed S383 as a function of temperature.

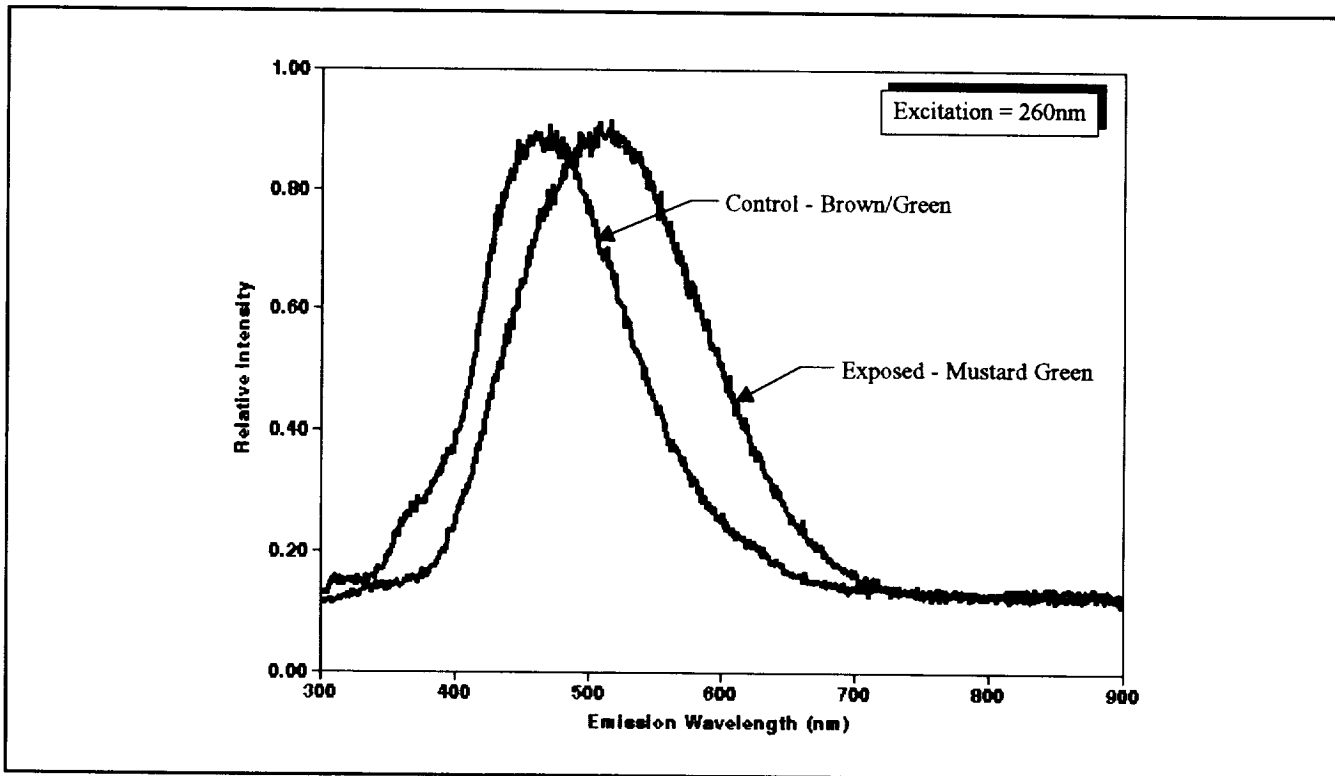


Figure 11. LDEF A0171 PEAK fluorescence curve.

