



ZEEMAN EFFECT IN PROMETHIUM-147

by

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Title: ZEEMAN EFFECT IN PROMETHIUM-147

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ABSTRACT

The Zeeman Effect has been observed in the spectrum of Pm-147. Zeeman effect data have been determined for twenty-six Pm II patterns and for nine unresolved triplets. No Pm I patterns were observed.

A method is described for making electrodeless gas discharge tubes from radioactive rare earth chlorides.

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TABLE OF CONTENTS

| | <u>Page</u> |
|---|-------------|
| ABSTRACT | 2 |
| INTRODUCTION | 4 |
| I. APPLICATION OF THE ZEEMAN EFFECT TO THE CLASSIFICATION OF COMPLEX SPECTRA | 6 |
| II. EXPERIMENTAL PROCEDURE | 9 |
| 1. The problem posed by radioactivity. | 9 |
| Figure 1. Decay Scheme of Pm ¹⁴⁷ | 10 |
| 2. The light source. | 11 |
| 3. Preliminary tests. | 13 |
| 4. The first promethium discharge tubes. | 14 |
| 5. Construction of the light sources. | 15 |
| Figure 2. Ion Exchange and Transfer Apparatus | 17 |
| Figure 3. The Reaction Oven | 19 |
| Figure 4. The Vacuum System | 20 |
| Figure 5. Vacuum System Arrangement | 22 |
| 6. Use of the discharge tubes. | 23 |
| Figure 6. Light Source Arrangement | 24 |
| 7. The spectrograms obtained. | 26 |
| Figure 7. A Portion of the Zeeman Spectrum. | 28 |
| Table I. Zeeman Data | 29 |
| III. RESULTS | 32 |
| ACKNOWLEDGEMENTS | 34 |
| BIBLIOGRAPHY | 35 |

INTRODUCTION

Promethium was first isolated in 1946 by Marinsky, Glendenin, and Coryell.¹ They chemically identified it as a rare earth and claimed that it was the long-sought element of atomic number 61. Examinations of the X-ray spectra^{2,3} and the emission^{4,5} and absorption⁶ spectra gave further evidence that a new element had been discovered.

A thorough investigation of the absorption and emission spectra of a sample of the material was conducted in 1951 by Meggers, Scribner, and Bozman.⁷ They confirmed that the material was indeed a new element and compiled a list of the wavelengths and intensities of 2,200 new spectral lines. However, possibly because of the large number of chemical impurities contained in their sample, the lines had the same relative intensities whether arc or spark excitation was used. Thus there was nothing to

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1. J. A. Marinsky, L. E. Glendenin, and C. D. Coryell, *J. Am. Chem. Soc.* 69, 2781 (1947)
 2. L. E. Burkhart, W. F. Peed, and E. J. Spitzer, *Phys. Rev.* 75, 86 (1949)
 3. W. F. Peed, E. J. Spitzer, and L. E. Burkhart, *Phys. Rev.* 76, 143 (1949)
 4. C. Feldman, *J. Am. Chem. Soc.* 71, 3841 (1949)
 5. D. L. Timma, *JOSA* 39, 898 (1949)
 6. G. W. Parker and P. M. Lantz, *J. Am. Chem. Soc.* 72, 2834 (1950)
 7. W. F. Meggers, E. F. Scribner, and W. R. Bozman, *J. Research, NBS* 46, 85 (1951)

indicate which lines were from neutral and which from ionized atoms.

There has been great emphasis in recent years on the investigation of complex rare earth spectra. However, except for the original work by Meggers, Scribner, and Bozman and a determination of the spin of Pm^{147} by Klinkenberg and Tomkins⁸, the investigation has not been extended to promethium.

The emission spectrum of promethium in high magnetic fields was observed for the first time in connection with this thesis. It is hoped that the information provided herein will form the basis for a classification of the spectrum of the element.

8. P. F. A. Klinkenberg and F. S. Tomkins, *Physica* 26, 103 (1960)

I. APPLICATION OF THE ZEEMAN EFFECT TO THE CLASSIFICATION OF COMPLEX SPECTRA

The emission spectrum of an atom arises from transitions from a state of higher energy to one of lower energy with the simultaneous emission of a photon whose energy is exactly equal to the energy difference between the two states. Each state is characterized by certain quantum numbers, including J , the total angular momentum, and g , the Landé g -factor. Transitions can occur between practically any energy levels consistent with the selection rules

$$\Delta J = 0, \pm 1 \quad (J=0 \rightarrow J=0 \text{ excluded}).$$

In complex spectra, such as those of rare earths, there are thousands of allowed transitions. A classification of the energy levels of such a spectrum simply by searching for combinations of wave numbers of the spectral lines is practically impossible to achieve, particularly when the wave numbers are not known very accurately. It is necessary to obtain additional information about the spectrum before a classification can be begun.

A common method of obtaining more information about the energy levels of an atom is to take advantage

of the action of a magnetic field on the spectrum. When an atom is placed in a magnetic field, each energy level splits up into $2J+1$ components. Each component differs in energy from the energy of the original level by

$$\Delta E = MgL$$

where g is the Landé g -factor, L is a term proportional to the strength of the magnetic field, and M is the projection of J on the direction of the field and is given by

$$M = 0, \underline{+1}, \underline{+2}, \dots, \underline{+J} \text{ for } J \text{ integral,}$$

$$M = \underline{+1/2}, \underline{+3/2}, \dots, \underline{+J} \text{ for } J \text{ half-integral.}$$

Transitions can occur between Zeeman levels according to the zero-field selection rules and the additional rules

$$M = 0, \underline{+1} \quad (M=0 \rightarrow M=0 \text{ excluded for } J \rightarrow J \text{ transitions}).$$

The spectral lines now appear as symmetrical patterns with intensities given by the following formulas.⁹

$J \rightarrow J$ transitions:

$$M \rightarrow \underline{M+1}, \quad I = A(J \underline{+M+1})(J \underline{+M});$$

$$M \rightarrow M, \quad I = 4AM^2;$$

$J \rightarrow J+1$ transitions:

$$M \rightarrow \underline{M+1}, \quad I = B(J \underline{+M+1})(J \underline{+M+2});$$

$$M \rightarrow M, \quad I = 4B(J \underline{+M+1})(J \underline{-M+1}).$$

9. H. E. White, Introduction to Atomic Spectra, p. 221, McGraw-Hill, New York (1934)

The constants A and B are merely normalization constants.

