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SOME LUMINESCENT PROPERTIES
of the
MIXED PHENYL- AND p-BIPHENYLYL- SUBSTITUTED SILANES
under
ULTRA-VIOLET, GAMMA AND BETA EXCITATION

THESIS

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Preface

It has been my intent in writing this paper to contribute to the vast amount of data and information currently available on scintillators. Because of the scope and time limitations of a master's thesis, I have had to leave many areas of interest for later study and investigate others less thoroughly than was desired. This study has shown that triphenyl-p-biphenylylsilane, diphenyl-di-p-biphenylylsilane, and phenyl-tri-p-biphenylylsilane, though less efficient than anthracene are nevertheless worthy of consideration as scintillators.

I am indebted to Dr. L. Spialter and the Chemistry Research Branch of the Aeronautical Research Laboratory for providing the silane compounds and a major portion of the equipment used in this study, and to W.L. Buck, Argonne National Laboratory, who determined the fluorescence decay constants of the silane compounds.

I wish to express appreciation to the Department of Physics and the School Shops of the Institute of Technology for the valuable assistance given during the course of this investigation. In particular, I would like to thank my advisors, Major Arthur Vetter and Dr. George John, for their assistance, encouragement, and understanding throughout this study.

Carl G. Weis

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Abstract

Some of the luminescent properties of triphenyl-p-biphenylylsilane (silane I), biphenyl-di-p-biphenylylsilane (silane II) and phenyl-tri-p-biphenylylsilane (silane III) have been investigated. These scintillators are all low-melting point, transparent, glassy solids which are easy to mold in various sizes and shapes. In comparing these phosphors with anthracene the following results were obtained:

a) Silane I is 8% less effective than anthracene in its gamma detecting ability.

b) The pulse height of the silanes is about 40% - 45% that of anthracene.

c) Each of the silanes is about 70% as efficient a beta detector as is anthracene.

Fluorescence spectra for each of the silanes under ultra-violet and beta stimulation are reported. Peak emission wave-lengths are 400 mμ, 400 mμ and 430 mμ for Silane I, II, and III respectively.

Fluorescence decay constants of 2.53 - 2.54, 2.62 - 2.63 and 3.29 - 3.31 nanoseconds were found for Silane I, II, and III.

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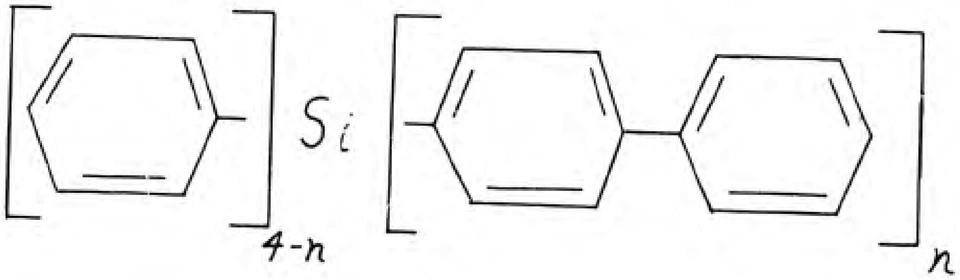
I. Introduction

Previous studies (Ref 9 and 12) of the mixed phenyl and p-biphenylyl substituted silanes have demonstrated that these organo-silicon compounds fluoresce when exposed to ionizing radiation. Three of these compounds not only have low-melting points, but form transparent, glassy solids when cooled from the molten state. These properties indicate that the silanes have possibilities for use as radiation detectors, offering the advantage of ease of fabrication.

The purpose of this report is to present results of a study of some of the luminescent properties of these compounds. These properties were examined for the purpose of adding to the information necessary to establish the full capabilities of these compounds as radiation detectors, and to shed some light on the factors which affect the luminescence.

Results of Previous Researchers

In 1955, Spialter published a report (Ref 26) outlining the synthesis of a whole family of tetraarylmonosilanes where the aryl substituents were phenyl and p-biphenylyl. These compounds can be expressed by the general formula



Three of the above compounds, triphenyl-p-biphenylylsilane, biphenyl-di-p-biphenylylsilane and phenyl-tri-p-biphenylylsilane, are low-melting point glassy solids which exhibit luminescent properties while in the glassy state.

Downs and Smith (Ref 12:94-96) used Cs^{137} to investigate the scintillation efficiency and properties of a mixture of two silanes, namely triphenyl-p-biphenylylsilane and phenyl-tri-p-biphenylylsilane. Their study revealed these glassy silanes have excellent properties as scintillators.

Cooper (Ref 9) investigated the scintillation properties of triphenyl-p-biphenylylsilane using Co^{60} and Cs^{137} as sources. He concluded that the pulse height of the silane was approximately 60% that of anthracene, that the pulse height in the silane was reduced 12% for each centimeter increase in phosphor thickness, that the detection ability of the silane for gammas was slightly superior to that of anthracene and that the quantum efficiency of light conversion for the silane is equal to that of anthracene.

Kemler (Ref 19) tried unsuccessfully to load a triphenyl-p-biphenylylsilane glass with boron to develop a neutron detector. He found that the resulting discoloration, coupled with gamma interference, prohibited development of a satisfactory scintillator for neutron detection.

Scope of Study

This study considered only the three members of the silane family which combine fluorescence and glass-forming properties, namely triphenyl-p-biphenylylsilane, biphenyl-di-p-biphenylylsilane and phenyl-tri-p-biphenylylsilane. These three compounds were investigated in light of the information needed to establish the full capabilities of these polyaryl silanes as radiation detectors. Areas considered were:

1. Purification of the commercial compounds to eliminate effects of impurities, and development of molding techniques.
2. Comparison of the luminescence spectrum with multiplier phototube response.
3. Fluorescent decay times.
4. Efficiency for conversion of deposited energy to fluorescent light.

A fifth important area, an investigation of self-absorption of the silanes for their own fluorescent light, was not considered because of lack of time.

A purification process for the silanes was developed as a portion of this study. It is felt that this process is superior to that used by previous experimenters (Ref 9 and 12) since it is much simpler and requires less time.

The luminescence spectrum of all three silanes was obtained, both under ultra-violet and beta excitation. These spectra were compared to the response curves for commonly used multiplier phototubes.

Since a rapid decay constant is an important prerequisite for any scintillation detector, decay constants for each of the silanes were determined. This determination was made for electron excitation since this source of ionizing radiation is more easily controlled than others.

To determine the efficiency for conversion of deposited energy to light, pulse heights and scintillation efficiencies for each of the silanes were compared to an anthracene standard. Relative scintillation efficiencies for betas were determined for all three silanes, but relative gamma efficiency was determined only for triphenyl-p-biphenylylsilane. Shortage of the other two compounds prevented molding of phosphors large enough for gamma efficiency evaluation.

Format of Report

The first portion of this report consists of a brief coverage of the theory involved. The second section deals with experimental methods and equipment used while the third presents results and conclusions. A fourth section proposes areas for further study. Fluorescent spectra for the silanes and for anthracene are contained in the appendix.

For simplification in writing, the silane compounds investigated will be referred to as Silane I, II, and III, the numbers corresponding to the number of p-biphenylyl groups contained in the molecule.

II. Theoretical Aspects of Organic Scintillators

Organic scintillators are commonly used for detection and measurement of ionizing radiation. They ordinarily take the form of pure crystals, mixed crystals, solid or liquid solutions. Solid solutions for the purpose of this discussion are defined as the addition of a fluorescent material such as tetraphenylbutadiene to polystyrene or other plastic materials to form a phosphor.

The silane glasses, being in a form different from those specified above, may possibly differ in behavior from that of compounds falling within one of the general categories. The formulation of a theory of the scintillation process in the silane glasses, with subsequent substantiation, is an extremely valuable and necessary project for the development of the silanes as scintillators. The following discussion of the theory of scintillation in organic materials is intended to indicate the possible relation of the silanes to other organic scintillators.

Very thorough explanations of the scintillation process in organic compounds can be found in publications by such authorities as Birks (Ref 3), Curran (Ref 10), Leverenz (Ref 21), Brooks (Ref 6), Sangster (Ref 25), Swank (Ref 28, 29, 30), Buck (Ref 8) and others. The theory contained in this report is limited to the more general aspects of the scintillation process as applied to pure organic scintillators.

The theory of scintillation in organic materials is virtually a theory in the processes of energy transfer. These processes may be divided into two categories: a) transfer of energy from ionizing radiation or particles to the scintillator molecules and b) subsequent intermolecular and intramolecular transfer with the production of fluorescense photons. The present concept of the scintillation process in organic scintillators is that the scintillation originates with electronic transitions in the molecules, that is, this process is an inherent molecular property (Ref 6:253).

Initial Excitation of the Scintillator

Very little is known about the mechanisms of energy transfer from an incident ionizing particle to the organic molecule. Buck, in a recent article (Ref 8) states that he knows of no experimental determination of the fraction of energy expended by a charged particle in optical excitation of an organic solid. His article outlines the theoretical determination of this fraction using electromagnetic theory. It is further indicated that slow-decaying components of the emission may arise from molecules left in excited states as a result of ion recombination.

The Scintillation Process

As mentioned above, it is well established that light emission in organic scintillators is associated with electronic transitions in molecules. A convenient approach to a description of the process must then establish:

a) the electronic levels in organic molecules which give rise to luminescent properties.

b) the processes of luminescence in an isolated molecule

c) changes in the characteristics of the isolated molecule when placed among others.

These three properties have been extensively studied under the simplifying conditions of photo-luminescence, that is by exciting the molecule with ultra-violet light. Investigations have shown the fluorescence spectra from organic scintillators to be independent of the type of excitation (Ref 8:51). However, decay times have been found to vary with the type of exciting particle (Ref 24).

Electronic Levels in Organic Molecules.

Electronic levels of an organic molecule are closely related to structure since both derive from the electronic properties of the constituent atoms, particularly carbon. Carbon has four valence electrons which normally assume one of three configurations. These configurations may be designated as tetrahedral, trigonal and digonal corresponding to valences of four, three and two respectively. In the

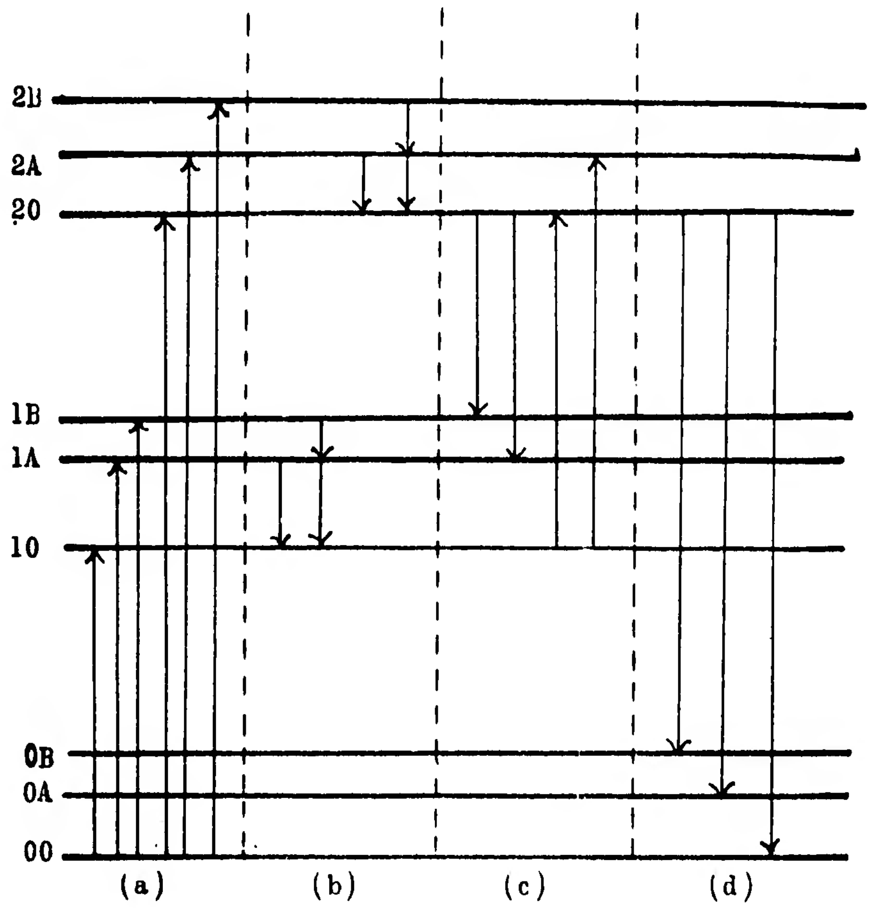


Figure 1

Energy Levels in an Organic Molecule Showing the Luminescence Process.

a) Excitation
b) Internal degradation
c) Singlet-Triplet Energy Interchange
d) Fluorescense

trigonal configuration, one p-orbital (that is a p electron in the carbon-atom's L shell) is left unchanged. In the digonal configuration two p-orbitals are left unchanged. In either case, two atoms may achieve maximum interaction by rotating their two nodal planes into coplanarity and combine into a π orbital, denoted π because it is mirror symetric with respect to the common nodal plane (Ref 6:255). The π orbital corresponds to the extra link between two carbon atoms in the structural formula. Thus acetylene ($HC \equiv CH$) has two π orbitals and ethylene ($H_2C = CH_2$) has one.

The π electron system like any other system has discrete energy states of its own. These states lie between the lowest level corresponding to the p orbitals interacting in phase to the highest π orbital corresponding to each p orbital being 180° out of phase with its nearest neighbors. Excited states result in the transition of an electron from the highest filled π orbital into one of the empty levels above it. The spins of the electrons in the excited level may be parallel forming a singlet, or anti-parallel forming a triplet of lower energy than the singlet.

Molecular Luminescence

The luminescence processes in the molecule may be described in terms of a simplified energy level diagram composed of singlet and triplet electronic levels and vibrational levels. Figure 1 on the opposing page is such a representation. The electronic levels are denoted as 0, 1, and 2 and the vibrational levels are indicated as discrete sub-levels (suffices 0, A, B). Energy level 1 represents a triplet while level 2 corresponds to the first excited singlet.

Different stages in the luminescence process of a single molecule are indicated by sections (a), (b), (c), and (d) of Figure 1. The molecule will normally occupy one of the vibrational states associated with the lowest electronic state at normal temperature. Excitation is represented by a transition of the type shown in (a), depending on the amount of energy absorbed. Vibrational and electronic states are simultaneously excited.

Figure 1 (a) represents internal degradation or the dissipation of energy as heat through collisions with surrounding molecules or by excitation of lattice vibrations. A transition from a singlet state to a triplet level of lower energy may occur as shown in figure 1 (c). As indicated, this process is reversible although the probability is low except where the electron passes close to the nucleus thus inducing spin reversal (Ref 9:96). The energy increase in this case must of course come from conversion of vibrational energy. Figure 1 (d) shows the emission of the fluorescence photon. In every case observed, luminescence occurs only from the first excited singlet level to the ground level (Ref 6:259).

A process competing with fluorescent emission is internal quenching, that is the conversion of electronic energy followed by degradation. This gives rise to a quantum efficiency (ratio of the number emitted to exciting photons) of less than unity. The process of excitation and internal degradation occurs in approximately 10^{-11} seconds while fluorescent emission occurs after a mean lifetime on the order of 10^{-9} seconds or longer.

Modifications for Bulk Materials. The theory above is, of course idealized and must be modified to apply to the practical case. The effects of environment, resonance transfer and reabsorption must be carefully considered.

In the idealized case, the environment of the molecule was neglected except in the case of interactions between electrons belonging to the atoms of the constituent parts of the molecule. In the practical case, one must consider, also, the intermolecular interactions. Conditions arising from collision of two molecules are such as to increase the probability of internal quenching, thus reducing both the quantum efficiency and mean lifetime of the molecule (Ref 6:260).