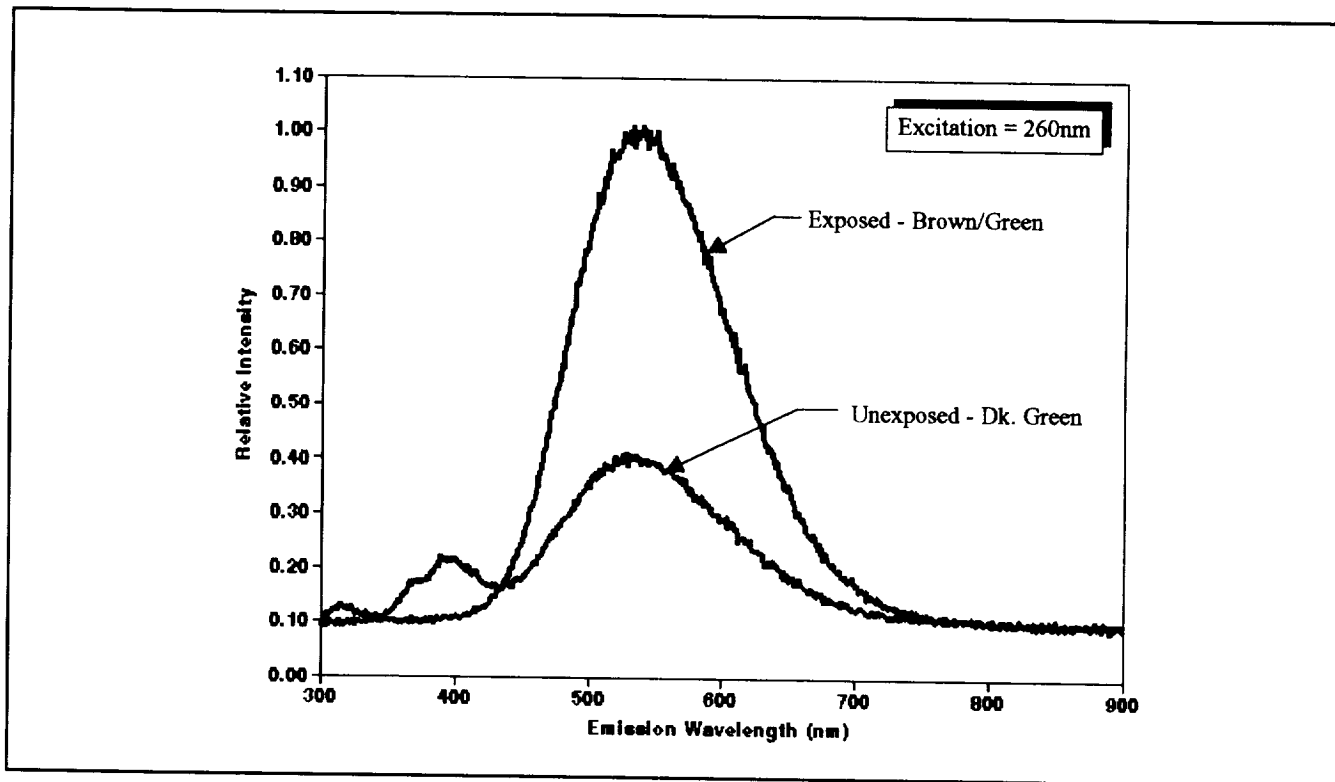


Figure 12. LDEF A0171 PMR neat resin fluorescence curve.

