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ABSTRACT

Experimental techniques are taught in a laboratory course designed with some student options available. Eight experiments which use vacuum systems, radiation sources, dispersion and detection systems are outlined. A course outline and time table are given. The final examination is described as 30 minutes of individual practical work and dialogue with the instructor. An itemized list of equipment costs is also included. This project was supported by a National Science Foundation grant. (Author/TS)

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An Undergraduate Vacuum Ultraviolet Spectroscopy Laboratory at Georgia Tech*

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A laboratory is described which provides the student with a transitional experience between the more structured laboratories in introductory courses and the open-ended individual special problems. Vacuum ultraviolet spectroscopy has been chosen as the area of major emphasis as it provides an excellent vehicle for the student to learn a number of valuable experimental techniques. Experiments are outlined which use vacuum systems, radiation sources, dispersion and detection systems to provide challenging laboratory experiences.

INTRODUCTION

One of the dangers of "mass-produced" laboratory experience via highly structured introductory laboratories is to divorce the student from exposure to techniques. Frequently these well organized laboratories can result in the development of student

012 441

inhibitions toward experimental physics for the same reason a student not having appropriate mathematical background would be "turned-off" by an elegant course in quantum mechanics. Although we are not proponents of developing special courses in laboratory practices, reasonable care must be exercised to be certain the student acquires experimental tools before developing an inhibition toward experimental physics. Vacuum ultraviolet spectroscopy has been largely neglected at the undergraduate level but we have found that a laboratory course in this area is an excellent vehicle for providing the students with contemporary tools for experimental research and at the same time providing interesting physical problems for investigation and understanding.

Georgia Tech is on a quarter system and the course has been offered three times:

Spring 1970 - 7 students,
Fall 1970 - 14 students,
Spring 1971 - 28 students.

Although the growth in popularity is encouraging, our present equipment is not adequate to handle 28 students without borrowing rather heavily on our research facilities. We plan to offer the course twice a year on an elective basis and would hope the enrollment would stabilize at approximately 20 students.

LABORATORY COURSE DESCRIPTION

The course consists of a one hour common discussion period each week and a three hour laboratory period each week. During the quarter we would typically have 9 to 10 discussion periods and

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8 to 9 laboratory periods. The final exam is thirty minutes of individual practical work and dialogue with the instructor. The grade is based on satisfactory performance in the laboratory including reports as well as the final examination.

The current text which we recommend for the course is Techniques of Vacuum Ultraviolet Spectroscopy by James A. R. Samson. The book is a good reference but suffers from not being written as an undergraduate text. Some of the discussion is too detailed and some is too abbreviated. This choice of text dictates that the instructor must play an important role which may speak for some of the success.

The course is designed around a McPherson Model 218, 0.3 meter, plane grating scanning monochromator. Most students have had no experience with vacuum technology and the first two experiments are designed to correct this deficiency. This experience with vacuum technology is followed by experiments on sources of radiation and is concerned with calibration lines as well as qualitative investigations of both line spectra and molecular bands. Comparison of the spectra from different gases and the different operating conditions of the plasma are investigated. Experiments on dispersion are concerned with the use of gratings with different blazes or with a different number of lines/cm. Other experiments relating to dispersion are concerned with the effect of slit width on both intensity and resolution. Experiments on detection systems include both a photomultiplier, as well as a channeltron.

The dc mode is used with the photomultiplier and is compared with photon counting using the channeltron. Other experiments include the transmission measurements of various materials such as LiF and quartz and the efficiency of sodium salicylate as a function of areal density.

A word of caution. The student needs to be informed the first day that he is going to become quite frustrated on several experiments as poor technique or faulty equipment will make itself known during the course. The opportunity to work with research grade equipment as well as these periods of frustration have been favorably commented on by students at the end of the course as being of value.

Course outline. As with any new course, we find the details changing rather dramatically with time. In fact we have adopted a philosophy that the students are completely free to change the suggested format of any experiment. We have found that the outline given below is satisfactory. A rather different distribution of time may be more satisfactory in another environment.