One excited molecule can interact with a neighbor, transferring excitation energy to the neighbor atom in what is termed a resonance transfer. Due to this transfer, the luminescence process can involve several molecules rather than one as in the case we considered above.

Experimental evidence indicates that this process may occur only between the first excited levels of neighboring atoms, both of which are in the lowest vibrational state. One important condition for resonance transfer is thus seen to be that the donor molecule must possess the same or more excitation energy than the acceptor. However, providing the molecules remain chemically unchanged, the characteristics of such a system are unaffected by the non-radiative transfer process.

The term energy transfer usually includes both radiative and non-radiative transfer. The former term is familiar and needs no clarification, whereas the latter perhaps needs further explanation as to its nature. When the π electrons of the two molecules interact strongly (that is their wave functions overlap) they reach a state where the electronic levels of the two molecules can no longer be resolved. A system of strongly interacting molecules becomes characterized by a banded energy level instead of several discrete molecular levels. In simple terminology, the electronic excitation energy is non-localized and belongs to the system rather than to the individual molecules. Thus the term intermolecular transfer loses its meaning. However energy transfer between different positions of the system has meaning and these positions are indicated by the molecule. Transfer can then result within the system through excitons. The exciton results when the excitation energy is insufficient to raise an electron from the highest broadened filled band to an empty one. An exciton is defined as an electron and its positive hole bound together in a series of states by one another's fields. Excitons may move through the system, transferring energy, but not charge.

Reabsorption of fluorescent photons is another important effect to be considered in a practical scintillator. This can occur in a fluorescent material whose emission and absorption spectra overlap. Where overlap is large, a considerable portion of the molecular fluorescence spectrum is removed. Thus overlap materially alters the emission spectrum. In the scintillator, the process of emission and absorption may occur many times until only the radiation to which the medium is transparent escapes and is detected by the photomultiplier tube. As a result of overlap, the quantum efficiency of a crystal is less than that of the molecule, and the mean lifetime of the crystal fluorescence emission is longer than that of the molecular emission (Ref 6:262).

Desirable Properties of a Scintillator

From the foregoing discussion and a knowledge of the operation of a scintillation detection system, the following properties are seen to be desirable in a scintillating medium:

1. Immunity to chemical or structural change resulting from ionizing radiation.
2. Short luminescent decay times to permit fast counting rates.
3. A high quantum efficiency and conversion of deposited energy, that is to say, a minimum of such processes as thermal degradation which compete with the luminescence process.
4. Material is transparent to the light it emits, that is negligible overlap of the absorption and fluorescence spectra.
5. Emitted light is in the wave length range in which available multiplier phototubes operate.

The above properties will be considered in the evaluation of the silanes as scintillation detectors.

III. Experimentation

Purification of Materials

The silane I used in the experiment was furnished in the purified form, having been previously purified by Cooper (Ref 9:12-13) for use in his work. However, silane II and III were received in the relatively impure commercial grade and were carefully purified to minimize the effects of trace impurities on the fluorescent properties of the material. The presence of impurities in silane II and III was obvious, since glasses formed from the commercial grade had a yellowish color.

Purification of Silane II. The purification process for silane II was basically that of Downs and Smith (Ref 12:94). However, pure benzene rather than a benzene and n-heptane mixture was used as a solvent. The process differs from that of Cooper (Ref 9) in both the solvent used and in the sequence of steps. The purification of the silane II was accomplished by:

1) Dissolving a 40 gram sample of the crude silane II in excess benzene,
2) passing the solution through an adsorption column packed with florisil sand in a solution of benzene, and thoroughly rinsing with benzene,
3) fractionally distilling the solvent from the silane solution by boiling gently until

approximately one-half liter of solution remained,

4) adding hot ethanol to the hot silane solution until the solution became cloudy (because of crystal formation) and allowing the solution to cool to room temperature,

5) decanting the mother liquor from the crystalline product, redissolving the crystals and repeating step four for a total of four recrystallizations with no further purification observably required,

6) air drying the powdered pure silane II.

<u>Purification of Silane III</u>. The purification process used for silane III was suggested by Spialter (Ref 27). It was found to be simpler than that used in purifying silane II, and may be used for purification of the family of silanes mentioned in section I of this report. The procedure consists of four steps as follows:

1) Dissolving the impure silane III in a hot 50-50 mixture by volume of benzene and methyl cyclohexane, allowing to cool, separating the resulting crystals from the mother liquor by vacuum filtration,

2) repeating step 1 with more benzene-methyl cyclohexane solution and washing the crystals with cold methyl cyclohexane,

3) dissolving the crystals from step 2 in hot methyl cyclohexane, allowing to cool slowly to room

temperature and separating the crystals from the liquor by vacuum filtration with no further purification observably required.

4) Air drying the pure powdered silane III.

<u>Physical Properties of the Silanes</u>. Some of the physical properties of the silanes as given by Spialter, et al. (Ref 26:6227-6229) are shown in Table I.

Table I

Properties of Silane I, II, III

Material	Melting Point	Boiling Point
Silane I or Triphenyl-p-biphenylylsilane	159° C	512° C
Silane II or Diphenyl-di-p-biphenylylsilane	170° C	570° C
Silane III or Phenyl-tri-p-biphenylylsilane	155 - 174° C	580° C

(From Ref 26:6228)

The melting points of the silanes purified for use in the experiment were as follow: Silane I - 156-158° C; Silane II - 169-171° C; Silane III - 173-175° C. After purification, all three silanes had a brilliant white appearance and were considered sufficiently pure for the molding of phosphors for use in the balance of the experiment.

Preparation of Phosphors

Two sizes of phosphors were prepared. Three large samples (1 in. dia. by 1/4 in. thick) of silane I were prepared for use in comparing the relative scintillation efficiency of silane I to that of an anthracene standard of similar size available in the laboratory. Smaller samples (approximately $\frac{25}{64}$ in. square by $\frac{7}{16}$ in. high) of all three silanes were prepared for use in obtaining the fluorescence spectrum. Subsequently, the smaller samples were compared to an anthracene standard of approximately the same size to determine the relative scintillation efficiency. Large samples of silane II and III were not prepared since these compounds were in short supply.

All samples were prepared by melting a portion of purified silane in a pyrex tube fitted with a glass handle. Melting was done in a regulated hot air furnace at approximately 200° C. An attempt was made to melt the silanes by applying a torch to the pyrex tube. However, this method resulted in yellowing of the compound except in the case of silane I. The torch method was attempted since gas evolution was noticed and the presence of solvent of crystallization was suspected. It had been planned to rid the molten compound of solvents by melting under vacuum, using the torch as a heat source. Since the available hot air furnace was not fitted with vacuum lines, the silane was allowed to stand in the molten state for approximately one hour or until the presence of gas bubbles was no longer noticeable. The silanes were then removed from the oven and allowed to rapidly cool to approximately 70° C (consistency of thick syrup) and then poured into a mold. The means used to judge the opportune time for pouring was visual and resulted in frequent remoldings until good technique was developed. An attempt to slowly cool the melt in the hot air furnace resulted in crystallization. Phosphors poured too hot began crystallizing as they cooled, whereas those poured too cool were prone to trap air and form bubbles in the glass. It was observed that a correctly poured phosphor had a mercury-type meniscus whereas a water-type meniscus resulted if the melt was poured while too hot. This fact was recorded by Cooper (Ref 9:15) in his report. Phosphors were accepted only when they showed no evidence of either crystallization or air trapping.

Molds. The mold used for pouring large samples was made from two pieces of one-quarter inch lucite stock. The lower portion of the mold was a two inch diameter disc, while the upper part was a one-inch-inside-diameter lucite ring with an overall diameter of two inches. In use, the two pieces were clamped firmly together with a hose clamp and the molten silane poured into the mold. Upon cooling. the silane glass bound the form together and the assembly was ready for use with a photomultiplier tube, the mold becoming a light piper.

Small samples were molded for use in a Bowman-Aminco spectrophotofluorometer, the apparatus used in obtaining fluorescence spectra. These samples were molded to a size corresponding to the inside dimensions of the quartz cell furnished with the spectrophotofluorometer. For this purpose a mold was constructed of wood to form a cavity with a 25/64 inch cross section. The wood was covered with household saran wrap prior to pouring the molten silane. The form was put together with screws and could be disassembled for removal of the completed silane glass.

The Samples. Three glasses of silane I were molded to the approximate size of the anthracene crystal used as a standard. The physical characteristics of these samples are contained in Table II on the following page. The thickness of the silane I glass is approximate since the mold was filled to capacity and the mercury-type meniscus extending above the form was neglected in measurements. The weight of glass "A" is only approximate since the weight of the mold was not recorded prior to molding. Dimensions and weight of the standard anthracene crystal are included in Table II.

Table II

Size and Weight of the Silane I Glasses And Anthracene Standard

Sample Designation	Size		Weight (grams)
	Diameter (inches)	Thickness (inches)	
Glass A	1	1/4	3.55*
Glass B	1	1/4	4.1818
Glass C	1	1/4	4.0024
Anthracene	1	9/32	4.5582

* Approximate - see text

One sample of each of the three glass forming silanes was molded for use in the spectrophotofluorometer. In addition, a piece of anthracene crystal was cut to approximately the same size as the silane samples. The cross section of each was 25/64 inch square while the heights were approximately 7/16 inch. Weights of the samples used in obtaining fluorescence spectra and later for rough comparisons of scintillation efficiency are contained in Table III.

Table III

Weights of Phosphor Samples

Phosphor	Weight (grams)
Silane I	1.0175
Silane II	1.0166
Silane III	0.9979
Anthracene	1.4337

All silane glasses used in the experiment developed a milky surface growth where exposed to air. This growth was easily polished off with lens tissue prior to use, but reformed in a short time. Storage of samples in light-tight boxes or under vacuum failed to prevent the formation of this milky growth. It is presently thought that the growth is crystalline in nature, forming first in regions stressed most in cooling, namely the surfaces exposed to air. Crystallization did not immediately occur along lucite-silane boundaries, although all the silane glasses crystallized to some extent during the period the experiment was in progress.

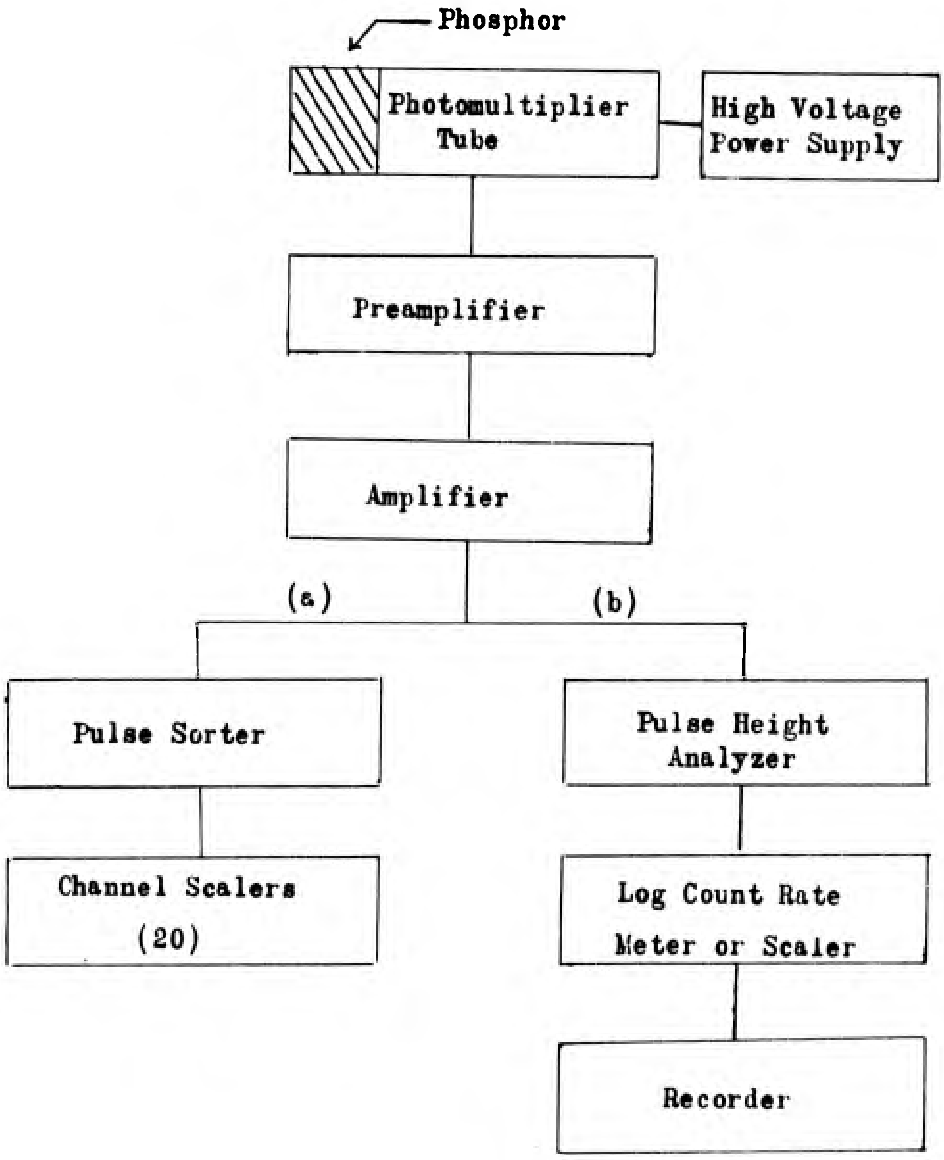


Figure 2

Spectrometer Counting Apparatus

Experimental Set-up and Equipment

An Aminco-Bowman spectrophotofluorometer, a single-channel scintillation spectrometer, and a twenty-channel scintillation spectrometer were used for data taking in this experiment. Both spectrometers were used in gathering the data for comparison of the scintillation efficiency of a given silane relative to that of anthracene, while the spectrophotofluorometer was used in determining the fluorescent spectra of the silanes and anthracene.

The Spectrometers. The arrangement of the spectrometers is shown in Figure 2 on the opposing page. In use, the single channel analyzer designated as (b) in the figure was used to obtain a preliminary differential pulse height plot. Then the twenty-channel analyzer was adjusted so that any peaks previously observed were covered within the range of one setting on the twenty-channel analyzer. This procedure permitted rapid location of interesting points of the spectrum, yet allowed computation of deviation in the recorded data.

One multiplier phototube, a DuMont 6292 was employed throughout this portion of the experiment. The silane glasses still in their lucite molds and the anthracene crystal in a light piper of the same dimensions as the silane mold were in turn optically coupled to the multiplier phototube with Dow Corning compound DC 200. It was found necessary to secure the phosphors to the photomultiplier tube to prevent slippage. A reflector of aluminum foil was placed over the phosphor and the complete phototube apparatus was placed in a light-tight chamber which had a removable aluminum cover. The base of the chamber was equipped with the phototube base together with appropriate electrical leads. The light-tight assembly was placed inside a lead shield to reduce background.

In the case of the small phosphors, an additional light piper was constructed of lucite. This piper was similar to that in which the large silane I phosphors were molded. It was, however, constructed to permit use of any one of the small silane phosphors or the small piece of crystalline anthracene. An additional feature was the fitting of a source holder to the light piper so as to insure exact duplication of source to scintillator geometry at all times.

Two Cesium 137 sources provided the radiation for the experiment. Gamma spectra were obtained by taping a vial of Cesium Chloride to the outside of the light-tight chamber, the wall of the chamber providing a beta shield. Gamma plus beta excitation was provided by a source placed inside the light-tight chamber at a fixed distance from the scintillating medium. This source was prepared by evaporating to dryness a few drops of cesium chloride on a mylar film. The film was then attached to an aluminum slide holder. Appropriate jigs insured duplication of source to phosphor geometry in all cases.