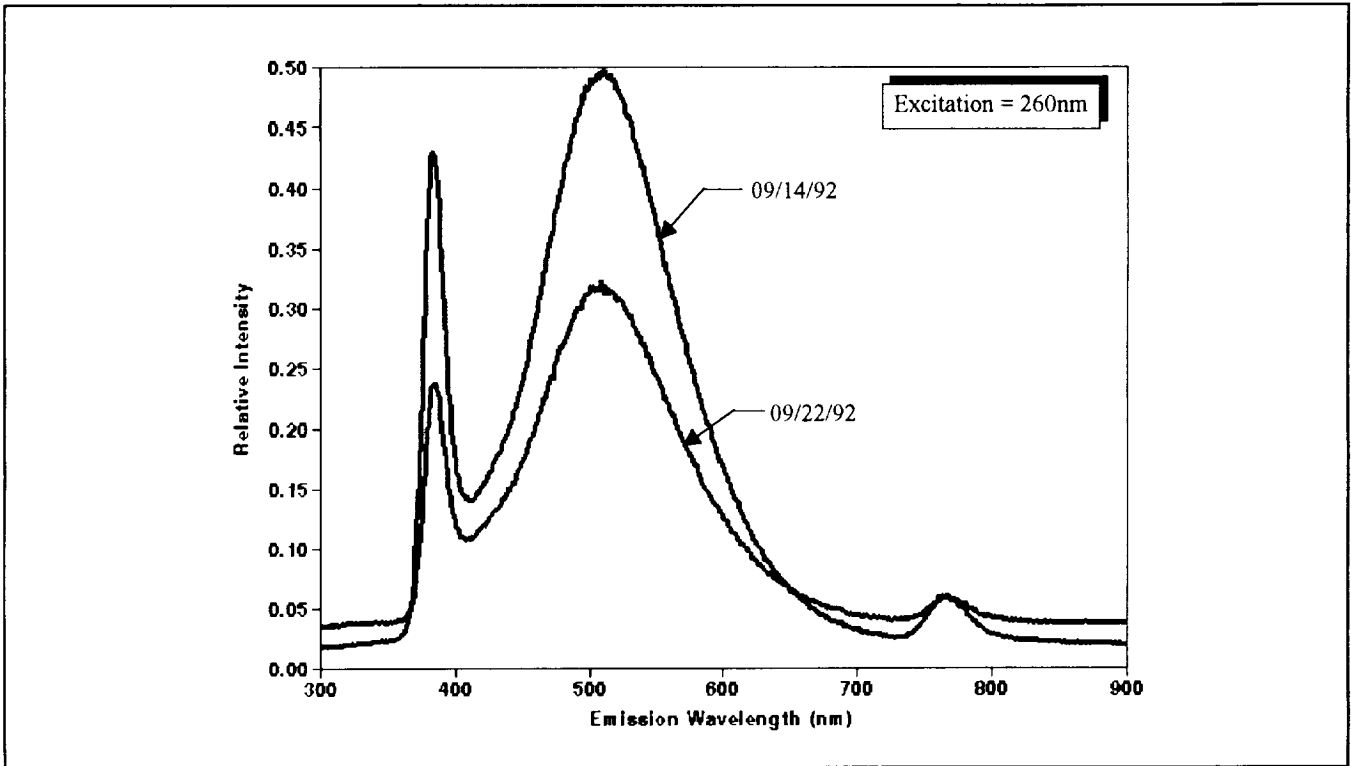


Figure 13. Fluorescence curves for A0034 S13-GLO measured on separate days in ambient laboratory conditions.