I. Vacuum Technology (2 to 3 Laboratory and Discussion Periods)

 A. Discussion periods

  1. Mechanical pump

  2. Sorption pump

  3. Gasp or aspirator pump

  4. Oil diffusion pump

  5. Ion pump

6. Liquid N_2 trap

7. Thermocouple gauge

8. Ion gauge

9. O-rings, viton gaskets, metal gaskets

10. Leak detection

B. Laboratory periods

1. Pumping speed and ultimate pressure of mechanical pumps

2. Pumping speed and ultimate pressure of oil diffusion pumps

3. Operation and characteristics of a thermocouple gauge and ion gauge

4. Effect of liquid N_2 on ultimate pressure

5. Operation of an oil free ion pumped system

II. Sources of Radiation (2 to 3 Laboratory and Discussion Periods)

A. Discussion periods

1. Operation and optical path of McPherson Model 218 monochromator

2. Operation and characteristics of Hinteregger arc

3. Atomic and molecular spectra

4. Synchrotron radiation

B. Laboratory periods

1. Familiarity and calibration of monochromator using Hg arc, both visual and photomultiplier detection techniques

2. Characteristics and optimum operating conditions for H_2 discharge

3. Characteristics and optimum operating conditions for He discharge

III. Dispersion (1 Discussion Period and 0 to 1 Laboratory Period)

A. Discussion period

1. Rowland circle

2. Design and characteristics of a normal incidence and grazing incidence vacuum monochromator

3. Gratings - meaning of blaze

4. Effect of slit width

B. Laboratory period

1. Comparison of spectra using gratings with different blaze angles

2. Examination of resolution in a narrow spectral range such as a doublet using a variable slit width

3. Effect of slit width on intensity

IV. Detection Systems (2 Discussions and 2 Laboratory Periods)

A. Discussion periods

1. Photomultipliers

2. Sodium salicylate

3. Channeltron

4. Ionization chambers

B. Laboratory periods

1. Efficiency of sodium salicylate vs areal density

2. Photon counting using a channeltron

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V. Materials in the Vacuum Ultraviolet (1 to 2 Discussion Periods and 1 to 2 Laboratory Periods)

A. Discussion periods

1. Filters
2. Polarizers
3. Coatings for gratings and mirrors
4. Reflection and transmission measurements

B. Laboratory periods

1. Transmission characteristics of LiF, quartz, and pyrex
2. Effect of pump oil on transmission of LiF
3. Excitation spectra of coating from fluorescent lamp
4. Transmission of biological materials

DESCRIPTION OF INDIVIDUAL LABORATORIES

A group of eight experiments which have been used in this laboratory are described below. These experiments are designed to exemplify basic experimental techniques, to develop an interest in experimental physics, to provide a transition from the structured laboratory to the research laboratory, and to encourage experimental inquisitiveness.

Experiment 1 - Vacuum Techniques

Students are provided with manufacturers' descriptions of two available mechanical pumps and are asked to compare observed pumping speeds with the manufacturers' literature. An experimental vacuum chamber which is approximately 2 ft. long and 15" I.D. was constructed from a piece of cast iron sewer pipe. The flanges and end plates were made from boiler plate. Neoprene gaskets between the flanges and end plates provide a vacuum seal which allows the chamber to be evacuated to approximately 10^{-4} torr. Positions for several thermocouple vacuum gauges are provided so that some appreciation can be obtained for the relative "reliability or unreliability" of these gauges. In addition several alternative pumping paths are provided to show the effect of constructions and right angle bends on pumping speed.

In parallel with the experiment on mechanical pumps students are asked to obtain the pressure vs time characteristics of a metal system which has an oil diffusion pump and liquid N_2 trap. This system is used later in the course as the pumping system for the vacuum monochromator. The equilibrium pressures with the mechanical pump, mechanical pump and diffusion pump, as well as the mechanical pump, diffusion pump, and liquid N_2 trap are determined. The operation of an ion gauge including the effect of degassing is made part of the experiment.

Although the experiments above use a metal diffusion pump, a glass diffusion pump is demonstrated in one of the discussion periods so that the action of the jets can be visually observed. As added demonstrations a thermocouple gauge has been cut open so students can view the junctions with a low power microscope and an old mechanical pump has been disassembled for their observation.

8

Experiment 2 - Vacuum Techniques

As contamination of optical components as well as samples by pump oil can result in serious problems, we have developed a laboratory experiment which utilizes an oil free stainless steel system. The system is shown schematically in Figure 1. Current vacuum technology is demonstrated and experience is gained with an aspirator pump, sorption pump, and ion pump. Students are asked to assemble the system using both viton gaskets and copper gaskets and then measure the pressure as a function of time.