The pulse height analyzer of the single-channel spectrometer will accept only those pulses with an amplitude falling between two preset voltages. Pulses of other amplitudes are discarded. The pulse height analyzer is equipped with a ten turn helipot which regulates the base level from zero to 100 volts. An automatic friction drive is attached to the helipot and provides a continuous scan of the energy range. A channel width of 1.0 volt was used throughout the experiment.

The base line of the twenty-channel analyzer is variable from zero to 50 volts. The pulse sorter separates pulses according to amplitude and sends the signals to twenty channel scalers. The pulse width recorded by each channel lay between two voltages separated by 0.5 volt. Thus the energy range of the instrument is from zero to 60 volts in steps of 10 volts. The twenty channel analyzer employs two additional scalers, one to record the total number of counts recorded in all channels during a run and another to record all pulses whose amplitude exceeded that recorded by the last channel. This latter scaler permits the operator to complete a run without scanning the whole energy range and still be certain that he has recorded the complete energy spectrum.

<u>The Spectrophotofluorometer</u>. The spectrophotofluorometer (hereinafter referred to as the SPF) was manufactured by the American Instrument Company, Incorporated. Its

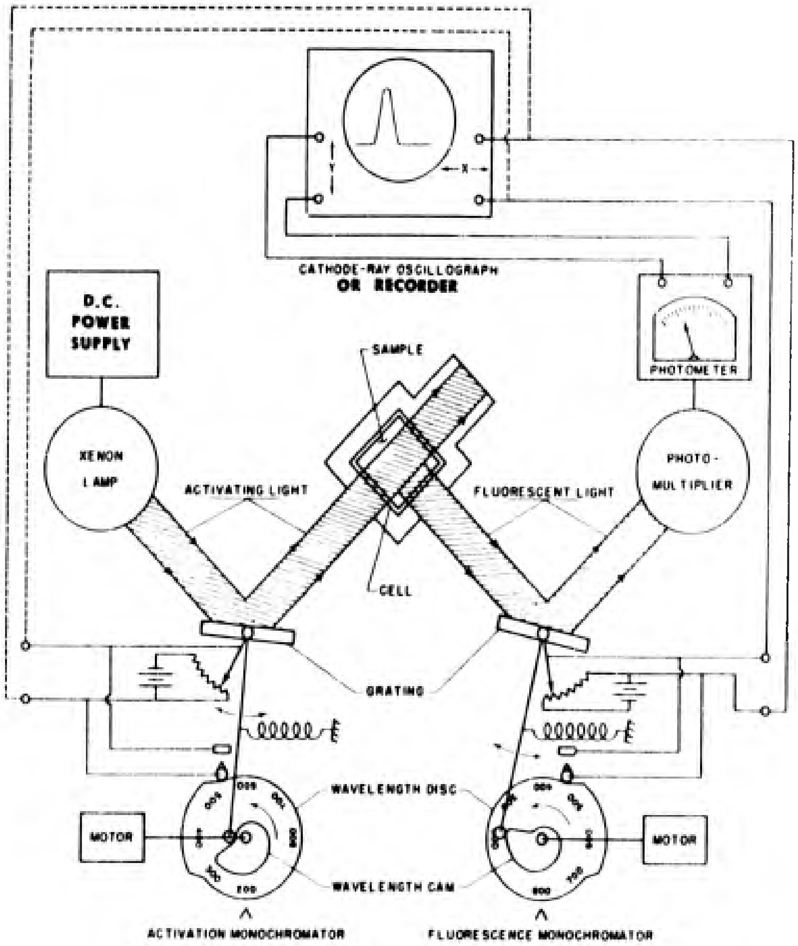


Figure 3

Schematic of Aminco - Bowman

Spectrophotofluorometer

(From Reference 17:2)

primary purpose is for quantitative assay of fluorescent compounds in solution. For this purpose it is necessary to prepare a set of work curves based on known concentration. In general, the instrument is not recommended for identification purposes since the width of fluorescent bands renders them comparatively non-specific (Ref 17:14). However, in certain cases where two materials are similar in assay and spectrophotometry, but differ in their fluorescent properties, the instrument can be used for identification purposes.

The schematic of the SPF is shown in Figure 3 on the opposing page. Activating light is provided by a xenon lamp whose spectral output extends from the limits of quartz transparency in the ultra-violet (slightly below 200 millmicrons) to about 1 micron in the infrared (Ref 17:36). The xenon lamp is located at the focus of a mirror which renders the light rays parallel and directs them to a plane grating. Dispersed light from the grating is redirected to a second mirror which focuses a monochromatic image of the lamp in the center of the sample cell. Three slits are placed in the path of the light between the second concave mirror and the sample holder (neither the slits nor the optics are shown in Figure 3). The spectral bandwidth of the activation monochromator (the optical arrangement) is determined by the width of the second slit in the light path. Fluorescent light from the sample passes through another set of three slits and into another monochromator identical to the first. This monochromator focuses the light on a seventh slit placed just before the photomultiplier tube.

As indicated in Figure 3, an electro-mechanical arrangement oscillates the plane gratings, operates a potentiometer which feeds a signal to the horizontal drive of an oscilloscope or recorder, and positions a graduated disc on the front of the machine. The graduated disc provides for visual observation and adjustment of wavelength. With the drive motor off, these discs can be moved manually. When the oscillograph (or recorder) input is connected to the scanning fluorescent monochromator and the activating monochromator is set at a wave length for maximum activation, a wave length versus intensity diagram (fluorescence spectrum) is displayed on the oscillograph screen or plotted by the recorder. Similarly, when the oscillograph X input is connected to the scanning activating monochromator and the fluorescent monochromator is set at a wave length corresponding to maximum fluorescence, an activation spectrum is displayed on the oscillograph screen. With the drive motors engaged, scanning rate of each monochromator is variable from approximately 10 to 40 seconds. Dead time, that is the interval between wave lengths of 800 millimicrons and 200 millimicrons on the wave length indicator, varies with scan rate from approximately two to six seconds. Spring loaded arms follow the cam rotation (see Figure 3) and oscillate the gratings. The maximum angular position of the gratings (for a wave length of 800 millimicrons) corresponds to the high point of the cam attached to the wavelength disc. During the return from maximum to minimum angular position, the X input is shorted by a switch actuated by a cam on the wavelength disc. Thus the trace disappears from the oscilloscope screen.

Light impinging on the photomultiplier tube from the fluorescence monochromator is converted to a weak electrical signal and fed to the photometer where it is amplified. The photometer output is coupled to the vertical axis of an oscilloscope or recorder and is simultaneously displayed on a scale contained within the photometer. The photometer is constructed in such a manner that photomultiplier tube dark current can be subtracted electronically. Photometer sensitivity is adjustable in steps of 1, 1/3, 1/10, 1/30, 1/100, 1/300, and 1/1000, the smaller of the fractions corresponding to greatest sensitivity.

For the purposes of this experiment, the SPF was modified by removing the quartz cell positioning screw from the sample holder and fitting another holder for the glass silane samples. The holder was simply a positioning block to permit precise location of the solid samples with respect to the light beam from the activating monochromator. Each sample was placed in the sample holder in such a manner that one side of the sample was parallel to the plane of the cell slits.

Calibration of the SPF was checked by inserting a Pen-ray quartz lamp into the sample holder. This lamp provided the lines of the mercury spectrum for calibration of the fluorescence monochromator. The activating monochromator was then calibrated against the fluorescence monochromator, using an aluminum foil reflector in the sample holder. A maximum reading on the photometer dial was taken as the point where the wavelengths of the activating and fluorescence monochromator coincided. Calibration was found to be good to within ± 3 millimicrons which is as close as the wavelength dials can be consistently read.

Various widths of cell and photomultiplier slits were furnished with the instrument. The arrangement used in the experiment was recommended by the manufacturer (Ref 17:13) as that giving the highest resolution. Widths of slits used (listed in the order of passage of light from source to photomultiplier tube) were 1/32, 1/64, 1/8, 1/8, 1/64, 1/32, and 1/64 of an inch.

Any one of four multiplier phototubes is recommended for use with the SPF (Ref 17:15). However, only two of the recommended types, an RCA 1P21 and an RCA 1P28, were on hand. Both tubes were tried and no significant differences were observed. Since the 1P28 is more sensitive at lower wave lengths, it was used throughout the experiment.

The experimental procedure was to scan with the fluorescence monochromator while manually adjusting the activating wave length and observing the trace on the oscilloscope screen. When fluorescence was observed, the activating wave length was left fixed and the fluorescent spectrum recorded on the X-Y recorder. The output signal from the photometer was strong enough that an additional load imposed by the recorder did not affect the spectrum. Fluorescence spectra were recorded for activating wave lengths progressively 10 millimicrons longer than the proceeding one. From these plots the emission wave length of maximum intensity was selected and an activation scan was run in a similar manner but with the fluorescence monochromator set on the emission maximum and the activating monochromator scanning. This procedure was repeated for each of the silanes and for anthracene.

The emission spectrum of each of the silanes and of anthracene was obtained for excitation by beta particles from a fifty millicurie Strontium 90 - Yttrium source. This source was placed at a fixed distance from the sample under consideration. The source was passed through a perforated lid to the sample holder. Since this arrangement was not light-tight, the spectra was recorded in semidarkness to eliminate effects of room light. To minimize effects of instrument fluctuation, all four samples were run during the same afternoon.

Decay Constants

Decay constants of all three silanes were determined by W.L. Buck, Argonne National Laboratory, using the equipment described by Swank, et al (Ref 30). This apparatus consists of a pulser which initiates a chain of events by sending pulses through delay lines to two sweep generators. One sweep generator operates an electron accelerator, a commercial 5XP oscilloscope gun, while the second triggers an oscilloscope. The electron beam in the accelerator is deflected across a slit to produce a short pulse of electrons. These electrons impinge on a scintillator coupled to a 1P28 photomultiplier tube. The resulting pulse shape from an oscilloscope, together with timing waves are recorded photographically, furnishing a record for analysis.

Decay constants are determined, based on an exponential decay model, i.e. $I=I_o e^{-t/\tau}$ where I is the intensity of emitted light at any time, I_o is the initial intensity, e is the base of the naperian logarithm, t is the expired time, and τ is the decay constant, all in consistent units.

Table IV

Luminescent Properties of Silane I, II, and III
(All relative values listed refer to an anthracene standard)

Compound	Detection Properties				Fluorescence Properties		
	Relative Scintillation Efficiency for Gammas	Relative Scintillation Efficiency for Betas	Relative Pulse Height	$W_{1/2}$*	Emission Peak Wave Length	Activating Wave Length for Peak Emission Intensity	Decay Constant
	%	%	%	%	$m\mu$	$m\mu$	nano-sec.
Anthracene	100	100	100	17.8	442	425	27.0
Silane I	~92	~71	~45	17.2	400	362	2.53 - 2.54
Silane II	—	~72	~40	18.1	400	362	2.62 - 2.63
Silane III	—	~71	~41	17.8	450	380-390	3.29 - 3.31

*$W_{1/2}$ is the percentage full width of the pulse height distribution curve at one-half the maximum.

IV. Results and Conclusions

The results of this experiment are summarized in Table IV on the opposing page. Fluorescence spectra of the silanes and of anthracene are contained in Appendix A. These spectra (Fig. 4 through 22) show the variation of the fluorescence spectrum with wave length of exciting light.

Despite the presence of silicon in the molecule, the silane phosphors appear to respond as pure organic materials to gamma excitation, that is, the Compton process is dominant. The pulse-height distribution spectrum is virtually featureless except for a Compton edge or peak. The peak observed in the silane I pulse-height spectrum was identified by comparing two spectra obtained with Cesium 137 as a source, one with gammas and betas as the exciting media, the other with the beta screened off. The gamma plus beta spectrum shows a well defined peak which was attributed to the Cs^{137} internal conversion electron. This peak and the linearity of the equipment permitted development of an energy scale. On this energy scale, the peak observed for gamma excitation corresponded to the energy expected for the Compton peak. This analysis also proved that no observable photopeak existed. If a backscatter peak is present, it is masked by the Compton distribution.

Results of experiments with each of the silanes are contained in the following paragraphs.

Silane I

Relative Scintillation Efficiency. Relative efficiency for gamma detection was computed by comparing areas beneath differential pulse-height curves obtained with a gamma source in fixed geometry. Comparison of such curves for silane I and anthracene revealed silane I to be about 92% as efficient a gamma detector as anthracene.

Beta detection ability was determined by comparing areas beneath the internal-conversion-electron peak, that is, by comparison of total counts. Comparison of gamma only and gamma plus beta pulse-height distributions revealed that the gamma spectrum tailed off to zero on practically a constant slope following the Compton edge. This fact was used as a basis for subtracting gamma contribution to the internal-conversion peak prior to comparing areas beneath the peaks. The corrected peaks were used in computing the resolution.

The above results are not in agreement with those of Cooper (Ref 9:34) who concluded that silane I was a better gamma detector than anthracene. It is to be noted that in this experiment, the silane and anthracene phosphors were similar in size and weight (see Table II, page 21) whereas Cooper states that identical geometries were not obtained in his work (Ref 9:28).

Fluorescence Spectra. The fluorescence intensity of silane I increased with increase in wave length of activating light until a maximum intensity was reached for an activating wave length of 362 mμ (see figures 4 through 6, Appendix A). For activating wave lengths greater than 362 mμ, the emission intensity decreased and the fluorescence peak moved to longer wave lengths. The author is not prepared to offer an explanation of this phenomena, nor of the fact that scatter of incident light was observed only for activating wave lengths greater than 362 mμ (see figures 6 through 8, Appendix A).

The fluorescence spectrum under beta excitation (Figure 20, Appendix A) is similar in shape to that obtained with ultra-violet light. However, the relative light output as compared to that of anthracene (Figure 21, Appendix A) is small.

John (Ref 18) has examined the fluorescence spectrum of silane I at room temperature and at liquid nitrogen and liquid helium temperatures, using the mercury spectrum as the exciting medium. He found that the fluorescence peak occurs at 3900 to 4000 angstroms. He further observed that the silane phosphoresces both at room temperature and at liquid nitrogen temperature with the peak intensity occuring at about 5440 angstroms. This phosphorescence decays, apparently in two components, with half lives of 1.6 to 1.9 seconds and about 0.4 seconds at liquid nitrogen temperatures. Undoubtedly this phosphorescence contributes to the tail observed in the fluorescence spectra. This phenomenon, which also occurs at room temperature, could be detrimental to the use of the silanes as radiation detectors since it could result in after-pulsing of the multiplier phototube.

Silane II

Relative Scintillation Efficiency. Beta efficiency of silane II was measured in a manner similar to that described above. In this case, it was necessary to assume that the pulse-height distribution curve following the Compton edge approached zero. Insufficient material was available to permit pouring a large phosphor and it was felt that the small phosphors used in other portions of this experiment were inadequate for an evaluation of gamma efficiency. Consequently, no comparison of gamma detection ability was made.

Resolution was again determined from internal-conversion electron peaks after correction for gamma contribution.

Fluorescence Spectra. Behavior of silane II under ultra-violet excitation differed from that of silane I. The emission peak increased in intensity up to an activating wave length of 362 mμ as before. However, the fluorescence peak occurred at a wave length of about 430 mμ for short wave lengths of activating light, shifted to 400 mμ as activating wave length approached 360 mμ, then to longer wave lengths as the activating wave length was increased (See Figures 9 through 11, Appendix A). Some structure, apparently a double peak, is discernible in

Figure 10. However this structure could not be resolved.