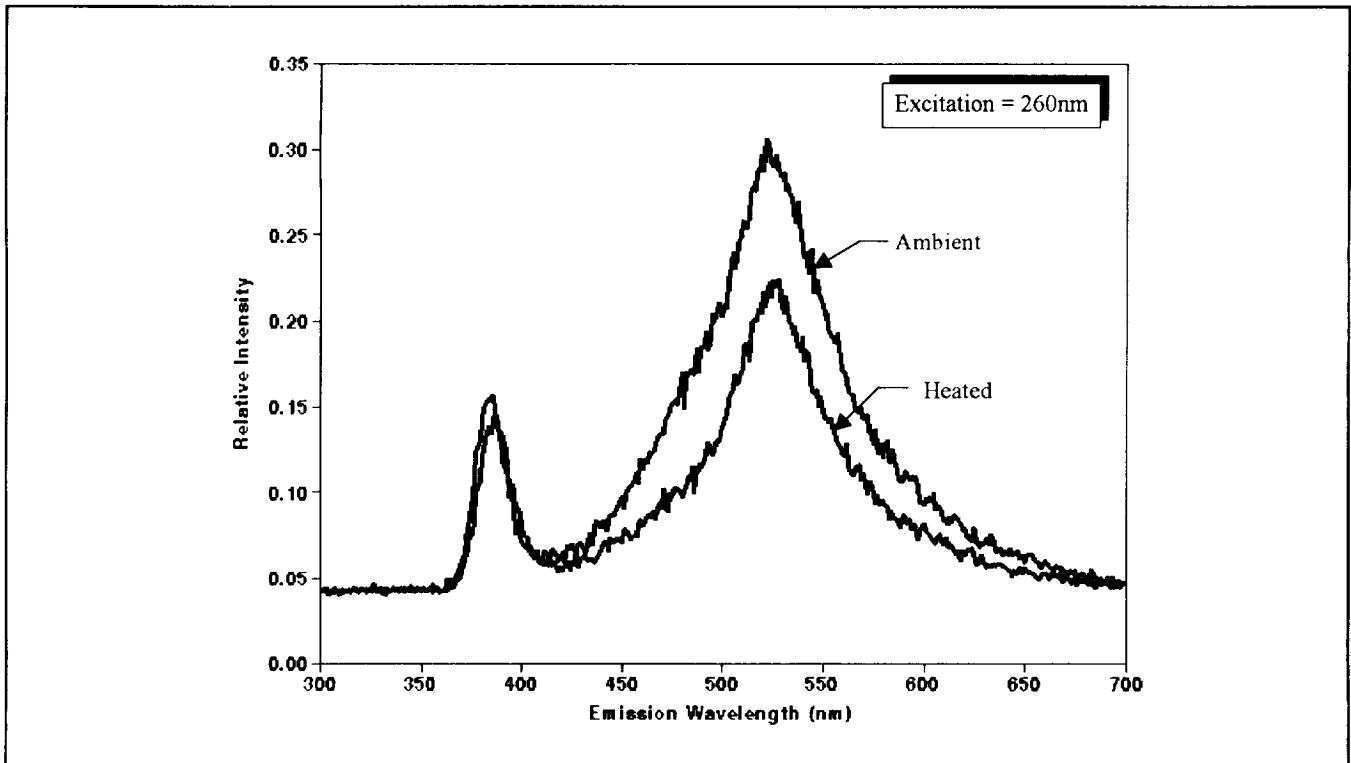


Figure 14. STS-5 exposed S13-GLO fluorescence curves as a function of temperature.