Later in the course a photon counting experiment is performed using a channeltron detector. The channeltron is housed in this stainless steel vacuum system with a LiF window providing the optical coupling. This experiment was not included in our original planning but is now an integral part of providing an awareness of current vacuum technology.

Fig 1

Experiment 3 - Familiarity and Calibration of a Vacuum Monochromator

Most of the laboratory centers around the McPherson model 218 monochromator. Using a GE AH-4 mercury lamp as a light source and with the top plate of the monochromator removed, the students are instructed in changing gratings and are asked to observe the optical path when the grating is set for zero order falling on the exit slit. A qualitative measure of the divergence of the beam after it passes through the exit slit is made. The visible spectrum is observed by the students as the grating is rotated. After

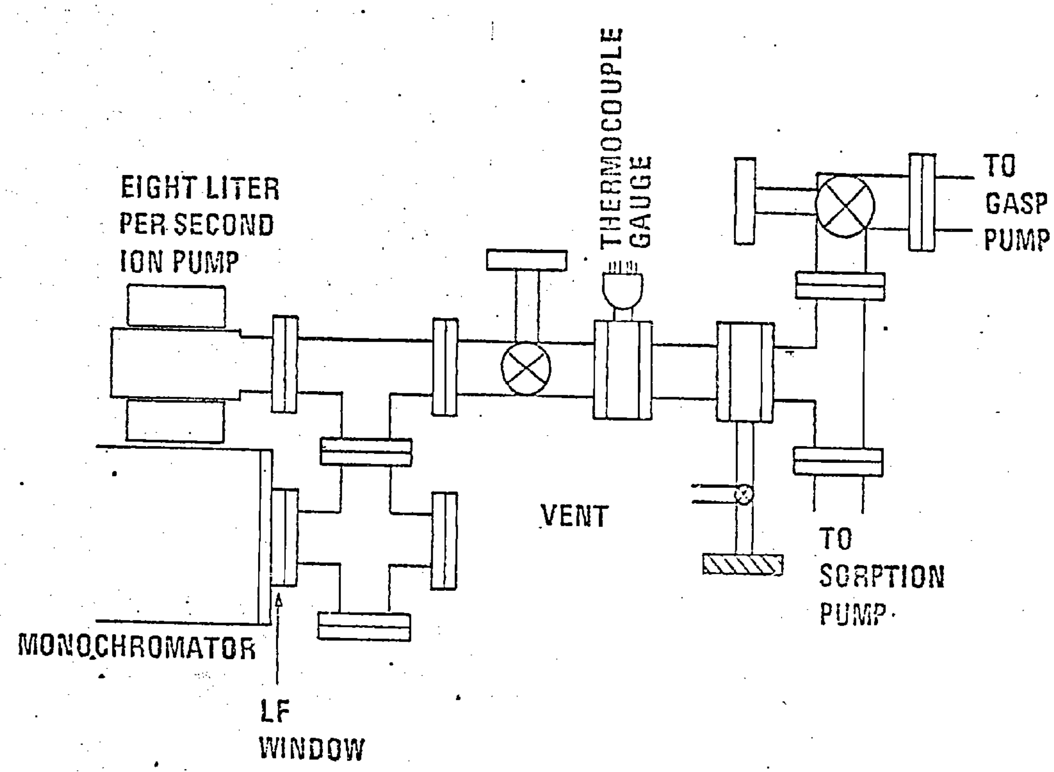

Figure 1 - Schematic Diagram of Stainless Steel Ultra High Vacuum System

visual observation in which known wavelengths are compared with dial readings, an AH-4 with the outer pyrex bulb removed is substituted as a light source. The spectrum is recorded using a photomultiplier tube viewing a glass plate previously coated with sodium salicylate. Figure 2 shows a diagram of our adaptor between the exit slit housing of the McPherson 218 and our photomultiplier housing. The design allows for introducing four different aperture configurations between the exit slit and the photomultiplier. The positioning of the apertures contained in the gear are made by rotation of a worm gear through an O-ring seal.

In addition to recording the spectrum the effect of slit width on resolution is observed. By using a weak line and narrow slits the effect of dark current noise is seen and the signal to noise ratio becomes meaningful to the students.