The fluorescence spectrum obtained with beta excitation peaked at 370 to 380 millimicrons instead of the 400 mμ found for ultra-violet excitation. However, the observed peak lies within the range of those encountered with ultra-violet light.

Silane III

Beta efficiency for silane III relative to that of anthracene was determined as outlined above. Due to shortage of silane III, no large phosphors were made and no analysis of gamma detection ability was attempted.

The fluorescence peak of silane III occurred at 450 mμ for short activating wave lengths, shifted to shorter wave lengths as the activating wave length approached 360 to 370 mμ, then back to 450 mμ for an exciting wave length of 380 mμ (See Figures 13 through 16, Appendix A). The emission intensity maximum occurred at the latter point. For activating wave lengths longer than 380 mμ, the emission intensity decreased and emission wave length shifted to longer values. As was the case for the other silanes, no appreciable scattering of activating light occurred until activating wave length was longer than that corresponding to the peak light output.

The fluorescence spectra under beta excitation was found to be ill-defined with no prominent peaks (Figure 20, Appendix A). Emission intensity was far less than that of the other silanes.

Discussion of Results

Experimental techniques and equipment used in comparing the efficiencies of the silanes relative to that of anthracene are deemed adequate. However, many more samples with many more sources of radiation need to be examined before results could be considered conclusive. Comparison of the fluorescence spectra under beta excitation (Figures 20 and 21, Appendix A) indicates that there should have been more difference in pulse-heights among the silanes. However, the device used to mount the small phosphors on the multiplier phototube was crude and the possibility exists that there was more light lost at lucite-phosphor interfaces in one case than in another.

Examination of the fluorescent spectra of the silanes and anthracene in view of the response curve for the DuMont 6292 multiplier phototube (see Figure 22, Appendix A) justifies the use of this tube in comparing scintillation efficiencies. That is, the response of the DuMont 6292 provides a reasonable match for the spectral output of the silanes and anthracene.

Two aspects of the determination of fluorescence spectra are questionable. No method was employed to insure that the surfaces of the various phosphors were equally smooth. Each phosphor was used as it came from the mold, except that great care was taken to polish off crystalline films which formed. The possibility of scattering from the surface definitely exists. The second questionable facet casts a small element of doubt on the shapes of the fluorescent spectra. The spectrophotofluorometer used a friction drive to move the gratings. At slow speeds, this drive was less powerful and the rate of scan was a little erratic because of slippage and stalling. Even the slowest rate of scan possible is deemed too fast. In rapidly changing regions of the fluorescence spectrum, the X-Y recorder was moving too fast and momentum of the pen carriage caused overshooting. However, within experimental error the spectra are comparable, that is, the rate of scan for each spectrum was about the same. It should be mentioned that the fluorescence spectra contained in the appendix are reproductions of experimental data and have not been corrected for response of the multiplier phototube.

V. Recommendations

In the conduct of this experiment, many areas of further research as well as improvements in procedural methods became obvious. These are listed below.

1. A larger number of phosphors of various sizes should be prepared and compared to equivalent sizes of anthracene and to each other. This study should involve the use of other radionuclides and possibly an electron accelerator as sources of ionizing radiation. Some system of insuring a high polish on the faces of the silane phosphors should be developed to minimize loss of fluorescence photons by internal reflection.
2. The luminescence process in these organic glass compounds should be studied. This would involve a theoretical study with subsequent verification of the theory. Such a project would provide a valuable contribution to the study of luminescence as well as the study of these glassy scintillators. This study should attempt to correlate the behavior of the silanes in the glassy state to that of the crystalline form. John (Ref 18) has been successful in growing a crystal of silane 1. Perhaps all three silanes could be prepared in the crystalline state.

3. The spectrophotofluorometer used in this study could perhaps be modified to permit a slower and more even rate of scan. This could be done by arranging a different and more powerful drive mechanism. Perhaps a complete new experimental arrangment could be assembled to work on the same principles as the SPF but eliminate the shortcomings inherent in using a piece of equipment for purposes other than that for which it was designed. Some attempt should be made to calibrate the intensity of the xenon lamp for each wave length of activating light. This may be impossible since the xenon lamp varies in intensity (Ref 17:14).
4. Other properties of the silanes should be thoroughly investigated. This should include but not necessarily be limited to a study of phosphorescence, self-absorption of the fluorescent light and radiation damage to the scintillators. Certainly any one of these properties could adversely affect the use of the silanes as scintillators. Both Cooper (Ref 9) and Downs and Smith (Ref 12) have stated that pulse height decreases with increasing glass thickness. This area should be investigated and a determination made as to the reason for this phenomena. Self-absorption or presence of impurities is indicated.

Phosphorescence could contribute to multiplier phototube after-pulsing if present to sufficient degree.

5. A study of pulse-heights versus energy of incident radiation should be conducted. An electron accelerator might be used for this purpose. Such a device would be convenient since it would provide a source of monoenergetic radiation.

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APPENDIX A

Fluorescence Spectra
of
Silanes and Anthracene

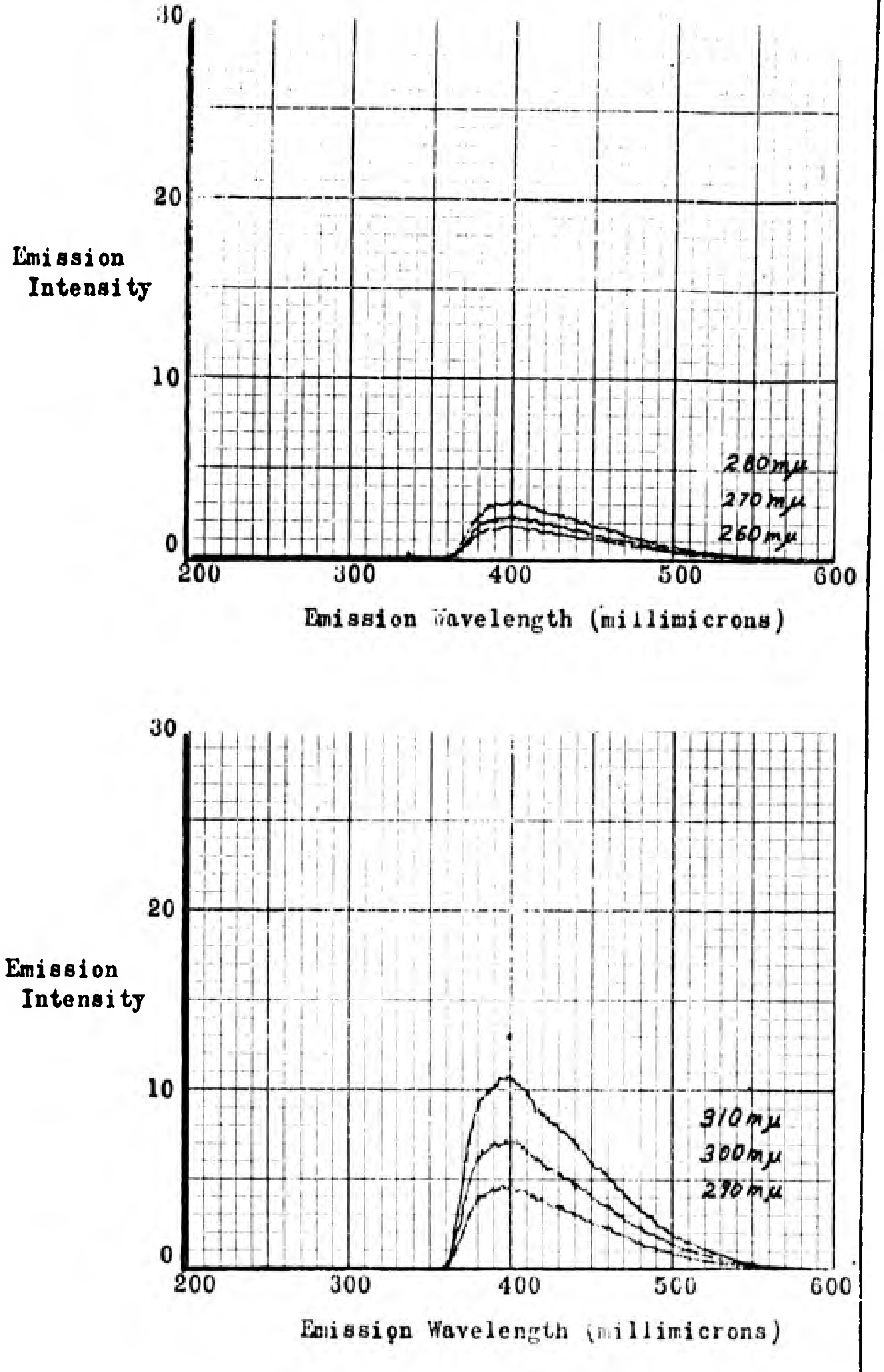

Figure 4
Fluorescence Spectrum of Silane I
U.V. Excitation: 260,270,280,290,300,310 Millimicrons

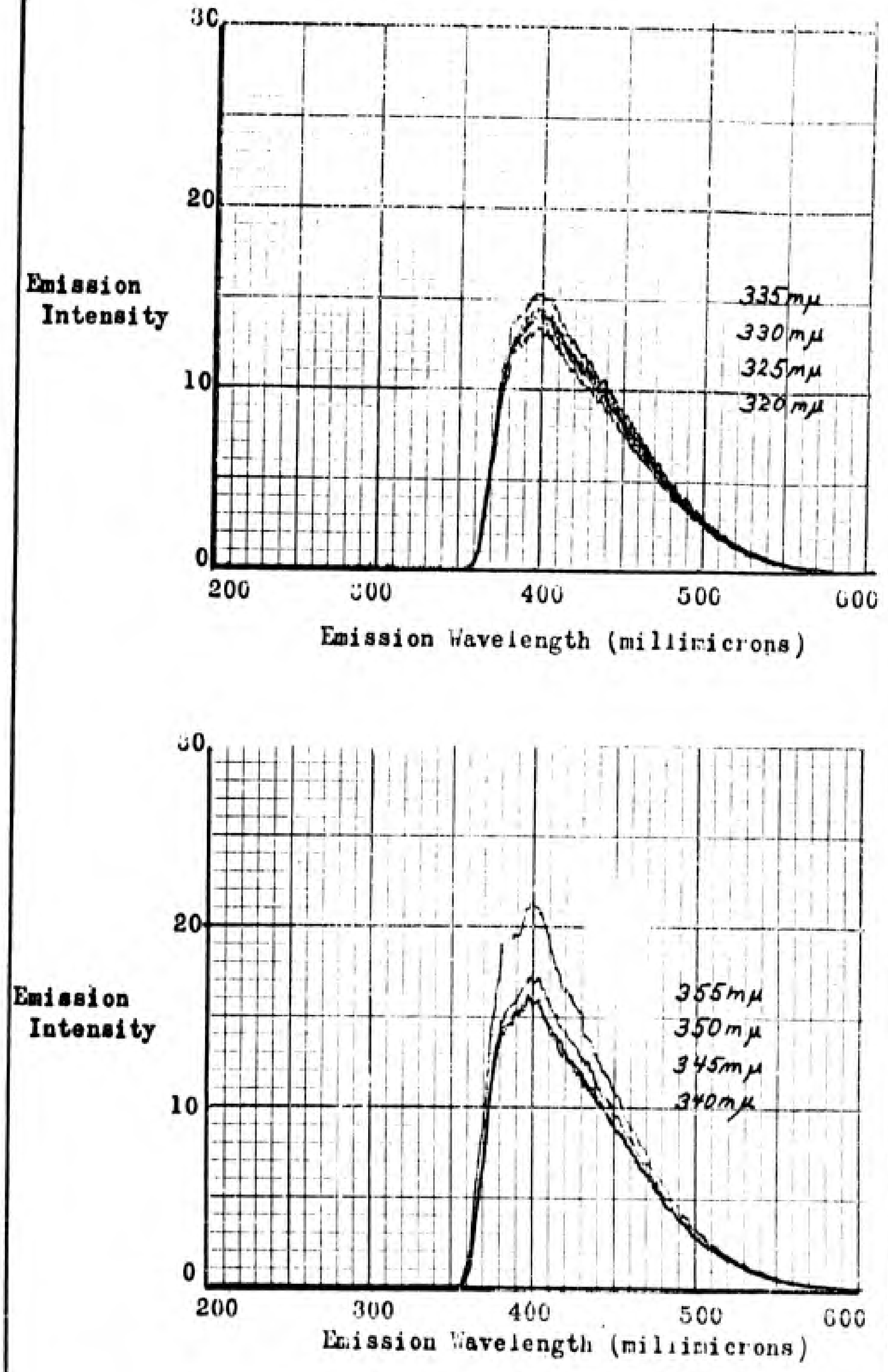

Figure 5
Fluorescence Spectrum of Silane I
U.V. Excitation: 320,325,330,335,340,345,350,355 Millimicrons