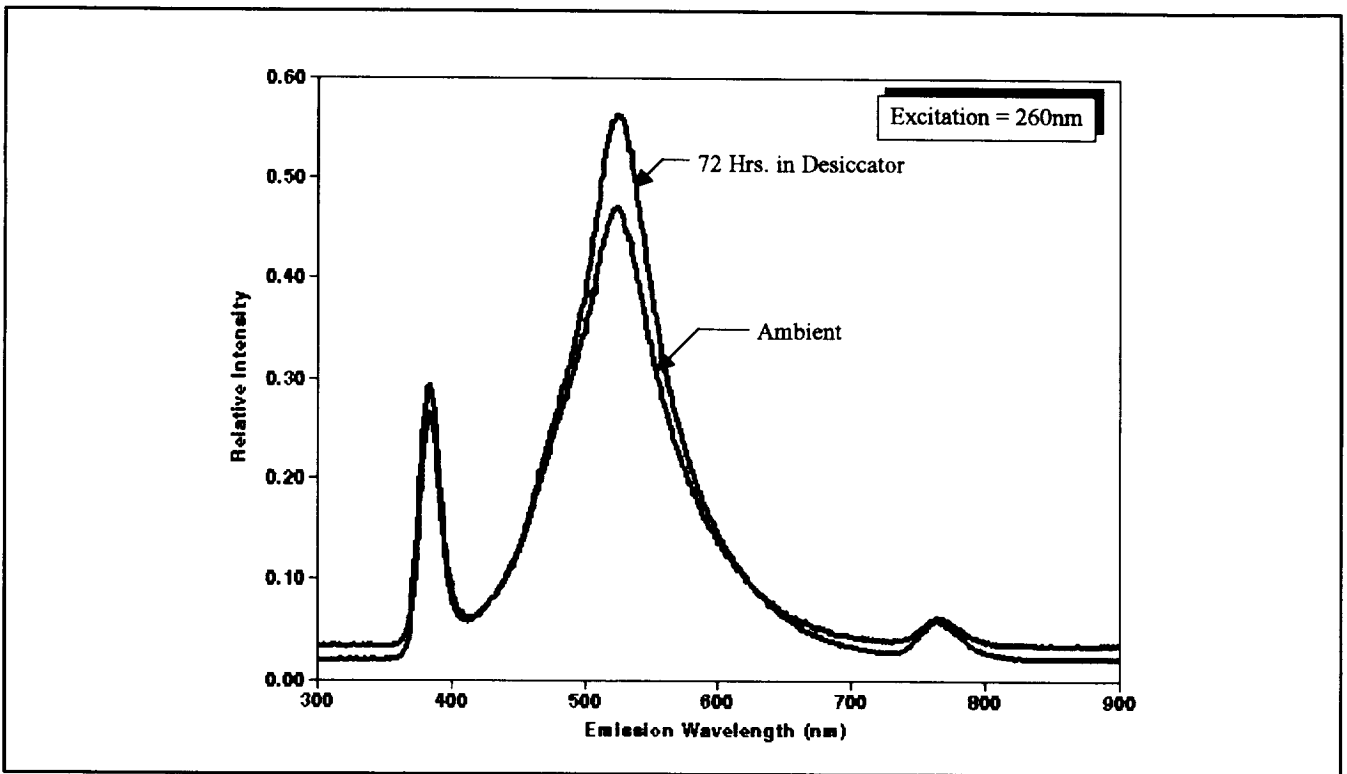


Figure 15. Humidity effects on the fluorescence of A0034 S13-GLO flight sample.