By knowing the strength of the magnetic field applied, therefore, one can determine the J's and g's of both levels involved in a completely resolved Zeeman pattern. This reduces enormously the number of random coincidences in a search for wave number combinations and significantly aids in the classification of the spectrum.

II EXPERIMENTAL PROCEDURE

1. The problem posed by radioactivity:

Radioactivity presents a rather serious handicap in the investigation of atomic spectra in that it limits the amount of material which may be used, either by constituting a radiation hazard or because the method used for the production of the isotope is not applicable to the production of large quantities of material, or for both reasons. This is particularly troublesome in the case of the rare earths because it is difficult to obtain suitable light sources from small samples. The problem is reduced somewhat in the case of promethium, for while there are no stable or long-lived isotopes of the element, there is one isotope which is readily obtainable from uranium fission products and which presents a comparatively small radiation hazard.

The isotope in question is the 2.6-year Pm^{147} . It decays by low energy beta emission to samarium-147, which has a half-life on the order of 10^{11} years. The decay scheme is shown in Fig. 1.¹⁰

10. D. Strominger, J. M. Hollander, and G. T. Seaborg, Rev. Mod. Phys. 30, 585 (1958)

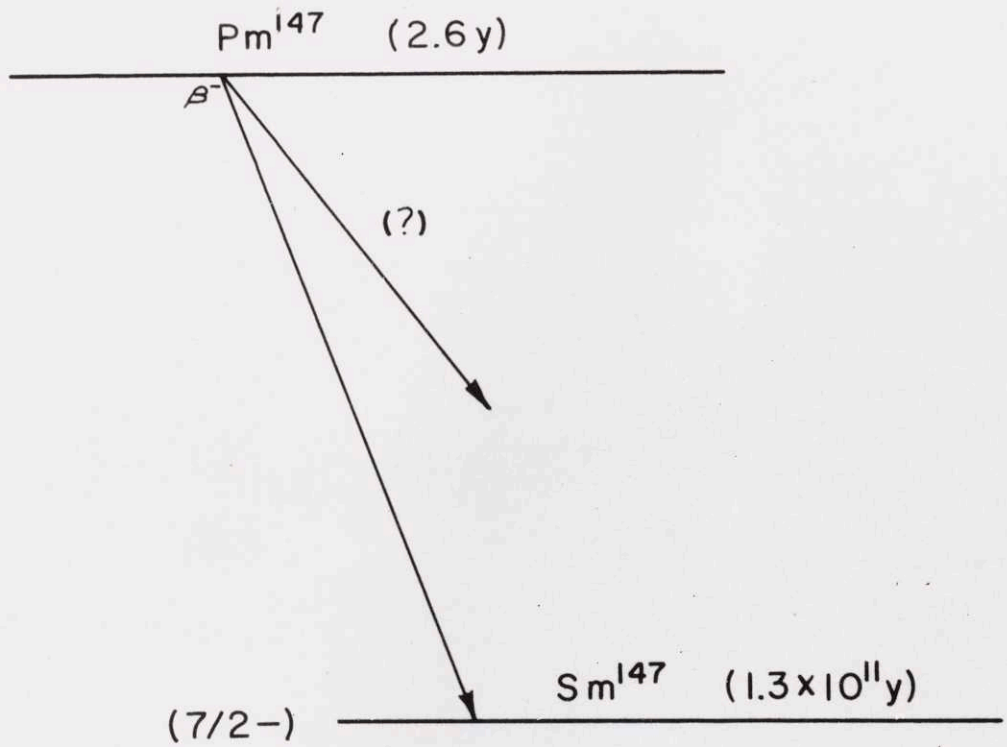


FIGURE 1
DECAY SCHEME OF Pm^{147}

Since Pm^{147} decays with the emission of only low energy beta rays, there is little external radiation hazard as long as the material is kept in a closed container. If very large quantities are used, there is, of course, a small problem presented by the low energy bremsstrahlung. The daughter product in the decay has such a long half-life that it is negligible as a radiation hazard. The chief concern in handling Pm^{147} is the prevention of radioactive contamination and of ingestion. It is desirable that the light source used in an investigation of the promethium spectrum be sealed.

2. The light source.

The type of light source which has been found to be most suitable for use in Zeeman studies of rare earths is the electrodeless discharge tube. This type of light source also satisfies the requirement for a sealed source and does not entail serious handling problems in its fabrication. It was, therefore, the first choice for use in the proposed Zeeman study of promethium. The problem which presented itself was how best to produce the light source.

Electrodeless discharge tubes have been made from

several rare earths, including promethium-147, by Tomkins and Fred.¹¹ These tubes were made from rare earth iodides which were in turn produced by a reaction of aluminum iodide with the rare earth oxide formed by igniting the rare earth over a micro-burner. However, the facilities available at MIT for handling radioactive materials required that a different method be used.

The material used for the promethium discharge tubes made at MIT was the commercial product sold by the Oak Ridge National Laboratory. It is in the form of PmCl_3 in a solution of HCl. Once the hydrated chloride has been formed, the simplest method of producing a material suitable for use in the electrodeless discharge tubes is the conversion of the hydrated chloride into the anhydrous form by evaporation in the presence of dry HCl gas.¹² With large amounts of material this is a relatively simple process, although it is necessary to control the temperature rather carefully to prevent conversion into the oxychloride. But special techniques had to be developed to adapt the process for use with a small sample of material.

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11. F. S. Tomkins and M. Fred, *JOSA* 47, 1087 (1957)
12. J. H. Kleinheksel and H. C. Kremers, *J. Am. Chem. Soc.* 50, 959 (1928)

3. Preliminary tests.

Preliminary tests were made using holmium and neodymium to get an indication of the minimum amount of material from which a successful discharge tube could be made. It had to be taken into account that the conversion of the chloride to the anhydrous form was never 100% efficient and that reconversion to the hydrated form occurs rapidly in ordinary atmosphere. This is especially true when the chloride is merely a thin coating on the walls of a reaction tube. A maximum limit was set for the entire process, including all losses of material while making the discharge tube, by the fact that MIT's AEC license restricts the total amount of Pm^{147} which may be possessed at one time to one curie (about one milligram).