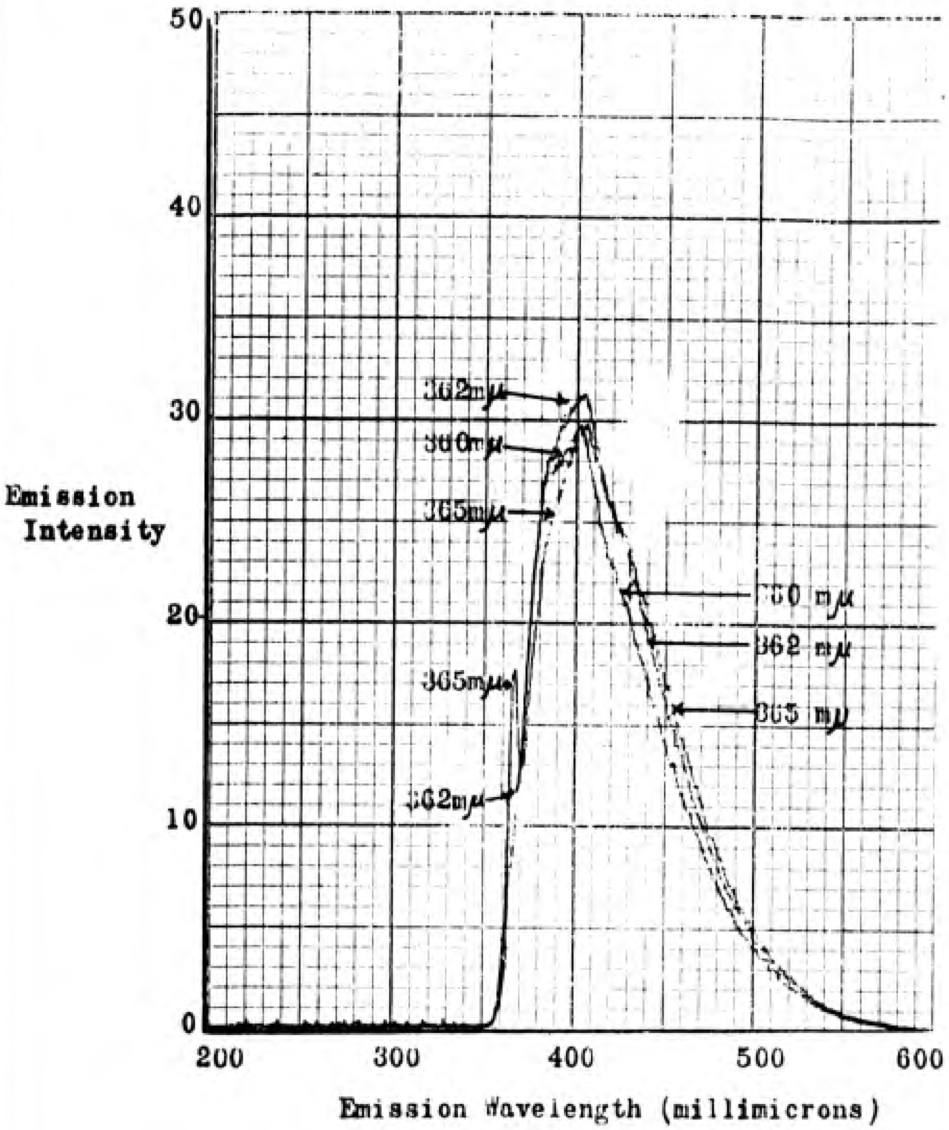

Figure 6

Fluorescence Spectrum of Silane I

U.V. Excitation: 360, 362, 365 Millimicrons

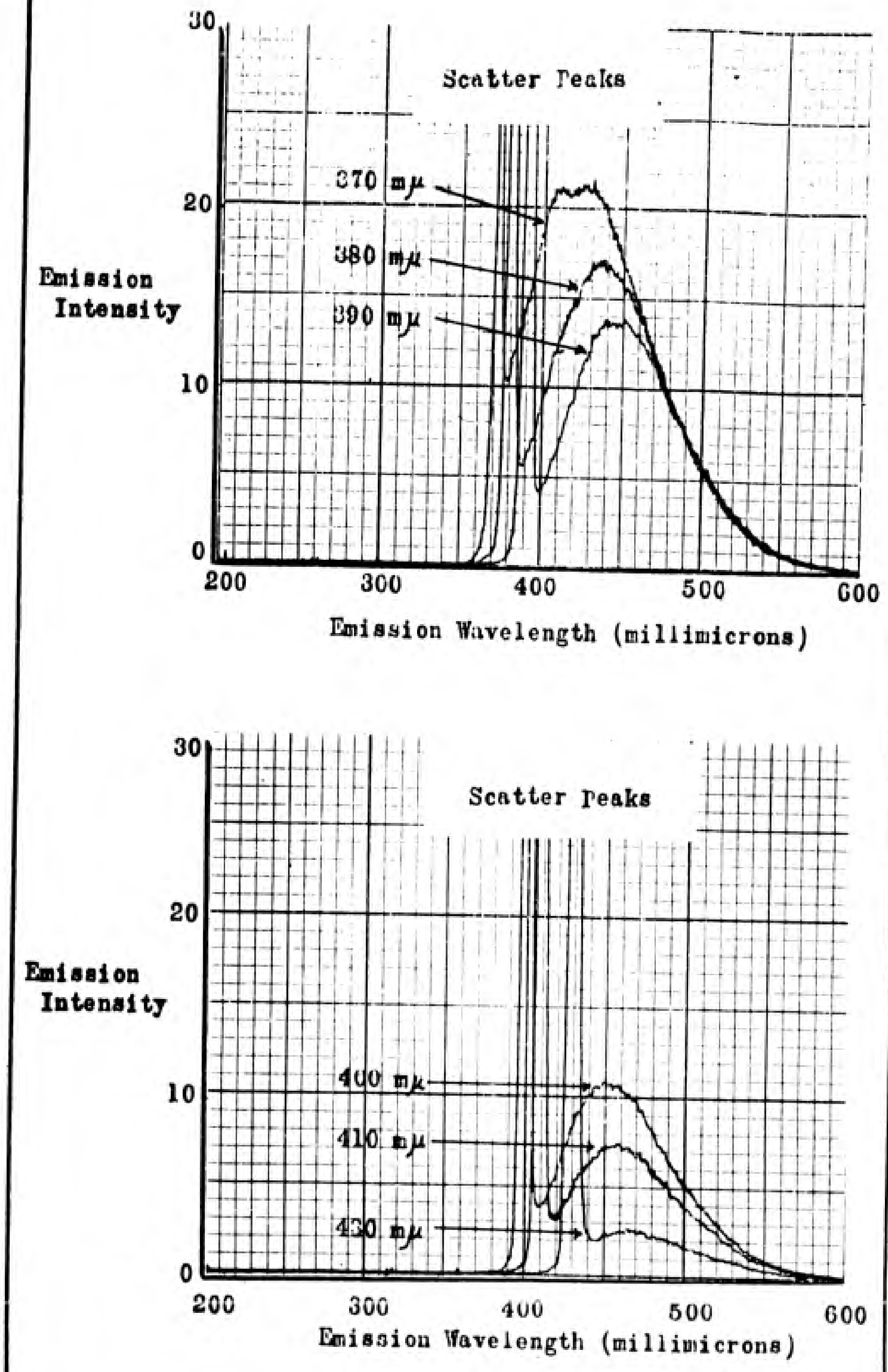

Figure 7
Fluorescence Spectrum of Silane I
U.V. Excitation: 370,380,390,400,410,430 Millimicrons

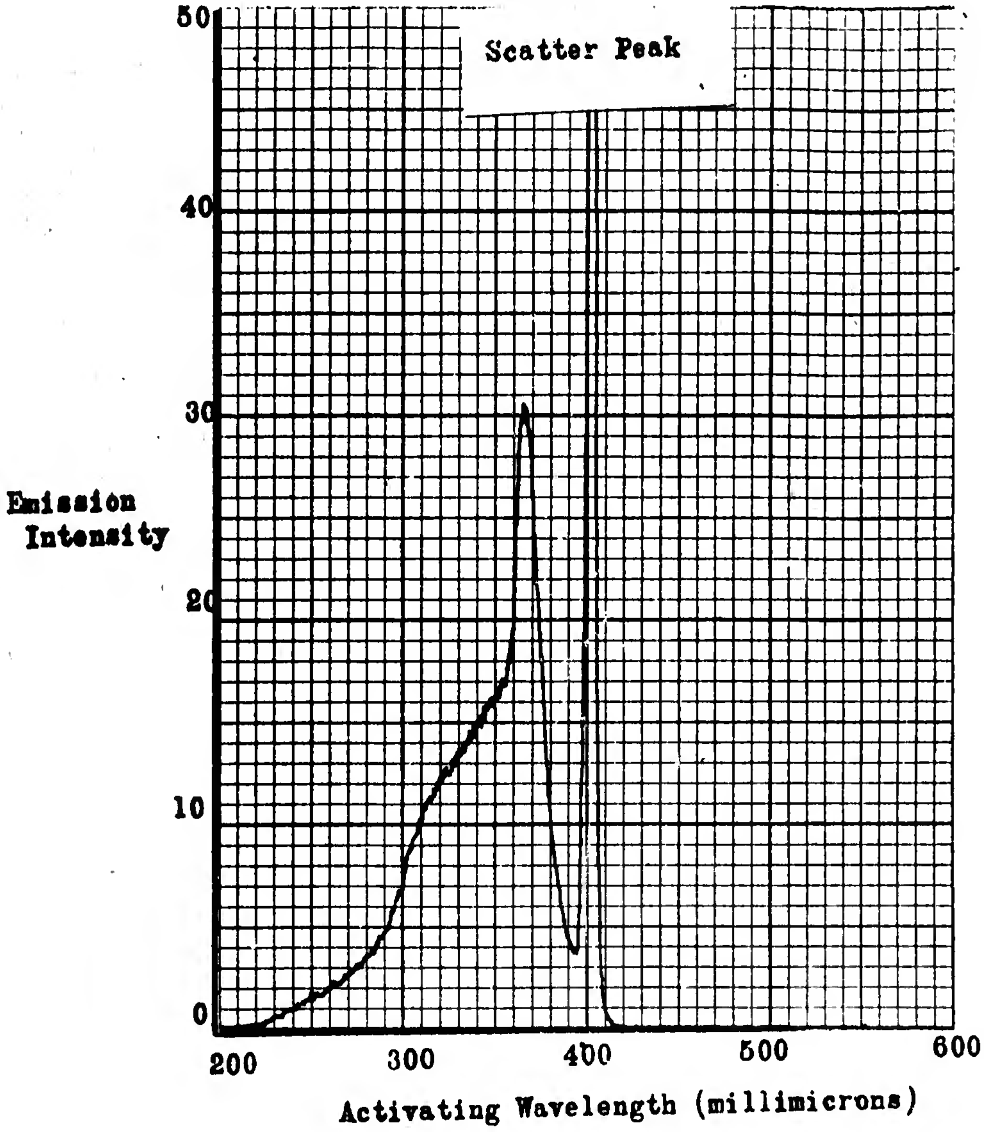

Figure 8

Silane I: Intensity of 400mμ Emission Line

As A Function of Activating Wavelength

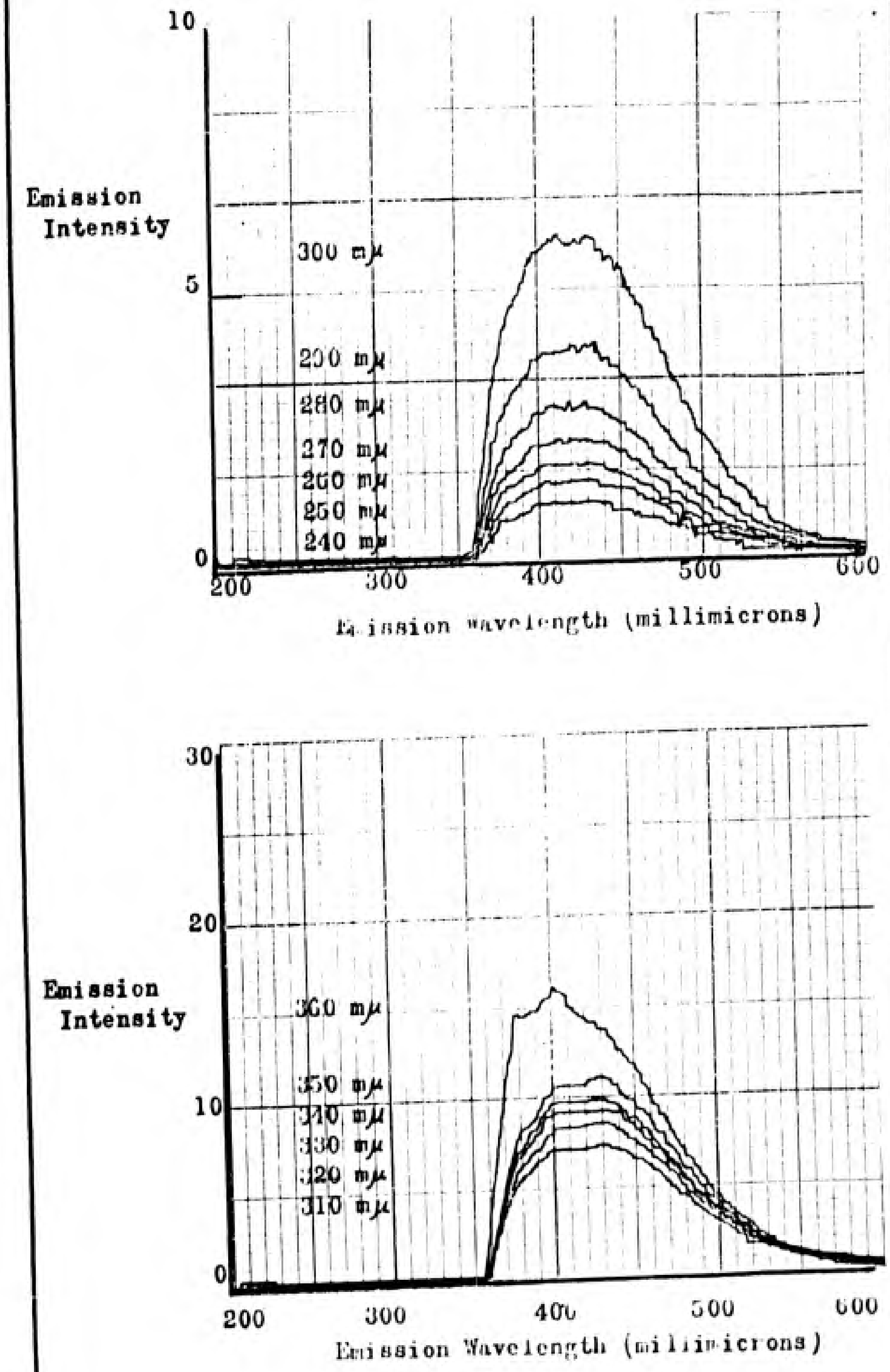

Figure 9
Fluorescence Spectrum of Silane II
U.V. Excitation: Activating Wavelength as Indicated

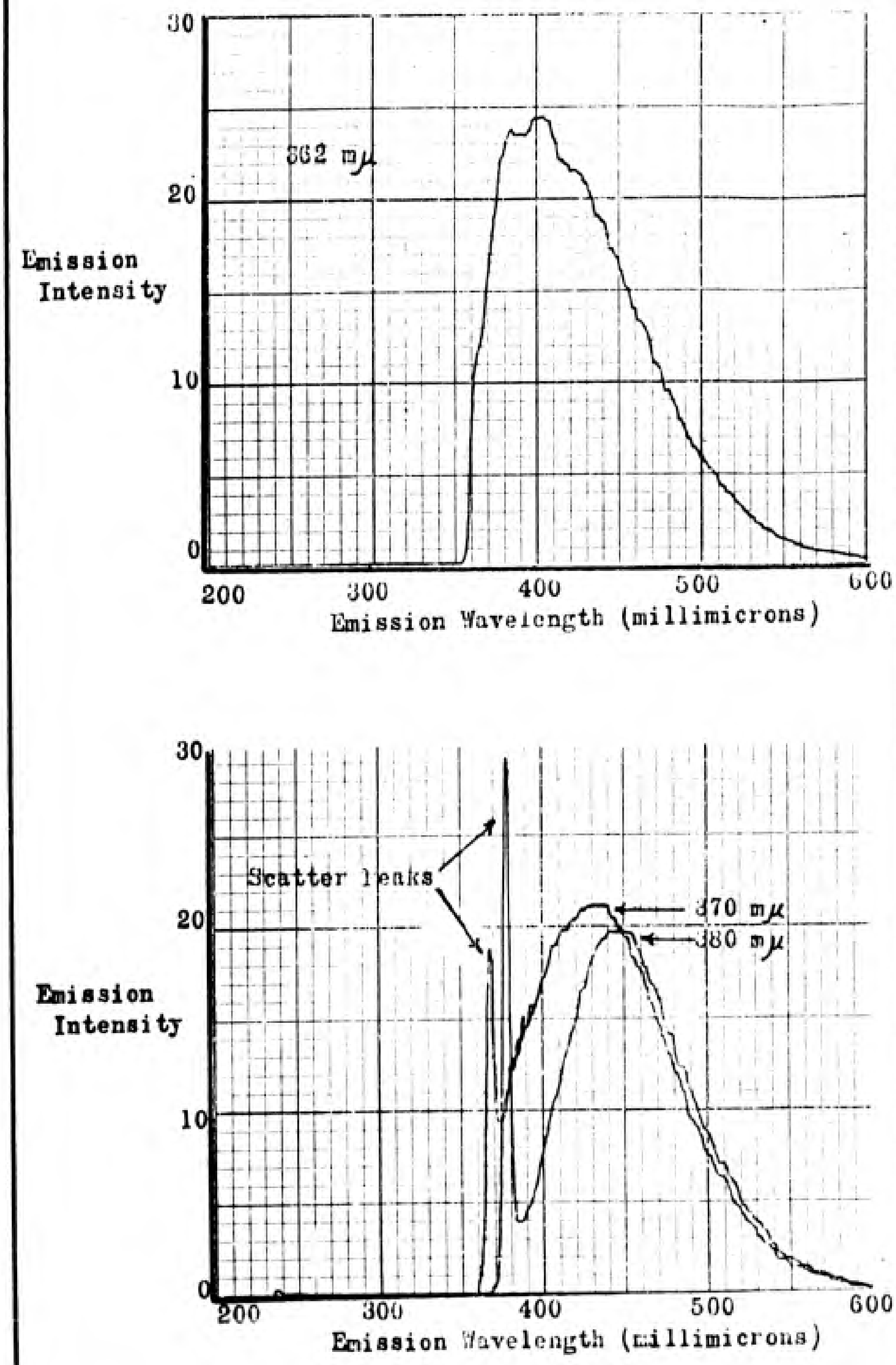

Figure **10**
Fluorescence Spectrum of Silane II
U.V. Excitation: 362, 370, 380 Millimicrons