Fig 2

Experiment 4 - Comparison of the Vacuum UV Spectra of Hydrogen and Helium

In this experiment vacuum techniques are combined with optical measurements for the first time. A Hinteregger discharge lamp is attached to the entrance slit of the monochromator and the instrument is evacuated. The mechanical pump is vented to the outside of the building so that hydrogen will not accumulate in the room. Students are asked to identify the Lyman α line in the hydrogen spectrum as well as to determine the useful limits of the spectrum. Using the Lyman α line, the pressure and voltage-current characteristics of the arc are investigated to determine optimum operating

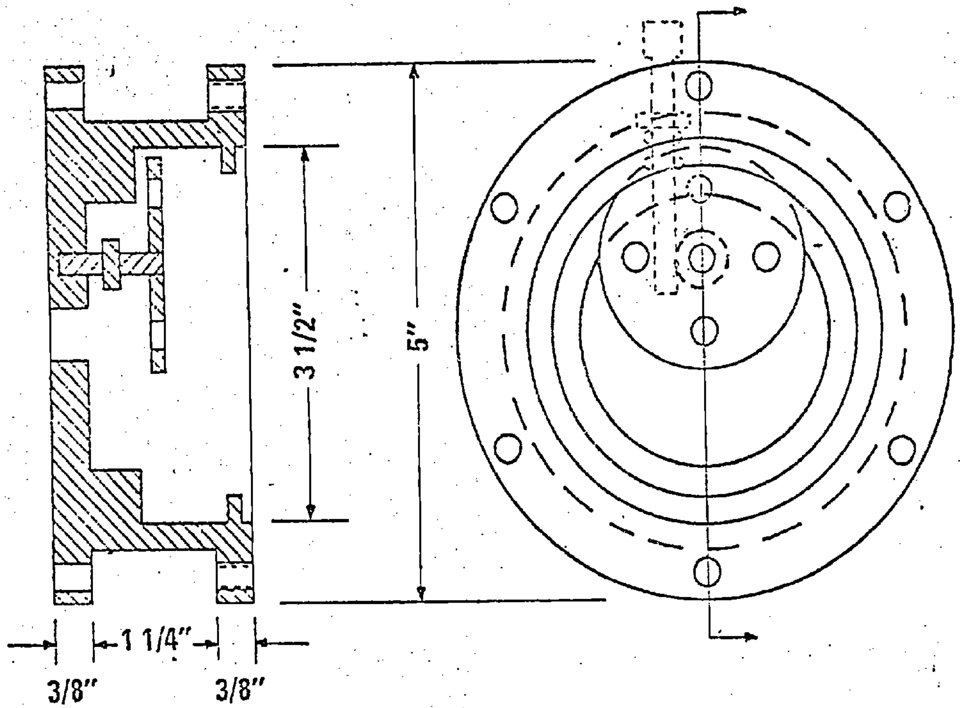

Figure 2 - Aluminum Adaptor Between Monochromator and Detection System

conditions which make the Lyman α intensity a maximum. The spectrum is then recorded. Similar measurements are made using helium. The students are requested to make an attempt at observing the 584Å resonance line in helium. Because of the multiple reflections used in the optical system of the Model 218, the line is quite difficult to observe. However, many inpurity gas lines are observed which is instructional. Later in the course the students are given an opportunity to observe the 584Å line using a normal incidence monochromator in our research laboratory.

Experiment 5 - Comparison of the Efficiency of Sodium Salicylate vs Areal Density of the Coating

The laboratory is designed to investigate a curve given in the text. Students prepare their sodium salicylate coatings on glass plates using a nebulizer and saturated solution of sodium salicylate in methyl alcohol. Preparation of a uniform coating is somewhat of an art but the frustration that results does not seem overwhelming to the student. The calculation of the areal density requires weighing the plates before and after spraying. The adaptor shown in Figure 2 allows for the rapid comparison of four different areal densities of sodium salicylate. The efficiencies are compared at the Lyman α line. The data from all of the different laboratory groups are recorded and made available to each student for his report.

Experiment 6 - Materials in the Vacuum Ultraviolet

By making use of the adaptor in Figure 2, measurements are made on the transmission characteristics of LiF, quartz and pyrex.

In addition the effect of pump oil on these characteristics is determined by placing a drop on the samples after their transmission has been determined. The transmission of the LiF plate is of importance to the next experiment in which a LiF window is needed to provide a high vacuum environment for the channeltron.