The most significant difficulty encountered in the preliminary tests was the fact that discharge tubes containing rare earth halides must be operated at a temperature of approximately 800°C. Apart from the necessity for extreme care in eliminating impurities which might vaporize more easily than the rare earth halide, it was necessary to find a method for establishing these high temperatures. No method could be

found for applying external heat to the discharge tubes while they were being operated in the high fields and confined space of the magnet used. The heat required was derived from a magnetron power supply and wave guide which heated the tube by highly exciting the discharge. However, the heating was not uniform, and the halide condensed on cooler parts of the lamp while other parts of the tube were heated almost to the melting point of quartz. Primarily for this reason, no successful lamps were obtained from less than a few tenths of a milligram.

It was found that the uneven heating of the discharge tubes necessitated the frequent reversal, end for end, of the lamps during operation. This meant that a tube had to be lit several times during an exposure. Tests were made using various pressures of both neon and argon as filler gases to determine which gas and which pressure produced the most reliable discharge tubes. The tests showed that a pressure of 1.5 mm Hg of argon produced light sources that were quite intense and could be started most easily.

4. The first promethium discharge tubes.

A sample of 900 mc was ordered for a first attempt

at obtaining Zeeman spectrograms. With this sample three discharge tubes were made singly. The first yielded a zero-field spectrogram in the region $\lambda 2500$ - $\lambda 3750$ A with external heat applied to the tube during the exposure. This spectrogram showed that the sample contained a large number of chemical impurities of which the most troublesome was iron. Unfortunately, because of the long exposures necessary in Zeeman work with rare earths, the discharge tubes did not have long enough lifetimes to obtain satisfactorily exposed spectrograms. However, they served to show that some purification had to be performed before successful discharge tubes could be obtained and that more than one discharge tube was necessary for each exposure.

5. Construction of the light sources.

The final form of the procedure for handling the radioactive material should be of interest not only for its application to promethium but also because it can be used equally well for other first and second series rare earth elements. Therefore the procedure is presented below in considerable detail.

All handling of the radioactive material through the completion of the conversion to the anhydrous chloride

was done in a radioactive chemical hood in the Central Radioisotope Laboratory and Storage Facility of MIT.

Fig. 2. shows the apparatus used in the transfer and purification of the sample. An ion exchange column was used to remove the iron impurity. After preparation of the ion exchange column the promethium solution was transferred to the separatory funnel, situated above the exchange column, with a transfer pipette. Concentrated hydrochloric acid was added to the solution to make the acidity 8M, and a drop of liquid bromine was added to insure that the iron to be removed was in the proper valence state. The solution was then admitted to the reservoir of the ion exchange column and allowed to pass through the column at a rate of about $\frac{1}{2}$ ml/min. A wash solution of 20 ml 8M HCl was used to recover as much promethium as possible, and the contaminated transfer pipette, separatory funnel, and ion exchange column were disposed of.

At this point the radioactive material was contained in the reaction tube in an acid solution of about 25 ml volume. The reaction tube was then clamped over a steam bath, and the solution was evaporated to a moist dryness while dry nitrogen and HCl gas flowed through the tube.

When no liquid remained in the reaction tube, the tube was transferred to a specially built, thermostatically-

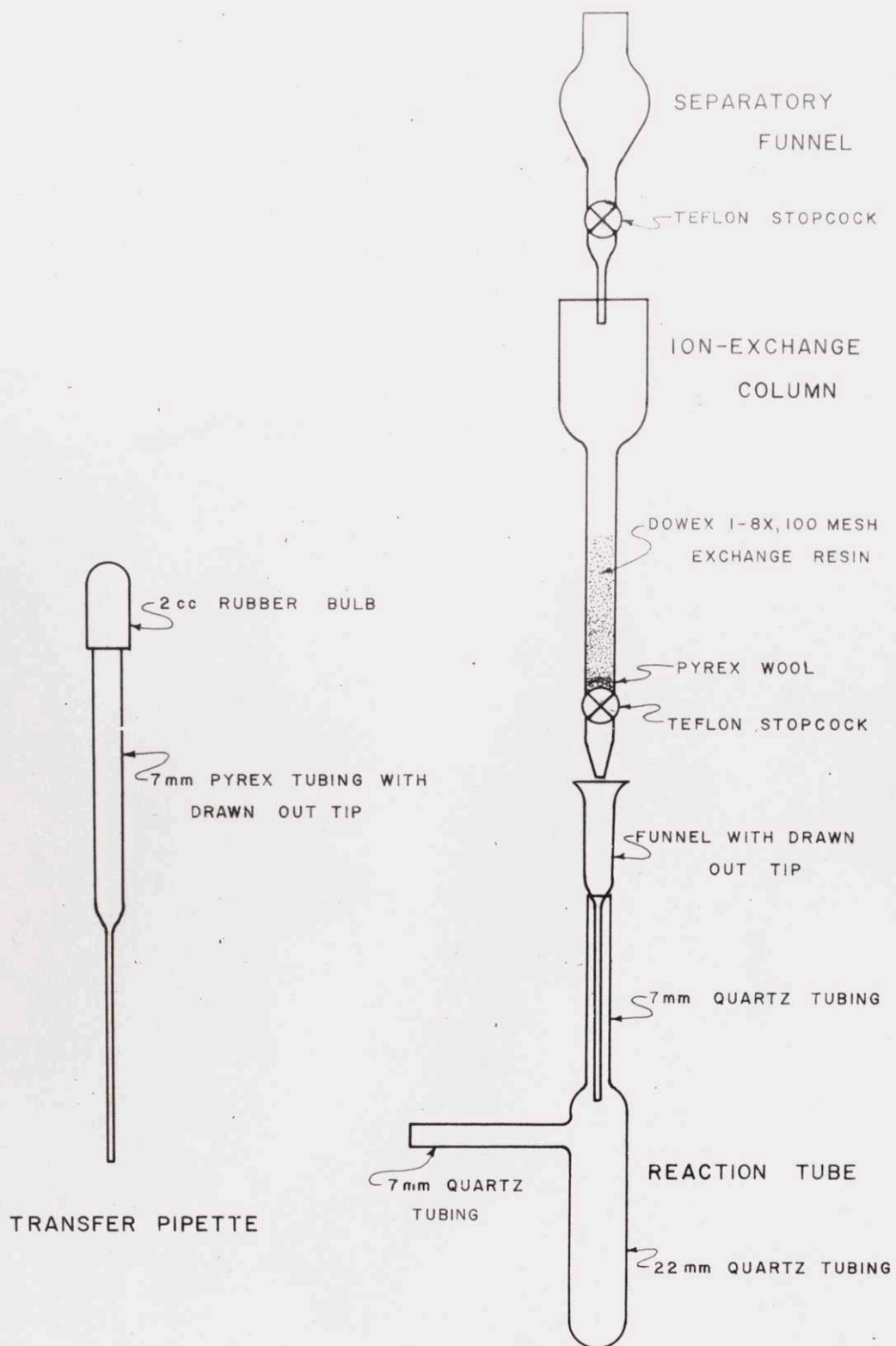


FIGURE 2

ION-EXCHANGE AND TRANSFER APPARATUS

controlled oven shown in Fig. 3. The temperature of the oven was raised from 100°C to 200°C at a rate of about 15°/half hour while dry HCl gas flowed through the tube. The temperature was then raised to 250°C for two hours to complete the conversion to the anhydrous chloride.