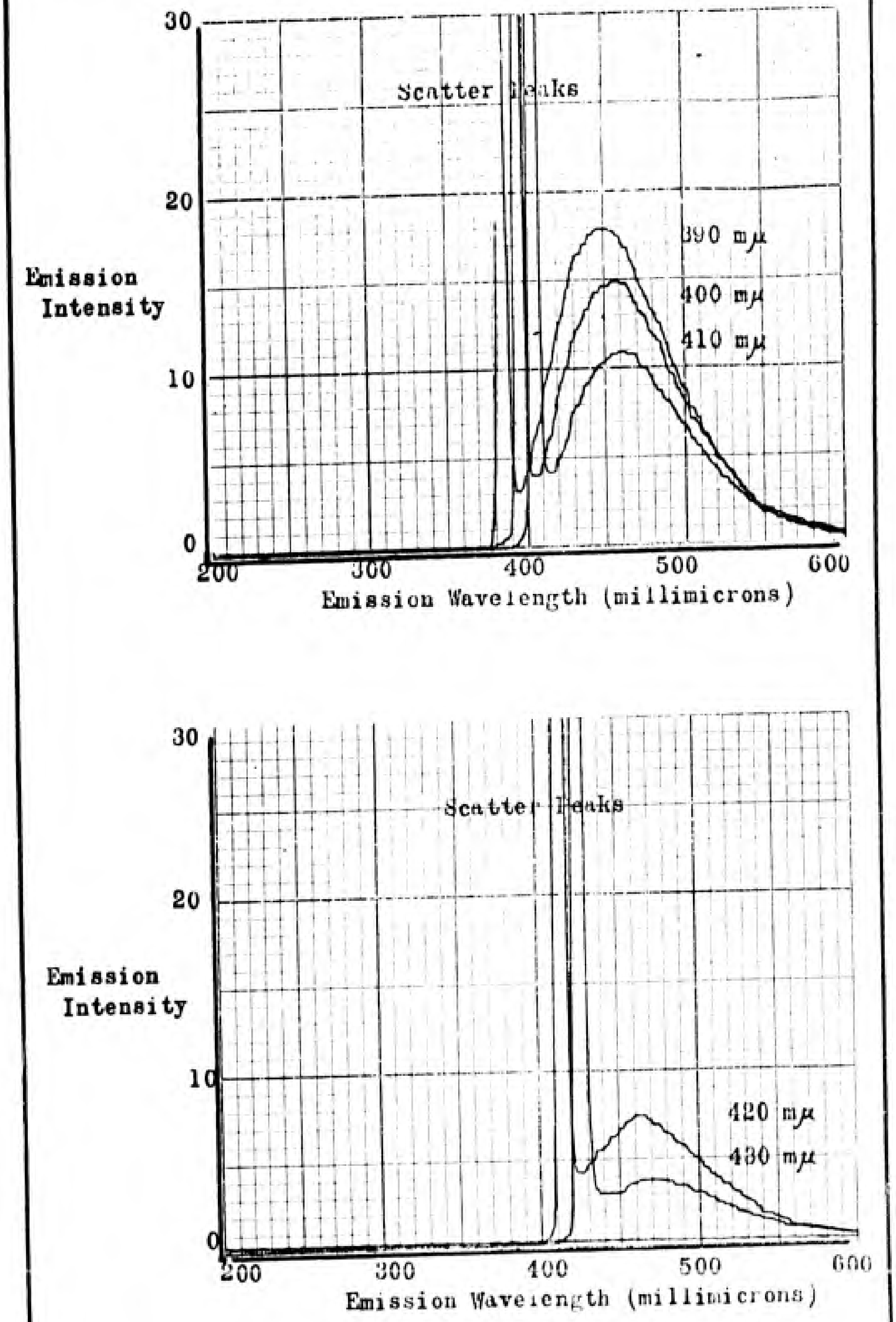

Figure **11**
Fluorescence Spectrum of Silane II
U.V. Excitation: 390, 400, 410, 420, 430 Millimicrons

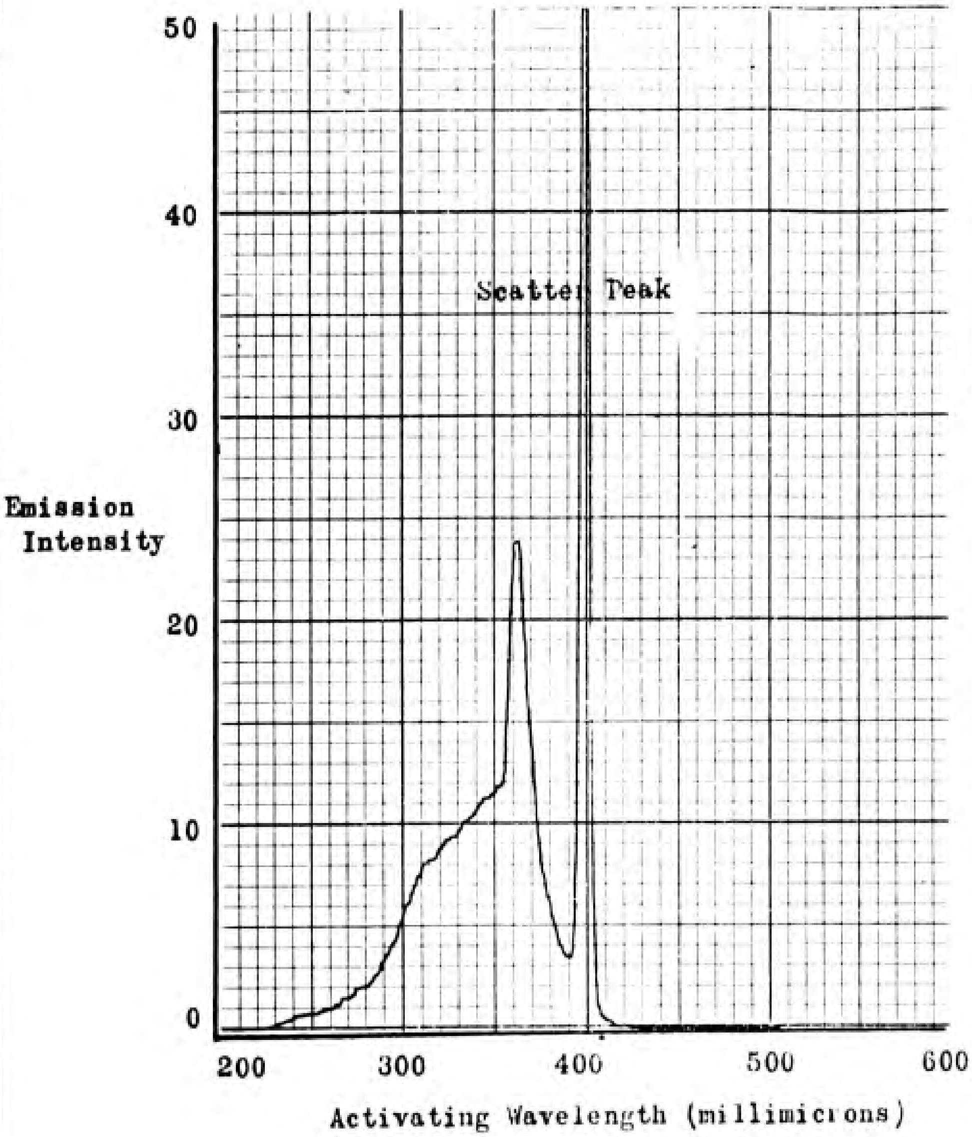

Figure 12

Silane II: Intensity of 400 mμ Emission Line
As a Funtion of Activating Wavelength

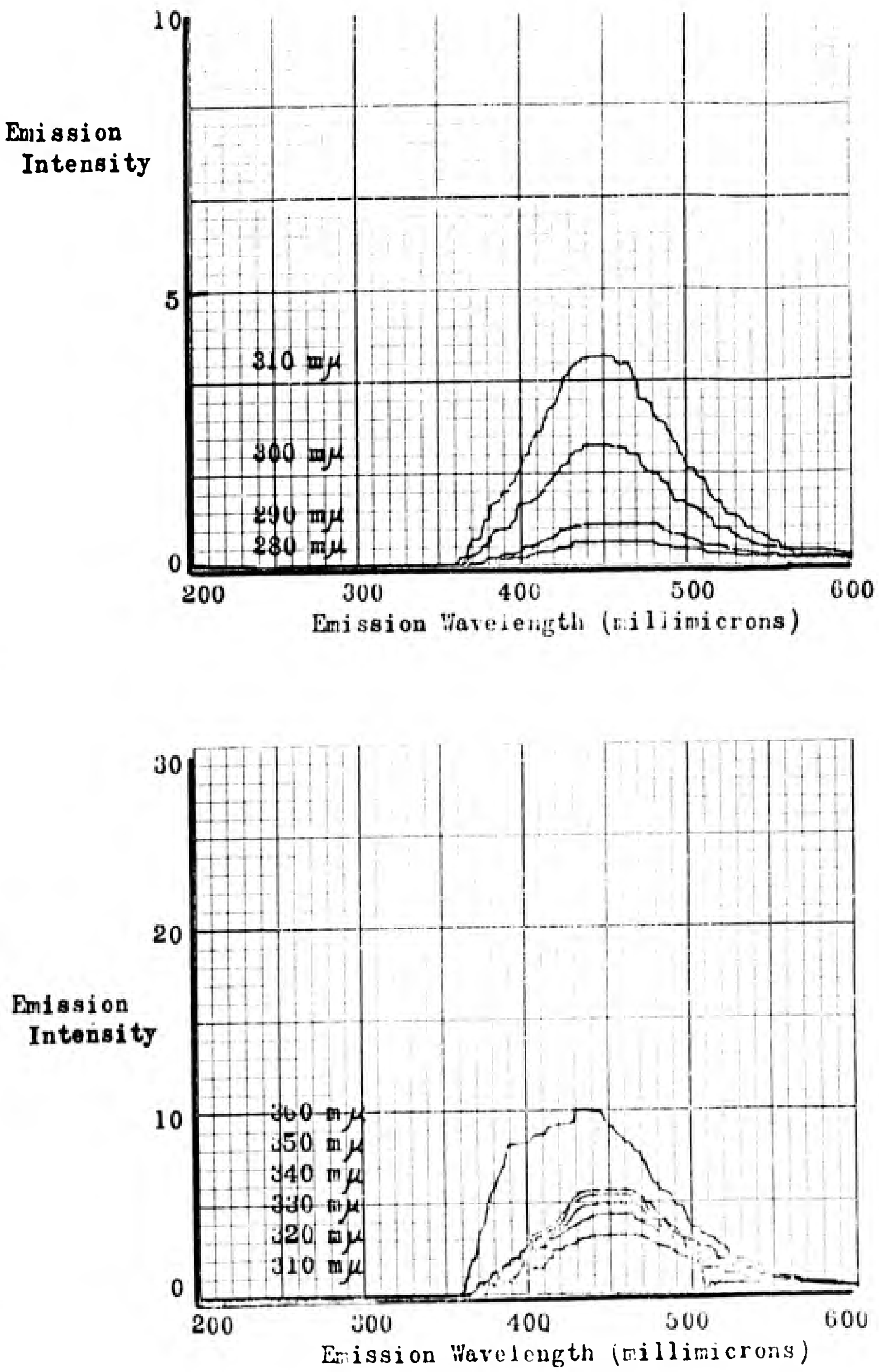

Figure 13
Fluorescence Spectrum of Silane III
U.V. Excitation: Activating Wavelength as Indicated

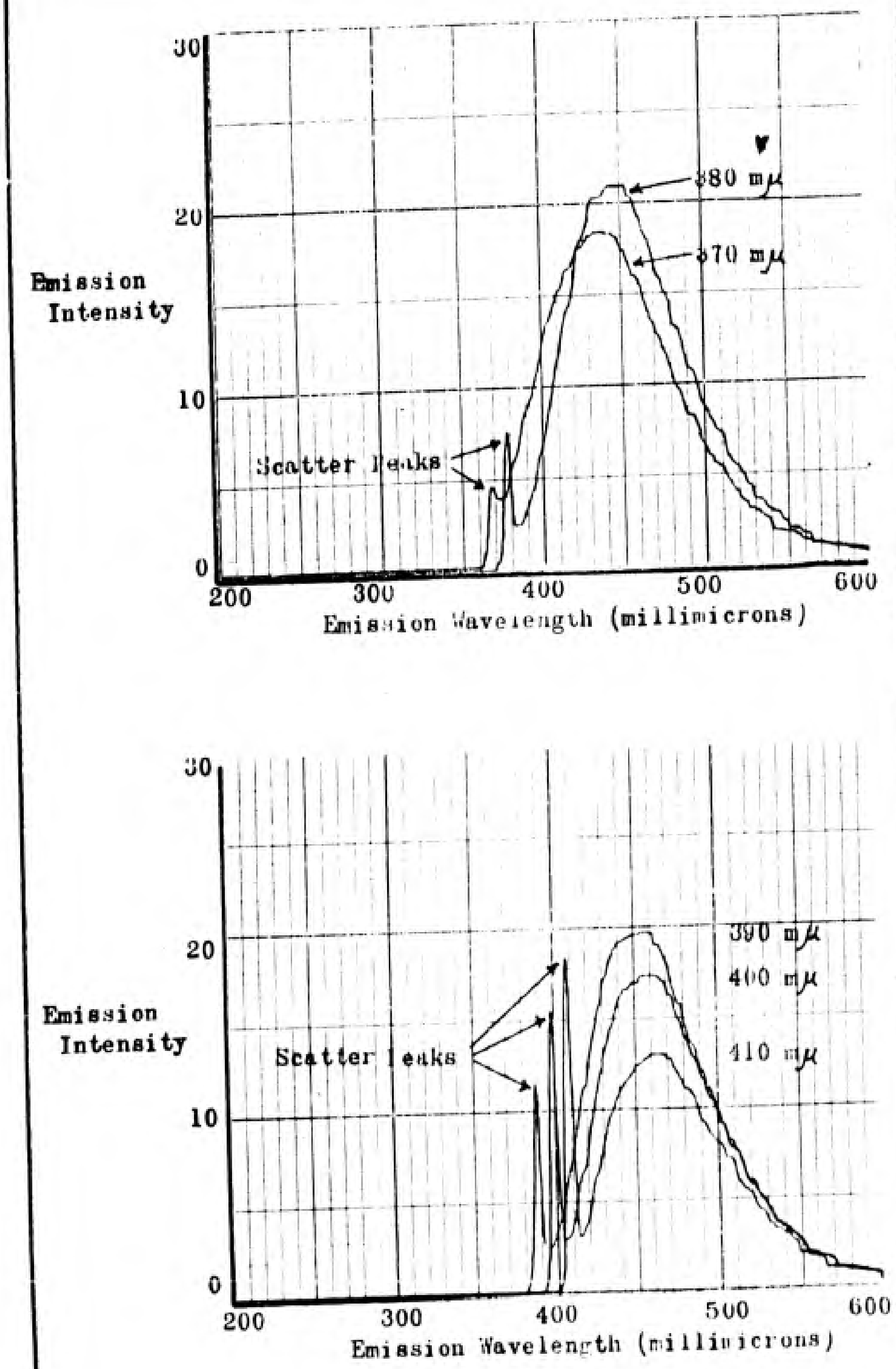

Figure 14
Fluorescence Spectrum of Silane III
U.V. Excitation: 370, 380, 390, 400, 410 Millimicrons