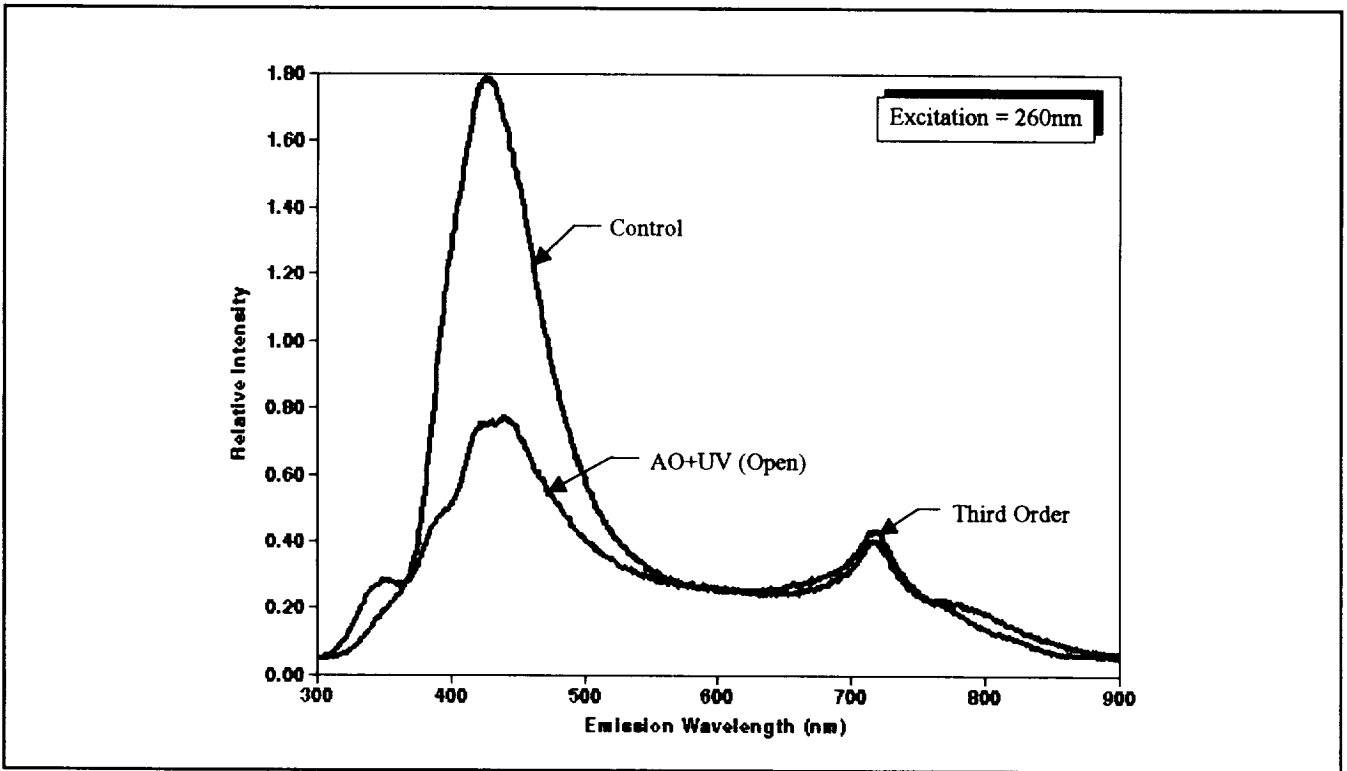


Figure 16. LDEF A0034 YB-71 leading edge paints fluorescence curves.