After the chemical conversion was completed, the side extension was sealed off from the reaction tube, and the reaction tube was sealed onto a vacuum system and evacuated. This step of the process had to be carried out as quickly as possible to prevent reconversion to the hydrated chloride while the tube was exposed to the atmosphere at room temperature.

The vacuum system on which the discharge tubes were made was situated in a ventilated enclosure, as shown in Fig. 4, as a precaution against possible failure of the glassware. There was no problem of radioactive material being pumped through the system because of the exceedingly low vapor pressure of PmCl_3 at room temperature. The system was equipped with a large oven used to outgas all glassware above the transite table at 450°C. Outgassing was performed in this manner for several hours under high vacuum both before and after the reaction tube was sealed onto the system. This eliminated all traces

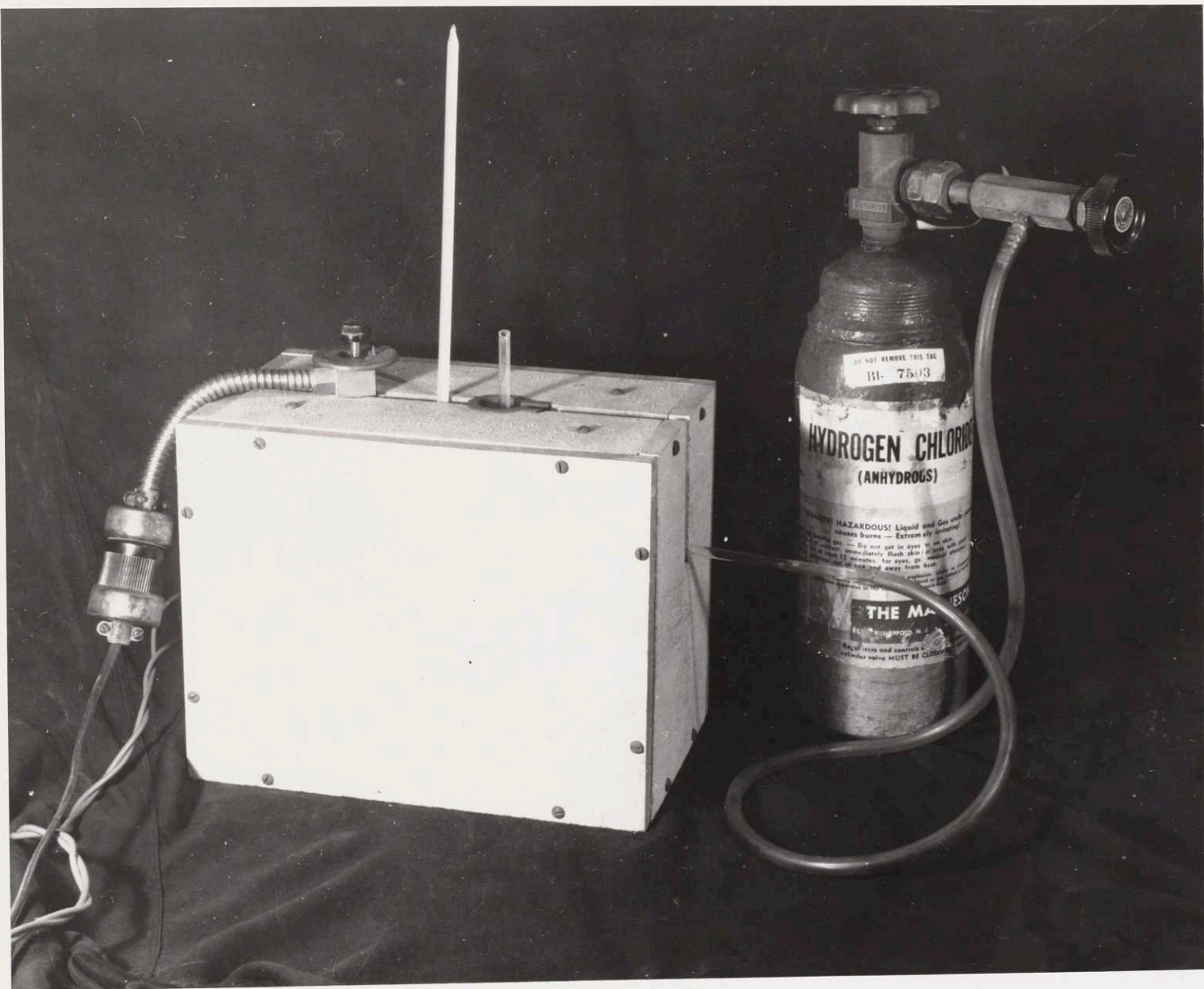


Figure 3.
The Reaction Oven

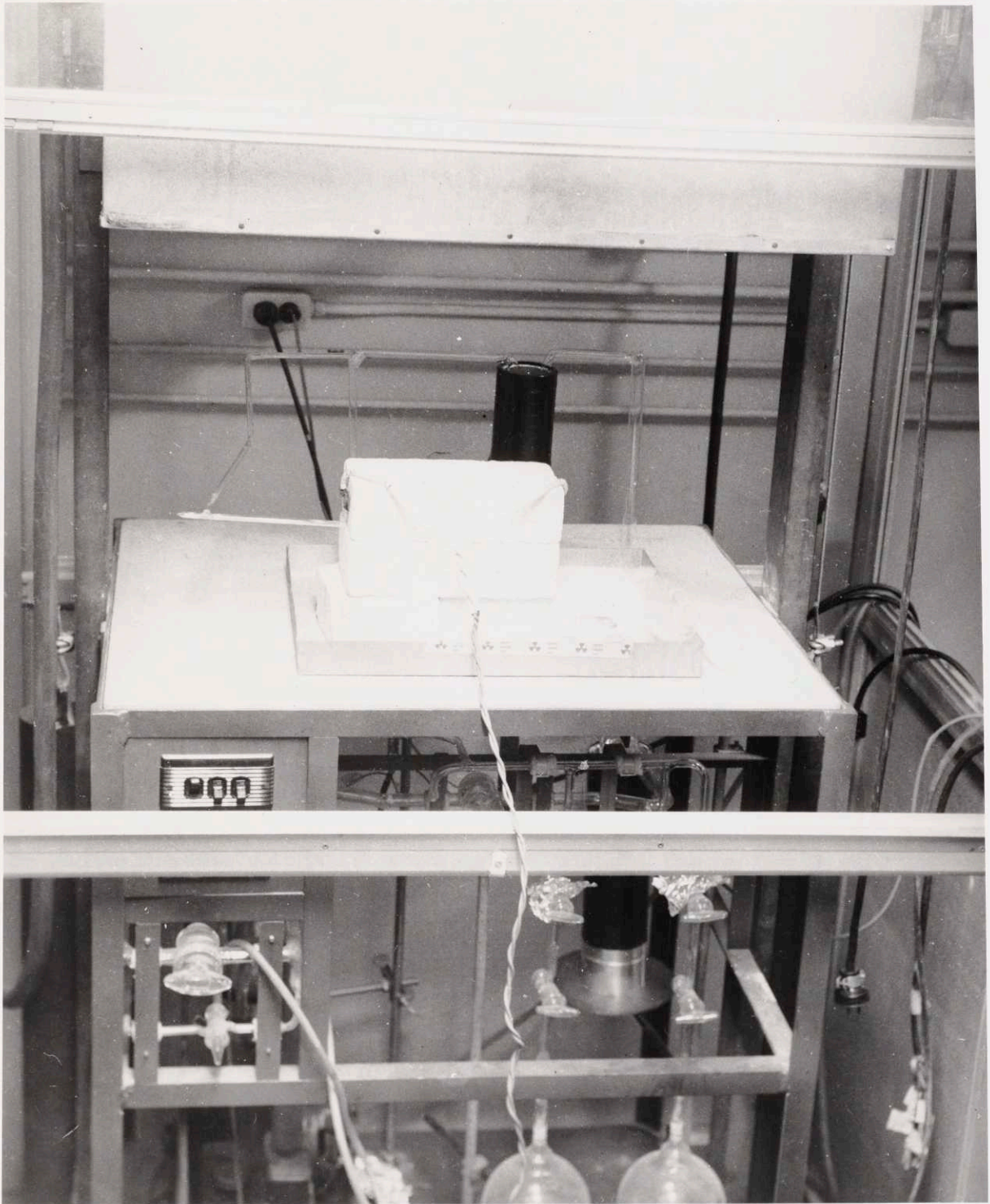


Figure 4
The Vacuum System

of diffusion pump mercury from the discharge tubes.

In addition to outgassing with the oven mentioned, a small electric furnace was used to outgas the discharge tube blanks at temperatures in excess of 1000°C. This was done for at least three hours, or until the ionization gauge, with which low pressures were measured, had been in equilibrium for at least a half hour. The tube furnace was then placed over the reaction tube, and the PmCl_3 was sublimed into the discharge tube blanks, which were arranged in a cluster as shown in Fig. 5. The reaction tube was then removed from the system by sealing it off at the constriction.

During the first outgassing the more volatile impurities were removed from the sample. The outgassing of the tube blanks with the small furnace removed almost all impurities adhering to the inside of the blanks, and impurities less volatile than PmCl_3 were left behind in the reaction tube after the sublimation process. In this way a further purification of the sample was effected. However, the sample naturally contained samarium into which promethium decays, and several other impurities, such as europium, neodymium, calcium, sodium, magnesium, and traces of iron which survived the ion exchange purification, were found in the spectrum. These interfered