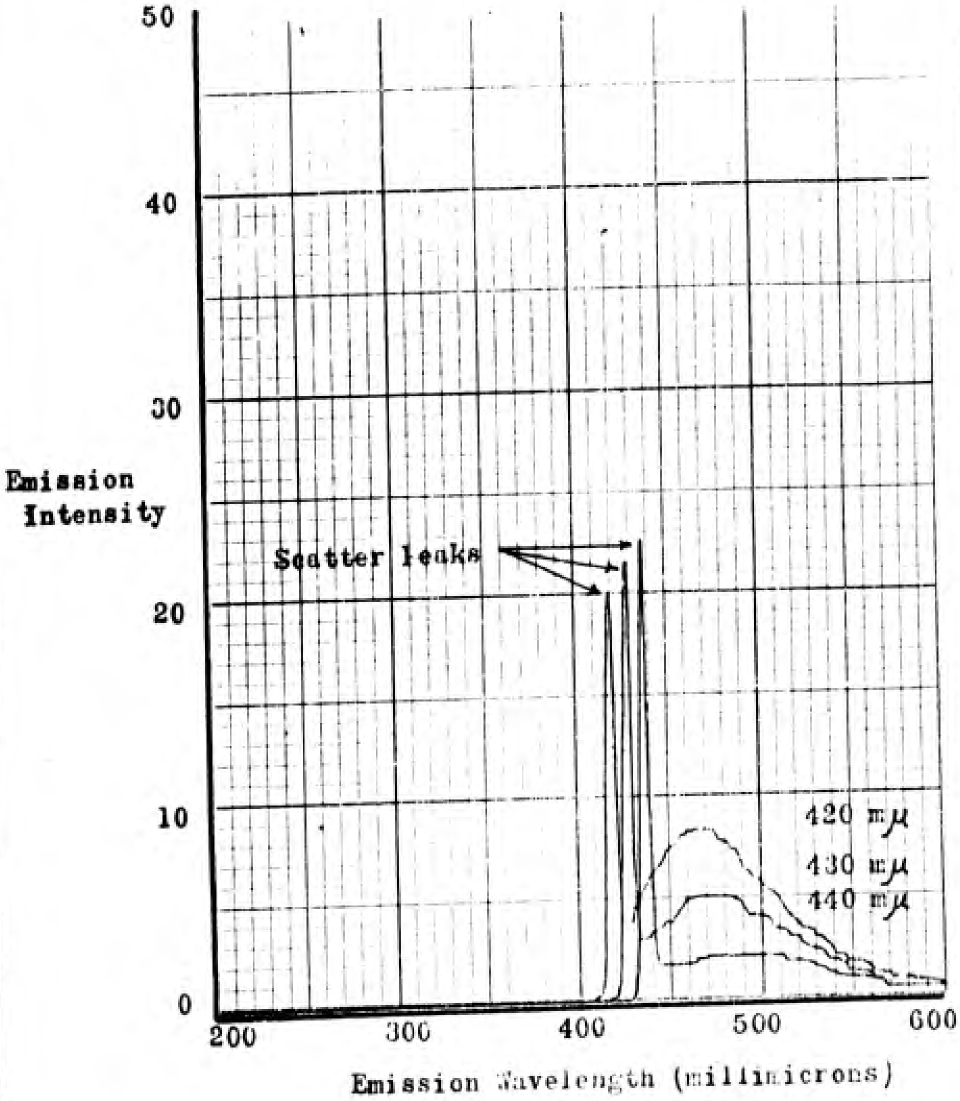

Figure 15

Fluorescence Spectrum of Silane III

U.V. Excitation: 420, 430, 440 Millimicrons

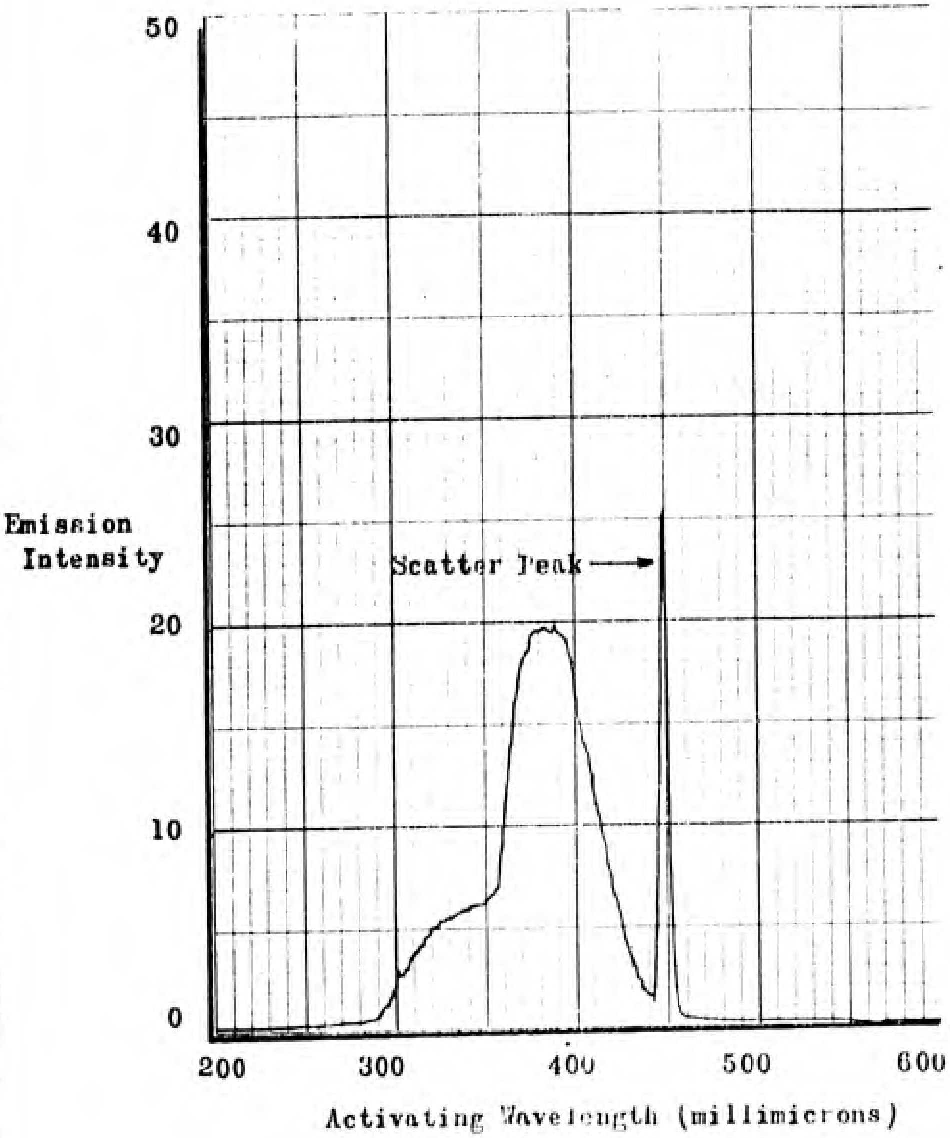

Figure 16

Silane III: Intensity of 450 $m\mu$ Emission Line As A Function of Activating Wavelength

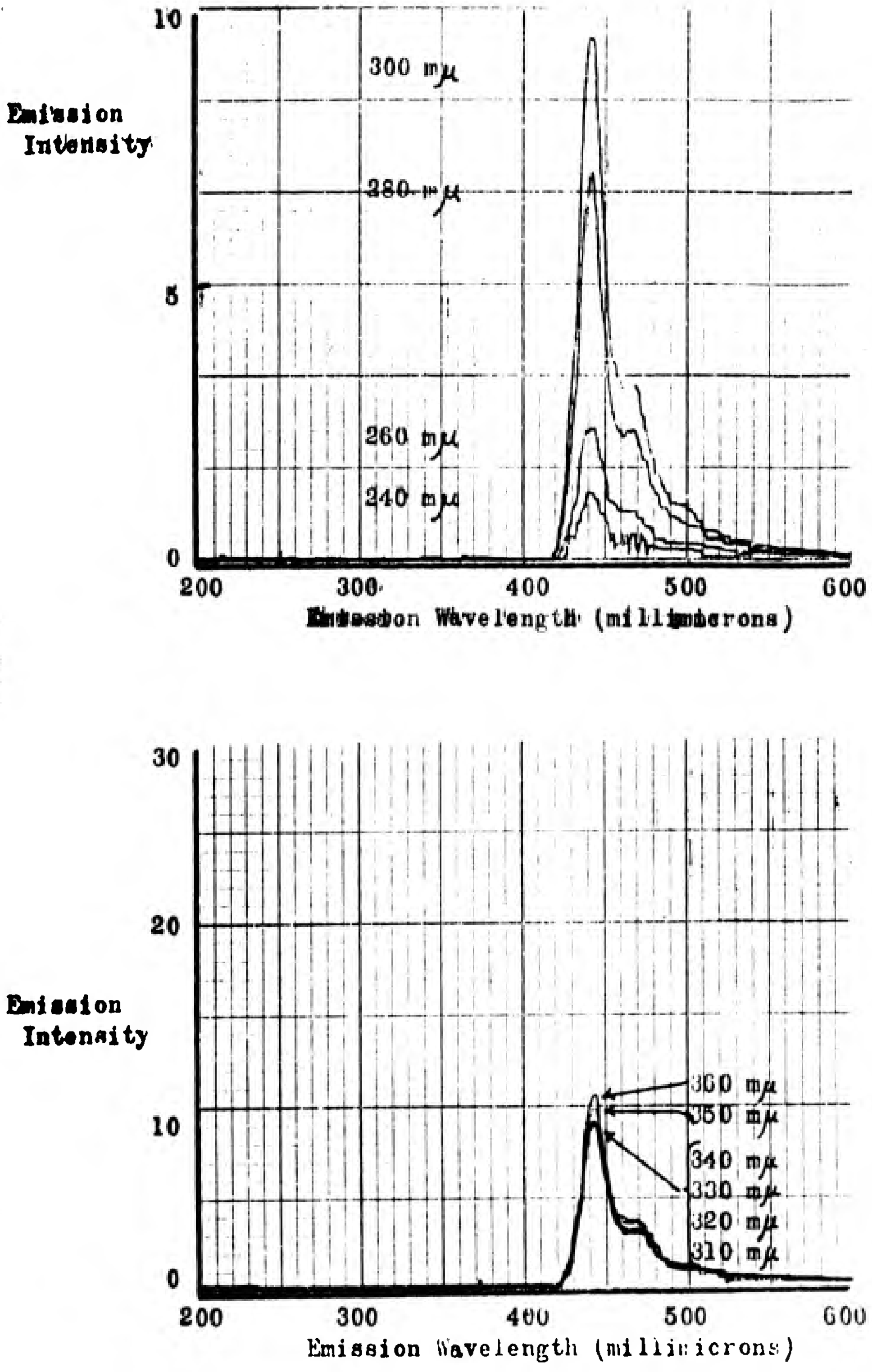

Figure 17
Fluorescence Spectrum of Anthracene
U.V. Excitation: Activating Wavelengths as Indicated