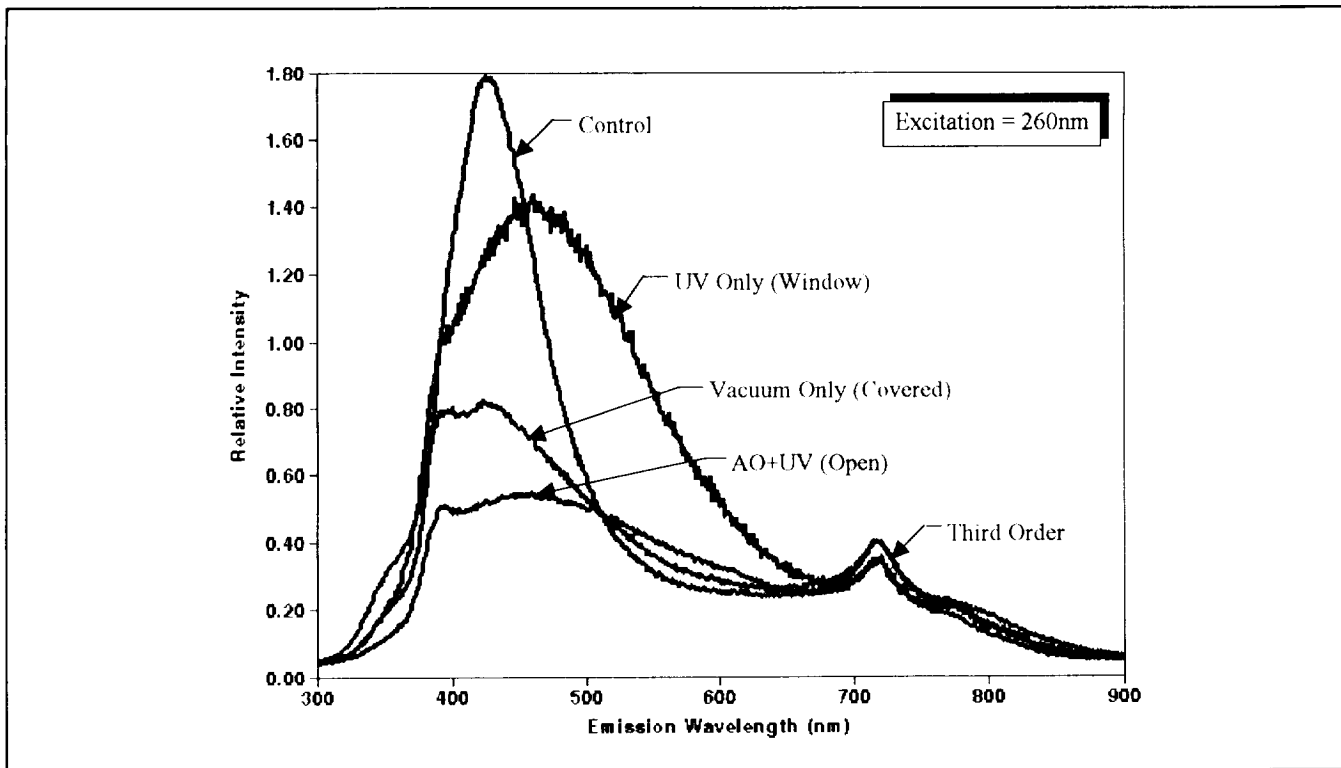


Figure 17. LDEF A0034 YB-71 trailing edge fluorescence curves.

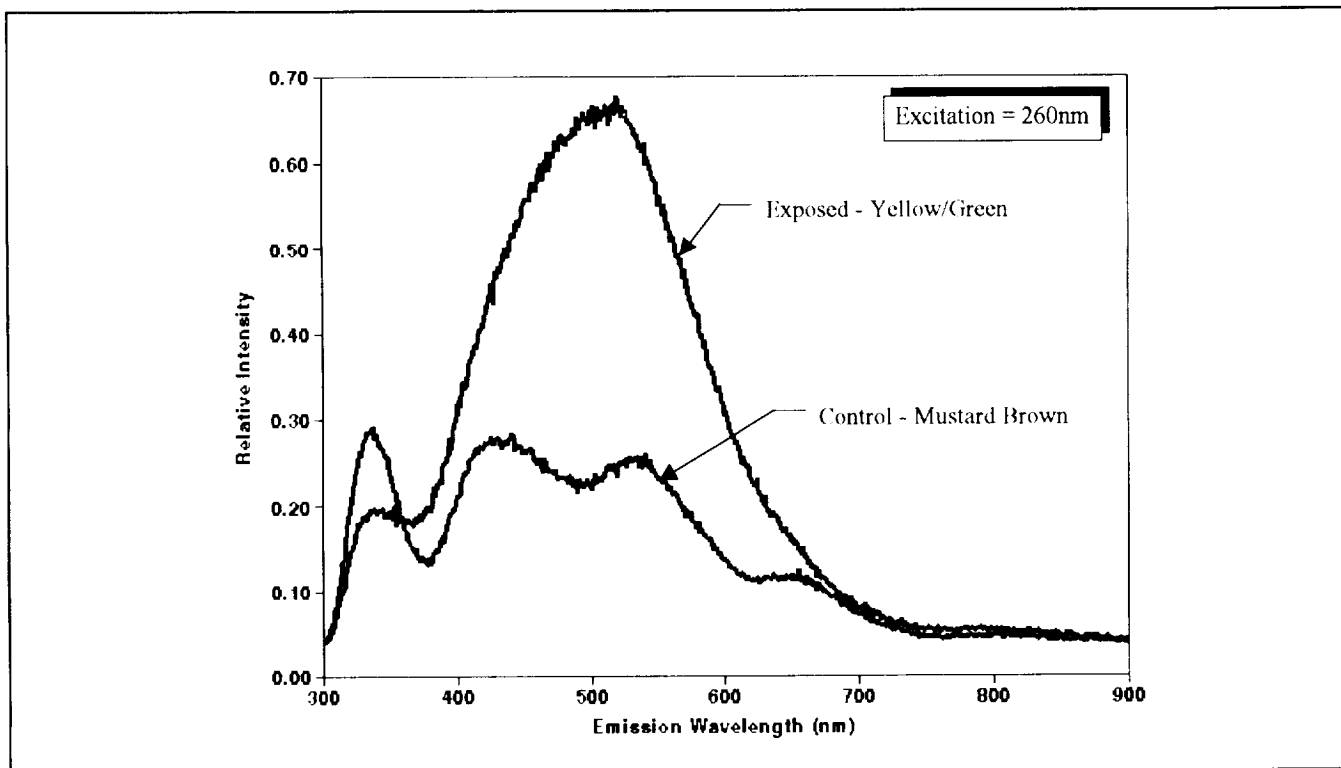


Figure 18. STS-5 Z306™ paints fluorescence curves.