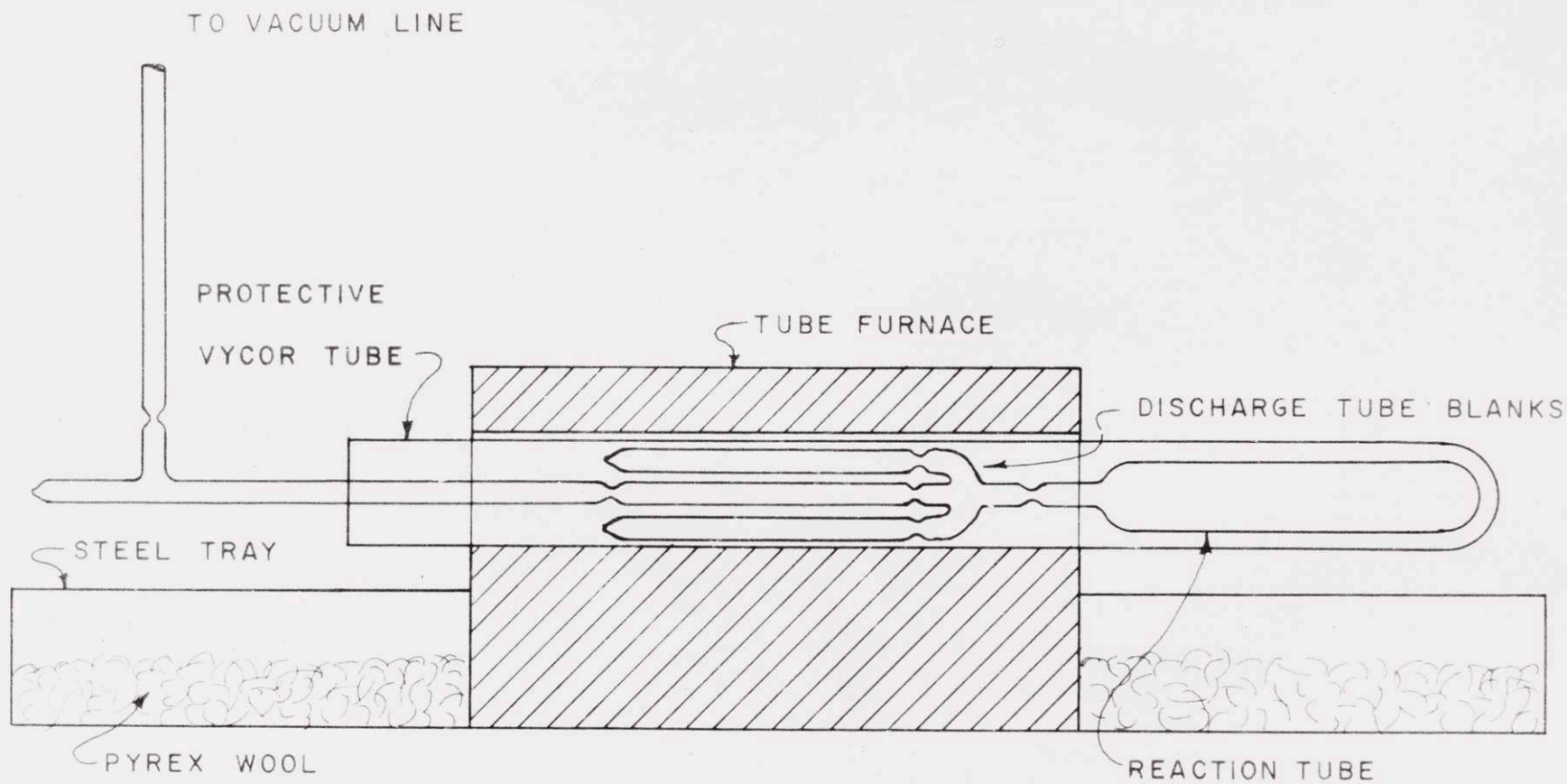


FIGURE 5

VACUUM SYSTEM ARRANGEMENT

greatly with the reduction of the already complex promethium Zeeman spectrum.

After the reaction tube had been removed from the system and disposed of, the system was pumped down to the lowest obtainable pressure, less than 10^{-6} mm Hg, and the tubes were filled to a pressure of 1.5 mm Hg with argon gas and removed from the system.

6. Use of the discharge tubes.

Fig. 6. shows the arrangement of a discharge tube as it was used in the magnet. The quartz jacket served to make the heating more uniform, to prevent the discharge tube from vibrating out of the wave guide, and to help confine the activity in case of failure of the quartz tube. An inner liner was used in the magnet to further protect it in case of lamp failure.

The magnetic field for the Zeeman study was produced by a Bitter solenoid.¹³ Although the magnet is designed to produce fields in the neighborhood of 100,000 gauss, it was anticipated that the exposures would have to be several hours in length, and to prevent overheating of the magnet, it was operated at only $4/5$ maximum current.

13. F. Bitter, Rev. Sci. Instr. 10, 373 (1939)

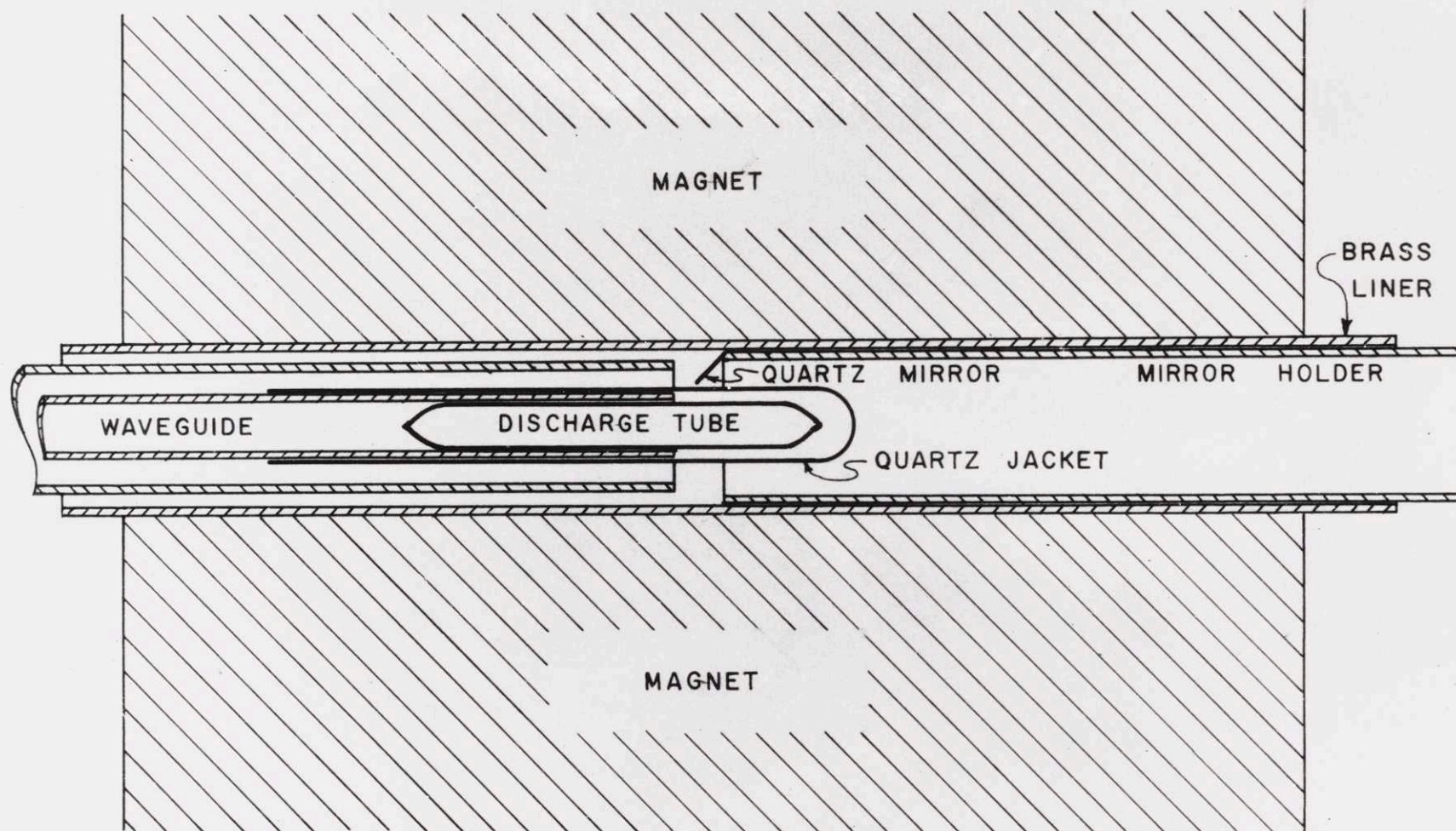


FIGURE 6
LIGHT SOURCE ARRANGEMENT

Under these conditions a field of 67,410 gauss was obtained with no difficulty.