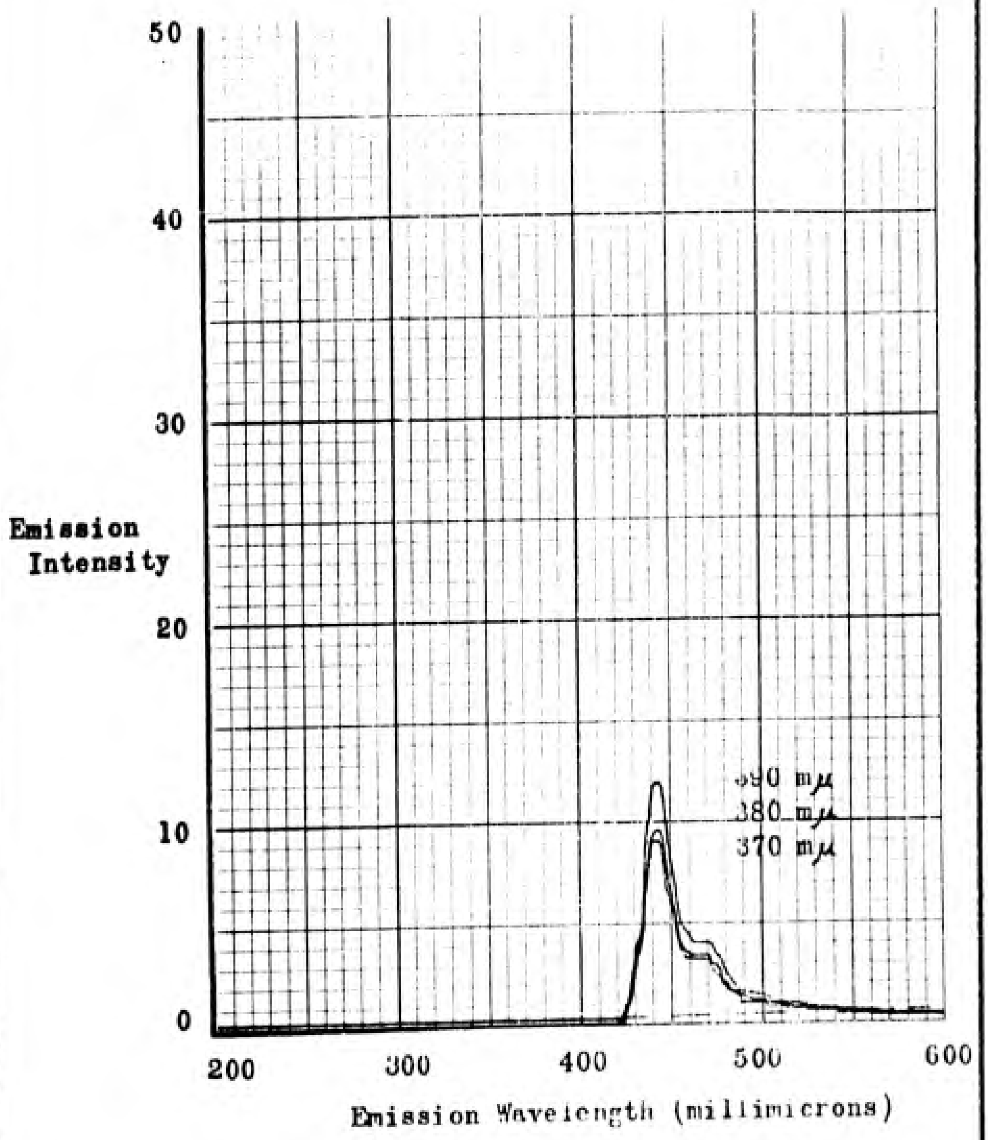

Figure **18**

Fluorescence Spectrum of Anthracene

U.V. Excitation: 370, 380, 390 Millimicrons

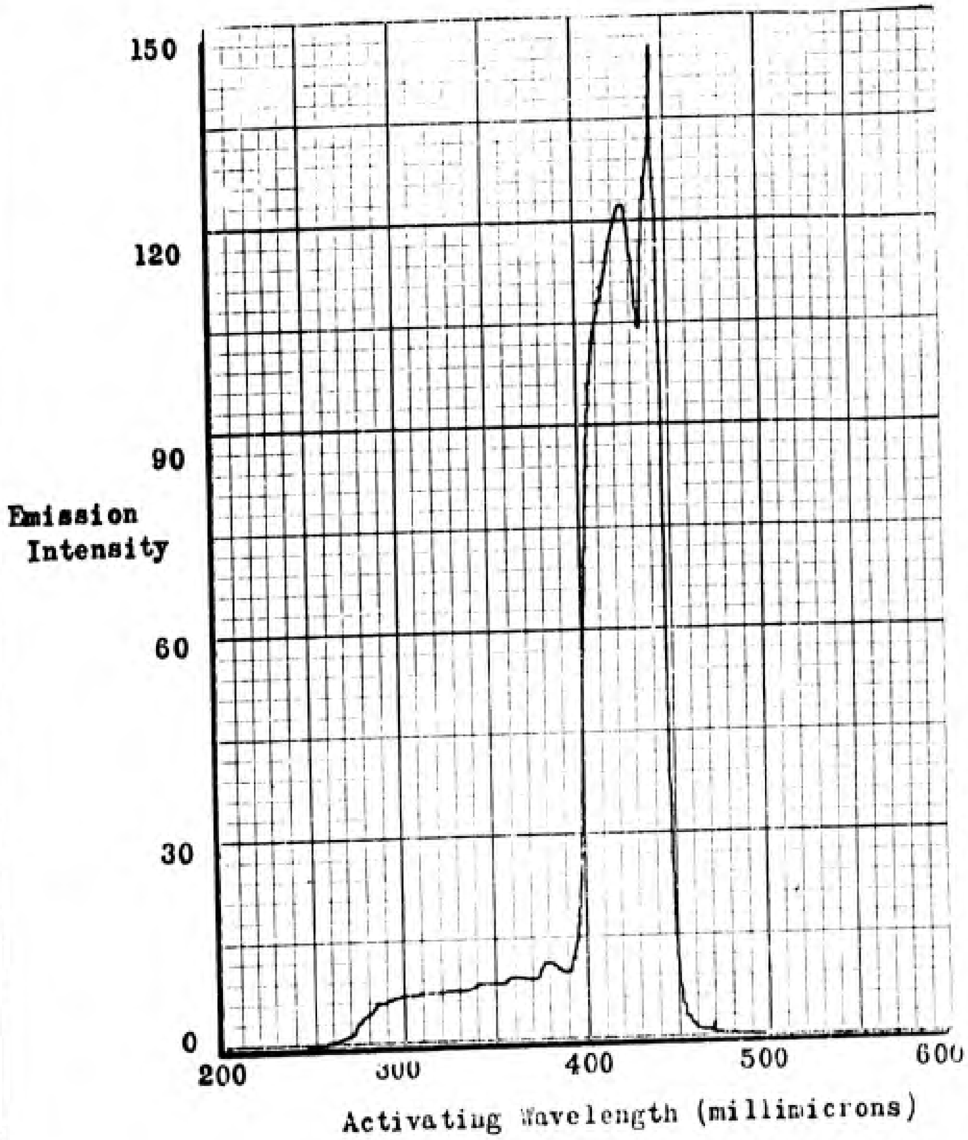

Figure 19

Anthracene: Intensity of 442 mμ Emission Line As A Function of Activating Wavelength

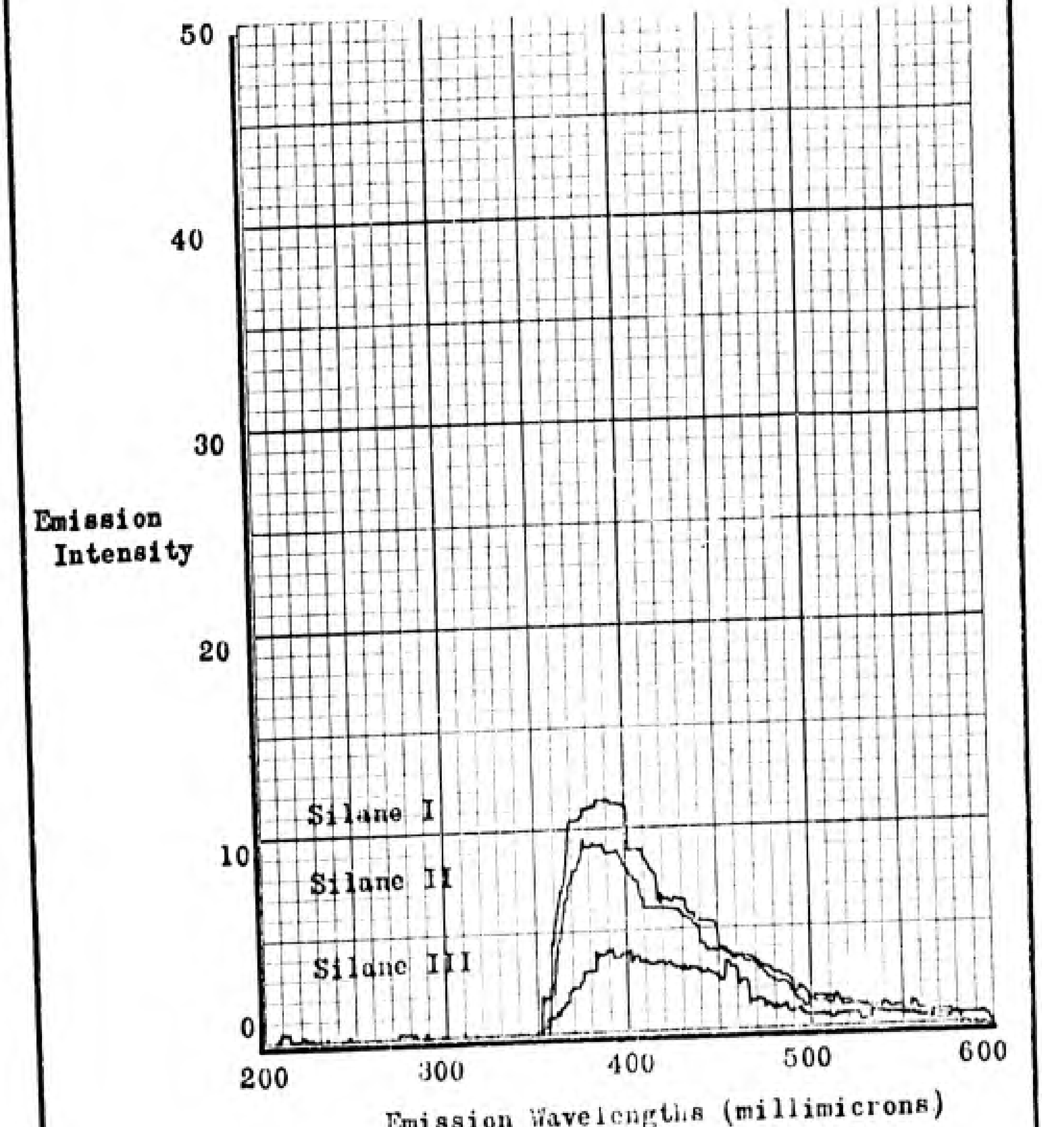

Figure 20

Fluorescence Spectrum of Silane I, II and III.

Beta Excitation With Fixed Geometry

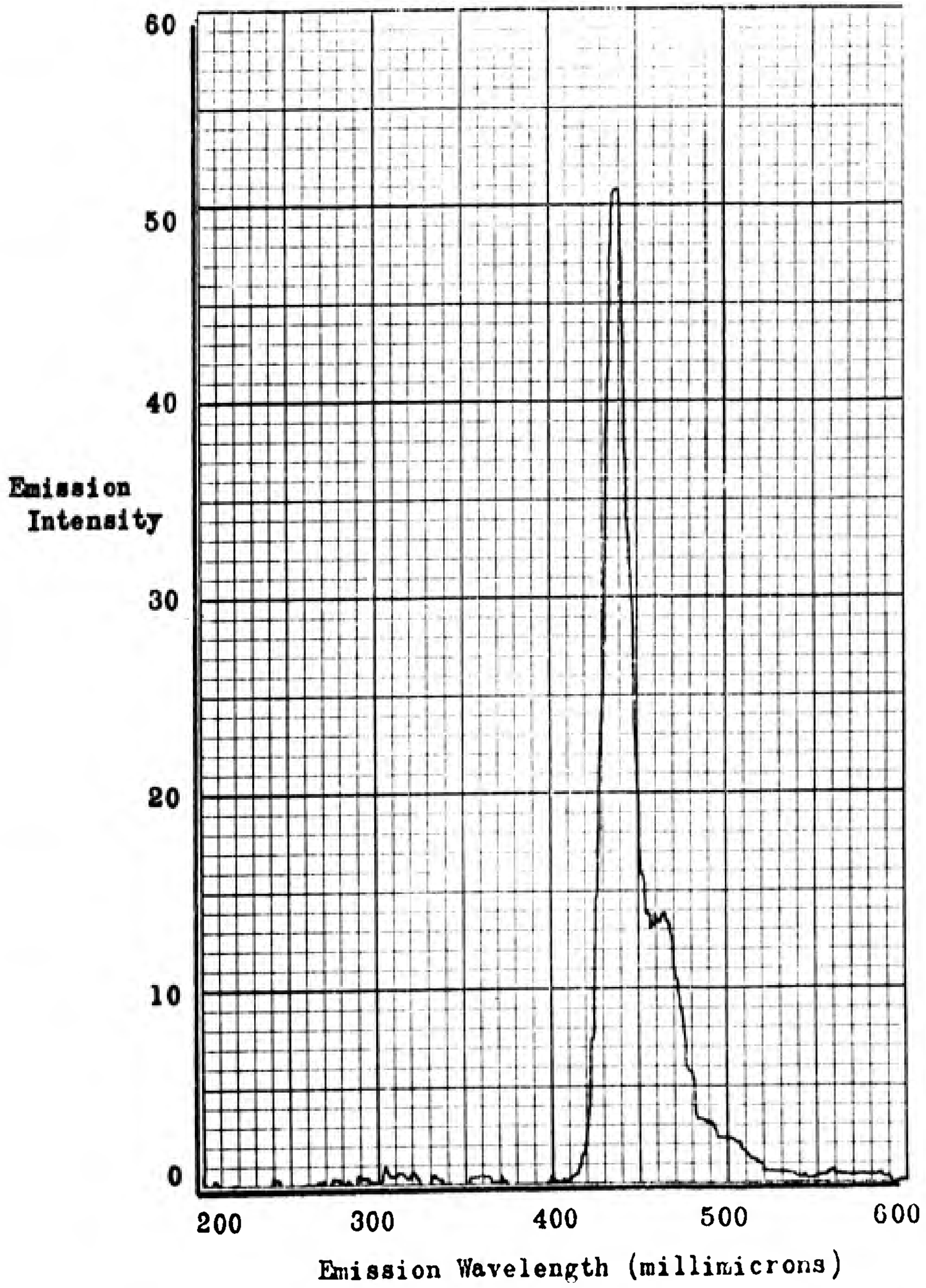

Figure 21

Fluorescence Spectrum of Anthracene

Beta Excitation

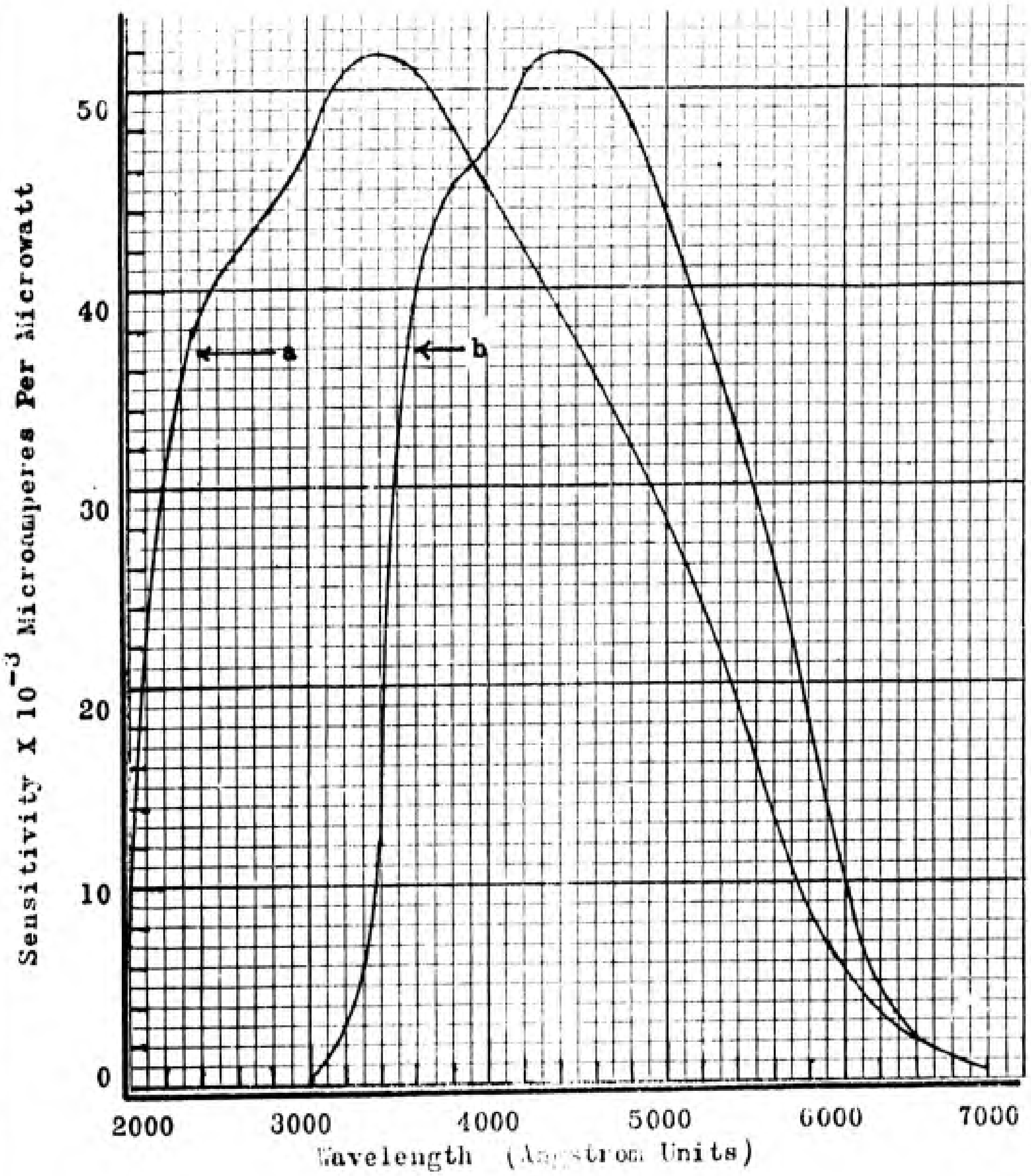

Figure 22

Response of Photomultiplier Tubes
a) S-5 (1P28)
b) S-11 (6292)

(Adapted from Ref. 12)

Vita

Carl Graham Weis [redacted] [redacted], the son of Earl Louis Weis and Minerva Crozier Weis. After graduation in 1949 from Central High School, Madison, Indiana, he enrolled in Purdue University, West Lafayette, Indiana. In May 1953 he was graduated from Purdue with the degree of Bachelor of Science in Chemical Engineering. After receiving his commission as Second Lieutenant in the USAF Reserve, he entered active duty in March 1954. His military assignments prior to coming to the Institute of Technology were as Personnel and Administrative Officer in various Ammunition Supply Squadrons, both at Hill AFB, Utah and the USAF Leghorn Ammunition Storage Area in Italy.

[redacted]

This thesis was typed by Mrs. Gail B. Prohaska