Experiment 7 - Photon Counting Using a Channeltron

A channeltron is mounted in the stainless steel system shown in Figure 1. As the system is constructed from commercially available 1-1/2" O.D. stainless steel components, we have found the helical configuration of the Bendix 4028 channeltron is desirable from space considerations. The flange containing the LiF window is a standard 2-3/4" high-vacuum flange and is bolted to an aluminum flange attached to the exit slit housing of the monochromator. The high vacuum assembly is put together and the ion pump started the previous week so that little time is wasted reproducing Experiment 2. Using the Lyman α line the effect of slit width, channeltron voltage, and discriminator setting on the counting rate is determined. A block diagram of the channeltron electronics is given in Figure 3. At this time in the laboratory, many students are quite impressed by the degree of sophistication and the amount of equipment necessary for the experiment. Fig 3

Experiment 8 - Student Determined Laboratory

Many questions have probably been suggested in the minds of the students and the laboratory now attempts to encourage this direction by asking students to choose a problem to probe more deeply for their next experiment.

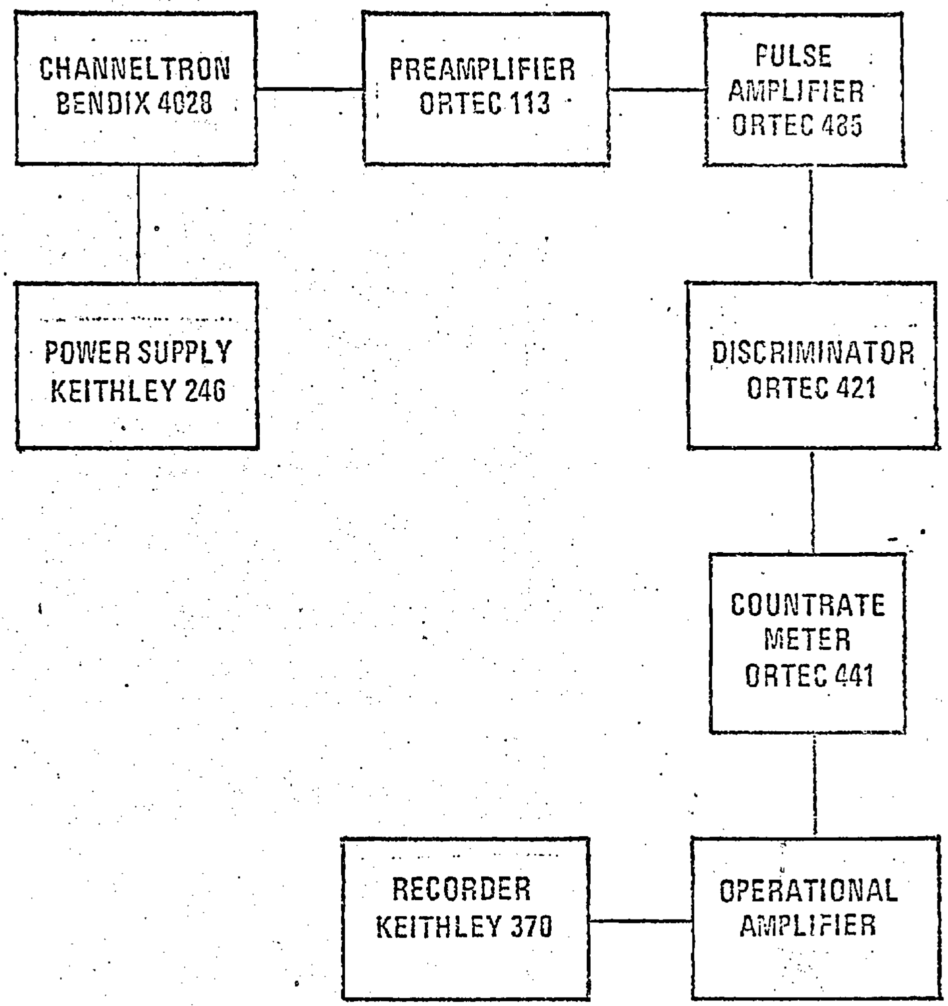

Figure 3 - Block Diagram of Channeltron Electronics for Photon Counting

Many times this will be a repeat of an experiment which did not work too well. Other times it may seek to provide greater depth such as making more quantitative measurements on signal to noise ratios, or the effect of slit width on intensity or resolution. Frequently students will wish to extend transmission measurements to include materials such as a specimen of skin.