To record the spectrum the new MIT echelle spectrograph system was used. When completed this system will consist of two separate spectrographs similar to the one previously used in the MIT Spectroscopy Laboratory.¹⁴ Each spectrograph will include a 10-inch echelle ruled on the interferometrically-controlled MIT ruling engine.¹⁵ One spectrograph will cover the region $\lambda 2350$ - $\lambda 3750$ A, and the other will cover the region $\lambda 3750$ - $\lambda 6500$ A. The system is not yet perfected because of the inability to obtain echelle blanks which are flat to within two or three fringes. The echelles used were capable of resolution greater than that permitted by doppler width of the source and hyperfine structure within the Zeeman patterns. The main disadvantage was astigmatism caused by the curvature of the echelle blanks. This reduced the intensity of the lines, but a worse aspect was the fact that it prevented proper calibration of the plates. The fiducial line usually used with this type of spectrograph focused several inches in front of the plate holder,

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14. G. R. Harrison, J. E. Archer, and J. Camus, JOSA 42, 706 (1949)
15. G. R. Harrison, N. Sturgis, S. C. Baker, and G. W. Stroke, JOSA 47, 15 (1957)

and no calibration spectrum could be photographed beside the promethium spectrum without overlapping of the two spectra.

7. The spectrograms obtained.

The first spectrogram taken was of the p-components in the region $\lambda 2350$ - $\lambda 3750$ A. The exposure time was over three hours, but only a few orders in the region $\lambda 3600$ - $\lambda 3750$ A were sufficiently exposed. The spectrograph for the visible region had not yet been set up, because an echelle of the proper spacing had not been ruled. The first spectrogram, however, made it evident that an echelle had to be set up for the visible region, even though the orders would be so long that the entire region could not be photographed with one setting of the echelle. This was done before more exposures were made.

The second set of exposures was made using both spectrographs. A Rochon prism was used to separate the p- and s-components. The s-components were photographed on the ultra-violet spectrograph, and the p-components and a portion of the s-components were photographed in the visible region. At the end of three hours all the discharge tubes had failed, but during that time

spectrograms were obtained showing the stronger lines of most of the region $\lambda 3600$ - $\lambda 5000$.

An enlarged portion of the promethium Zeeman spectrum is shown in Fig. 7. The very strong pattern is the calcium line $\lambda 3993.666$. This picture makes obvious the principal reasons why an extensive list of J and g values could not be compiled for Pm¹⁴⁷. Many of the patterns which are resolved in the center are unresolved farther from the center because of broadening of the components by hyperfine structure. Of the five most intense lines listed by Meggers, Scribner, and Bozman, only two could be sufficiently resolved. Many other lines of less intensity were similarly unresolved. Most of the resolved patterns shown in the spectrum are from impurities.

Numerous other patterns were unresolved because the p- and s-components overlapped in such a way that neither could be resolved. Others were blotted out by patterns from other promethium and impurity lines.

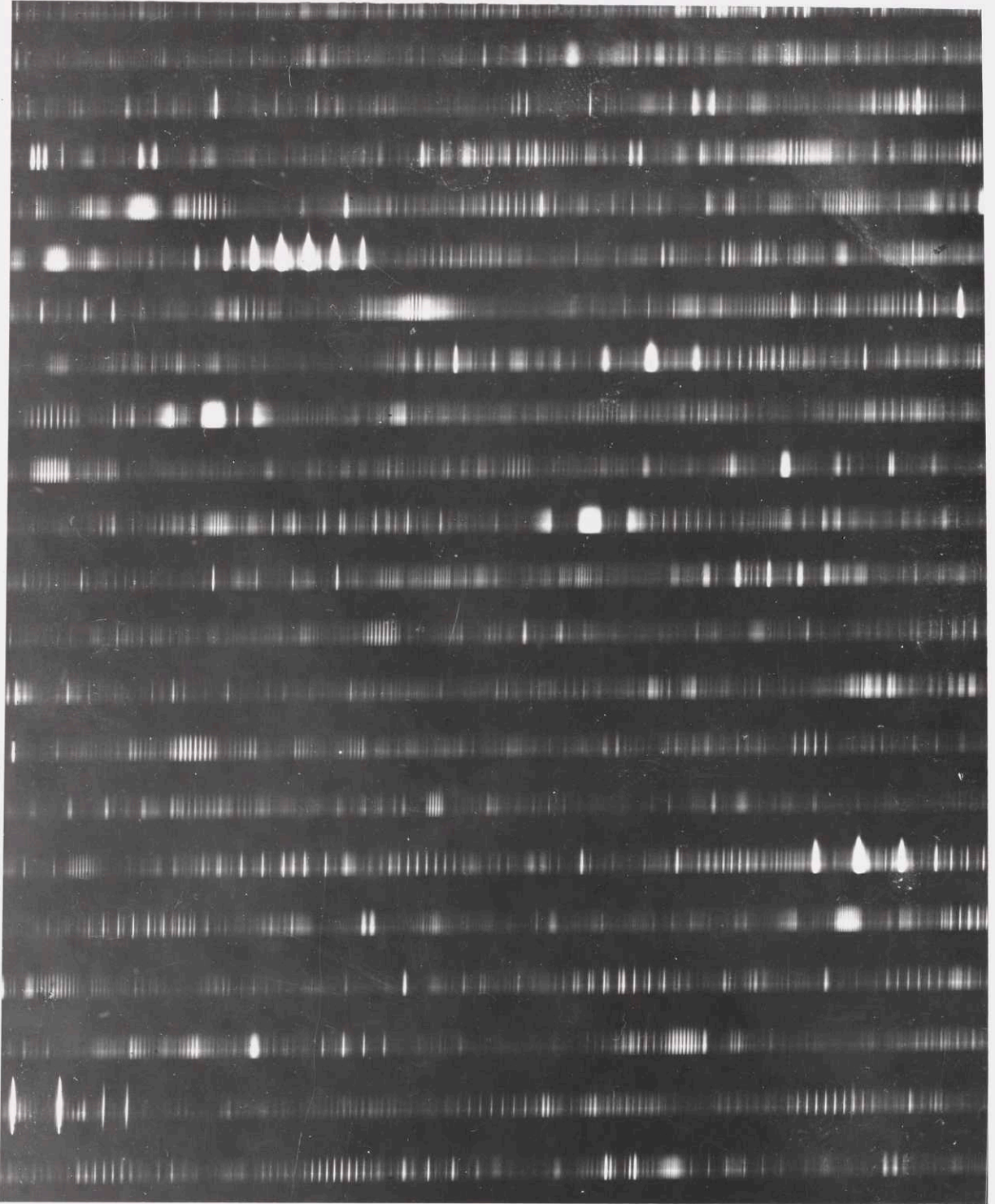


Figure 7

A Portion of the Zeeman Spectrum

Zeeman Data

Table I.