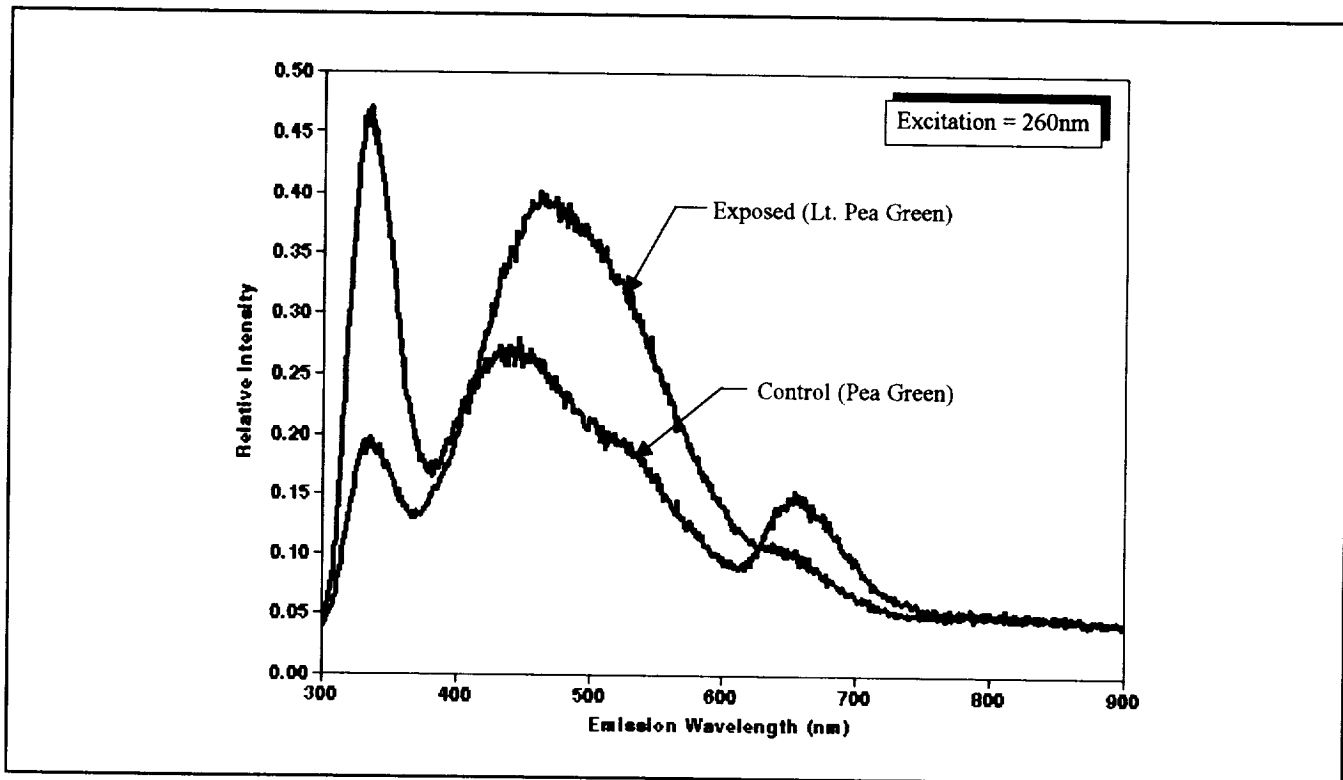


Figure 19. STS-8 Z306™ paints fluorescence curves.