FINAL EXAMINATION

As mentioned earlier, the final examination is thirty minutes of individual practical work and dialogue with the instructor. This examination is scheduled during the last week of the laboratory and during the students regularly scheduled period. Typically upon arriving at the examination the student might be asked to demonstrate the cut-off of LiF. In addition to this practical problem the student would be given a card containing four questions which would be used to initiate the dialogue. One group of these questions is given below:

(1) Explain the operation of an oil diffusion pump.

(2) The thermal conductivity of H_2 is higher than air. Explain what effect this would have on the readings of a thermocouple gauge calibrated in air but used to monitor H_2 pressure.

(3) Sketch the optical path of the vacuum monochromator used in this laboratory.

(4) If a photomultiplier were used instead of the photo-multiplier-phosphor combination, what constraints would be placed on the wavelength range?

Although the questions are useful to initiate the dialogue, the conversation will undoubtably cover many topics and be revealing to both the student and instructor. Erroneous concepts and ideas are quickly corrected and the student provides the instructor with a useful and constructive critical assessment of the laboratory.

EQUIPMENT COST

The laboratory is not cheap and is probably set-up more efficiently when research activities are present which complement the techniques used. Although we only schedule the laboratory on 2 of the 4 quarters, the equipment is utilized in all 4 quarters by making it available for special problem study during the alternate quarters. An itemized list of equipment and its cost is given below.

Item	Cost (1969-70)
McPherson model 218, 0.3 meter plane grating scanning monochromator with MgF_2 over-coated mirrors and 2400 lines/mm grating. Model 847 air inlet valve installed.	$5,500
McPherson model 630 Hinterregger arc with associated gas regulators	1,200

Item	Cost (1969-70)
Bendix Model SDC-100 high voltage power supply for arc discharge	$ 900
Two stage gas regulators for use with hydrogen and helium. Two needed ea. $50	100
Control circuit for thermocouple vacuum gauge 10^{-3} torr to 1 torr. Veeco model TG-7. Two DV IM gauge tubes and cable	200
Control circuit for thermocouple vacuum gauge, 0 to 20 torr. Veeco model TG-27. Two DV 4M gauge tubes and cable	250
Pico-ammeters. Keithley model 414. One required but 2 desirable ea. $450	900
Photomultiplier power supply. Keithley model 246 ea. $450. One required but two are desirable.	900
Strip chart recorders for use with pico-ammeters. Keithley model 370. One necessary but two are desirable. ea. $700	1,400
Photomultiplier tubes. EMI type 9514S. One needed but 2 are desirable - ea. $175.	350
Bendix channeltron model 4028	500

Item	Cost (1969-70)
Gasp roughing pump mounted with viton-sealed right angle valve on conflat (2-3/4") flange. Varian no. 942-1000.	$ 300
Standard VacSorb pump, Varian No. 941-6001	250
Vac-ion pump, 8 liters/second, Hughes No. VP-8	300
Control unit for ion pump, Hughes No. VPC 8	400
LiF window sealed to Varian Conflat flange. Harshaw Part No. 8960-1	100
Electrical Feedthroughs on 2-3/4" Conflat flange. Varian No. 954-5008	150
Tees, 1-1/2" O.D. stainless steel tubing, Varian No. 952-5051. 2 needed - ea. $75	150
Cross, 1-1/2 O.D. stainless steel tubing. Varian No. 952-5050.	100
Viton-sealed right angle valve, Varian No. 951-5058	150
Double sided conflat flange with mounted DV-6M gauge tube and 1/2" valve. Varian No. 951-5058	200
Copper gaskets, screw and nut sets, viton gaskets	100

Item	Cost (1969-70)
Modular system bin with power supply. Ortec model 401A/402A	$ 600
Preamplifier, Ortec model 113	100
Pulse amplifier, Ortec model 485	250
Discriminator, Ortec model 421	200
Countrate meter, Ortec model 441	300
	$15,850

In addition to the above items, several components were used from available equipment. These included two mechanical pumps, a small metal vacuum system which uses a 2" oil diffusion pump and cold trap, an ion gauge and ion gauge control. Shop time and materials for construction of the large volume vacuum system in addition to the adaptor shown in Figure 2 are not included. Supplies such as liquid N_2, hydrogen and helium gas, hand-tools, chart paper, pump oil, and nebulizer are dependent on the number of students involved. The availability of an analytical balance is assumed.

The power supply for the discharge lamp requires 220 volts be available. In addition water cooling is necessary for the discharge lamp and outside venting is desirable for the mechanical pump when hydrogen is being used as a discharge gas.

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