| Wavelength | Type | J_1 | J_2 | g_1 | g_2 | σ_{g_1} | σ_{g_2} | Δg |
|------------|------|-------|-------|--------|--------|----------------|----------------|------------|
| 4477.46 | | 3 | 2 | | | | | 0.157 |
| 4473.23 | 2 | 5 | 4 | 1.1102 | 0.9204 | 0.0036 | 0.0040 | |
| 4445.41 | 7b | | | 1.398 | 1.398 | | | |
| 4443.67 | 7b | | | 1.104 | 1.104 | | | |
| 4432.51 | 2 | 4 | 3 | 0.8570 | 0.5342 | 0.0007 | 0.0008 | |
| 4417.98 | 2 | 4 | 3 | 1.1025 | 0.9009 | 0.0026 | 0.0031 | |
| 4388.76 | | 4 | 3 | | | | | 0.201 |
| 4342.11 | 3 | 4 | 4 | | | | | 0.181 |
| 4336.53 | 2 | 3 | 2 | 0.7251 | 0.4643 | 0.0013 | 0.0017 | |
| 4216.30 | 2 | 4 | 3 | 0.8543 | 0.7256 | 0.0004 | 0.0005 | |
| 4185.74 | 3 | 6 | 6 | | | | | 0.178 |
| 4156.07 | 7b | | | 1.072 | 1.072 | | | |
| 4142.87 | 3 | 4 | 4 | | | | | 0.125 |

Table I. (continued)

| Wavelength | Type | J_1 | J_2 | g_1 | g_2 | σ_{g_1} | σ_{g_2} | Δg |
|------------|------|-------|-------|--------|--------|----------------|----------------|------------|
| 4139.72 | 3 | 5 | 5 | | | | | 0.246 |
| 4086.10 | 2 | 3 | 2 | 0.7434 | 0.0000 | 0.0010 | 0.0011 | |
| 4075.85 | | 3 | 2 | | | | | 0.549 |
| 4055.20 | 7b | | | 0.768 | 0.768 | | | |
| 4040.74 | 2 | 6 | 5 | 1.2651 | 1.1846 | 0.0012 | 0.0013 | |
| 4028.20 | 2 | 4 | 3 | 0.9100 | 0.7247 | 0.0012 | 0.0015 | |
| 3998.96 | 1 | 5 | 4 | 0.9431 | 1.0368 | 0.0017 | 0.0023 | |
| 3995.05 | 7b | | | 1.370 | 1.370 | | | |
| 3983.10 | 7b | | | 0.921 | 0.921 | | | |
| 3980.73 | 7b | | | 1.109 | 1.109 | | | |
| 3947.22 | | 3 | 2 | | | | | 0.235 |
| 3921.80 | | 3 | 2 | | | | | 0.302 |
| 3909.51 | 1 | 3 | 2 | 0.7307 | 0.8608 | 0.0005 | 0.0007 | |

Table I. (continued)

| Wavelength | Type | J_1 | J_2 | g_1 | g_2 | σ_{g_1} | σ_{g_2} |
|------------|------|-------|-------|----------|----------|----------------|----------------|
| 3892.16 | 1 | (5) | (4) | (1.2290) | (1.3650) | 0.0021 | 0.0024 |
| 3798.17 | 3 | 2 | 2 | 0.7139 | 0.0000 | 0.0003 | 0.0004 |
| 3747.10 | 3 | 3 | 3 | 0.7323 | 1.0569 | 0.0011 | 0.0011 |
| 3742.51 | 3 | 6 | 6 | (0.8744) | (1.0383) | 0.0009 | 0.0009 |
| 3734.31 | 2 | 3 | 2 | 0.6223 | 0.0000 | 0.0018 | 0.0021 |
| 3718.88 | 3 | 6 | 6 | (1.4242) | (1.5508) | 0.0010 | 0.0010 |
| 3702.64 | 3 | 4 | 4 | 1.2621 | 1.4850 | 0.0012 | 0.0012 |
| 3691.70 | 7b | | | 0.810 | 0.810 | | |
| 3689.78 | 3 | 2 | 2 | 0.2224 | 0.0000 | 0.0002 | 0.0002 |

III RESULTS

The Zeeman effect data obtained is presented in Table I. The types of patterns listed refer to the Meggers convention.¹⁶ The g-values for the patterns which showed s-components of sufficient intensity were calculated by a least squares method according to Vander Sluis.¹⁷ The values are listed in the table to the fourth decimal place, along with their standard deviations. For patterns which showed p-components but no s-components the differences between the g-values involved are given. These were calculated by averaging the plate measurements weighted according to the M-values of the components. These values are listed in the table to the third decimal place. The only unresolved patterns which were measured were the triplets where one can assume reasonable accuracy in the determination of the g-values. All values were calculated using a field strength of 67,410 gauss obtained from measurements of the patterns of the mercury lines $\lambda 2536.5063$ and $\lambda 4046.5712$. All of the resolved patterns listed belong to the Pm II spectrum. No Pm I lines, identified by their even multiplicity, could definitely be identified.

16. G. R. Harrison, W. E. Albertson, and N. E. Hosford, JOSA 31, 439 (1941)

17. K. L. Vander Sluis, JOSA 46, 605 (1956)

The electronic configuration predicted for the ground state of the ionized promethium atom is $4f^5 6s$. Hund's rule predicts that this configuration will have a ground state term of 7H_2 . The J's and g's for the ground state multiplet of the configuration, assuming LS coupling, are¹⁸

| J | g |
|---|--------|
| 8 | 1.375 |
| 7 | 1.339 |
| 6 | 1.286 |
| 5 | 1.200 |
| 4 | 1.050 |
| 3 | 0.750 |
| 2 | 0.000. |

While it is impossible to to predict with any degree of certainty any of the energy levels simply by examining the values listed in Table I, it seems probable that at least the two lower levels of the predicted multiplet are verified. It is hoped that a thorough investigation of wave number differences, aided by the measured values of the table, will establish at least a few of the lower levels in a classification of the spectrum of promethium.

18. C. E. Moore, Atomic Energy Levels, p. xx, NBS Circular 467 (1949)

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