VACUUM TEACHING FOR UNDERGRADUATE STUDENTS AT THE UNIVERSITY OF COIMBRA

UČENJE VAKUUMA ZA ŠTUDENTE NA UNIVERZI COIMBRA

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Prejem rokopisa – received: 2006-04-18; sprejem za objavo – accepted for publication: 2006-07-18

The teaching of vacuum science and technology is often neglected at universities due to the limited time available to establish a comprehensive physics curriculum for students, although it is important in many different aspects of everyday life. An overview of an introductory course of about 60 hours on vacuum science and technology, offered by the Physics Department at the University of Coimbra to students majoring Physics, Physics Engineering and Material Science Engineering, is presented. The course not only covers lectures with the fundamental concepts and processes involved in vacuum production and measurement, as well as a detailed analysis of the main types of vacuum equipment and systems, it also contains a large practical component with a set of experiments involving actual measurements, like rotary and diffusion pumping-speed measurements, roughing-line conductance measurements, out-gassing rates and permeability constant measurements of different materials, in order to provide students with a first contact and training in vacuum equipment. The laboratory classes also feature a full maintenance service for diffusion-based high-vacuum systems. The issue of vacuum science and technology applications to research and industry is supported by visits to the different vacuum systems. These last visits are organized in close collaboration with an agent/supplier of vacuum equipment and a vacuum-system designer/manufacturer, which also organizes one or two lectures.

Key words: undergraduates teaching, vacuum science and technology, vacuum measurements

Poučevanje vakuumske vede in tehnologije je na univerzah pogosto zanemarjeno zaradi omejenega časa, ki je namenjen pouku fizike, čeprav je pomembno v mnogih vidikih vsakodnevnega življenja. Predstavljen je pregled uvodnega tečaja za vakuumsko vedo in tehnologijo v obsegu približno 60 ur na Oddelku za fiziko Univerze Coimbra za študente fizike ter inženirstva fizike in materialov. Tečaj obsega temeljne koncepte in procese, pomembne za proizvodnjo in meritve vakuuma in detaljno analizo osnovnih vrst vakuumskih naprav in sistemov. Obsega tudi precej praktičnega znanja z vrsto eksperimentov z meritvami, npr. meritve pri rotacijskih in pri difuzijskih črpalkah, prevodnost predčrpalnih vodov, hitrost razplinjevanja in določitev konstant permeabilnosti za različne materiale, vse z namenom, da študentje dobijo prvi stik z vakuumsko opremo in vadijo z njo. Laboratorijsko delo je namenjeno tudi vzdrževanju difuzijskih visokovakuumskih sistemov. Pomen uporabe vakuumske vede in tehnologije za raziskovanje in industrijo je obogaten z obiski pri različnih vakuumskih sistemih v Oddelku za fiziko in v nekaj podjetjih, ki uporabljajo velike vakuumske sisteme. Ti obiski so organizirani s sodelovanjem dobaviteljev in načrtovalcev vakuumskih sistemov, ki izvedejo tudi eno ali dve predavanji.

Ključne besede: poučevanje študentov, vakuumska veda in tehnologija, vakuumske meritve

1 INTRODUCTION

Vacuum science and technology is an important, established tool in research and industrial environments, with applications in large areas of human activity and knowledge. The improvements achieved in vacuum science and technology often contribute to advances in other areas of knowledge, such as surface physics, high-energy physics, electronics, metallurgy, pharmacy and the food industry. The increasing dependence of research and industry upon more elaborate, well-designed vacuum systems leads to the need for well-trained staff and engineers. This requires a corresponding response in the teaching of such curricula in undergraduate university education, both in the physical and engineering sciences.

Often, the education and training of researchers and technicians is achieved through a formal teaching of the principles, followed by actual practice in the laboratory. This practice has relevant importance and significance in a field like vacuum science and technology and the need for an essentially practical approach has been emphasized over many years. This is of particular importance for undergraduate university education, given the limited time available for teaching those curricula and the need for many students to have those skills to succeed in their postgraduate studies and, in some cases, diploma work.

In addition, it is very important for students' education and training that actual quantitative measurements can be obtained during the experimental work, rather than just the simple operation of vacuum systems and qualitative analysis. Simple experiments allowing students to perform direct measurements of the characteristics of different vacuum components and material properties are, thus, important. On the other hand, the perception and skills that are acquired by the students depend on the infrastructures available, which should include the most important vacuum components and systems.

The purpose of this paper is to summarize the topics covered in a one-semester introductory course on vacuum science and technology offered by the Physics Department of the University of Coimbra, which includes a large practical component and a broad applications overview in addition to the theoretical lectures. The course is designed for senior-level undergraduate students or first-year graduate students majoring in Physics, Physics Engineering and Material Science Engineering.

2 COURSE SCOPE AND OVERVIEW

This course aims to provide students with knowledge of the basic concepts involved in vacuum physics and the means to have their first contact and experience in operating different vacuum systems and how to obtain information on their operating conditions, capabilities and limitations. Rather than present to students detailed foundations of the fundamental processes involved in vacuum physics, we provide them a more practical and technical approach on the comprehension, operation and selection of different vacuum equipment and practices. We aim to present students with a training that is more directed at the user's point of view, rather than that of a researcher involved in vacuum science.

With the Bologna Agreement, the student's curricula are reduced and a course like vacuum science and technology has to be implemented in less time than would be desirable. At the University of Coimbra, an total of 4 hours per week, altogether 56 to 60 hours, has been planned for this introductory vacuum course.

In our case, it was decided to structure the course in two major parts, dividing the time available for the course in half, between theory and experimental classes. During the first part, students are introduced to the basic aspects of vacuum physics and technology. Theory follows a common approach: fundamentals of vacuum physics, ideal vacuum systems, real vacuum systems and materials for vacuum, vacuum pumps and accessories, pressure measurements and (ultra)-high-vacuum systems and system design.

The extensive use of catalogues of the different manufacturers and suppliers of vacuum equipment, as well as the use of equipment manuals are implemented whenever possible. They present a useful reference for the students in their future positions, either in industry or in research laboratories, and an important way to extract relevant information for their future needs.

The laboratory portion of the course is structured in 4-hour laboratory classes. During the operation of vacuum systems, students are able to perform direct quantitative measurements of the characteristics of different vacuum components and/or material properties, and compare with the values tabulated in the literature. Experiments include mechanical and diffusion/turbomolecular pumping-speed measurements, roughing-line conductance measurements, out-gassing and permeability measurements from materials under vacuum, residual gas analysis and thin-film thermal evaporation.

A small, comprehensive high-vacuum system is used with a rough vacuum gauge (e.g., Pirani) at both the high-vacuum chamber and the mechanical pump inlet ¹, **Figure 1**. This allows the monitoring of the pressure in the vacuum chamber during the roughing procedure and after the high-vacuum valve is closed and the pressure quickly exceeds the high-vacuum gauge scale.

Helium-leak detectors and mass spectrometers have become common in both research and industrial environments. They have changed from the luxury equipment, requiring expert handling, to economic, reliable and powerful monitoring instruments that are relatively easy to use. Therefore, it is important to include these systems in the experimental training of the students. A simple experiment, using a He-leak detector to measure the helium permeability constant of different materials and the leak-throughput dependence on material thickness, area and helium pressure differential, is available to students. With a mass spectrometer, students are able to perform direct quantitative measurements of isotopic abundances for gases, like neon, argon, krypton, and xenon.

The systems available for laboratory classes are simpler and much more limited than those existing in research/teaching institutions such as our Physics Department. Since it is important to present to students an overview of vacuum systems and their applications,

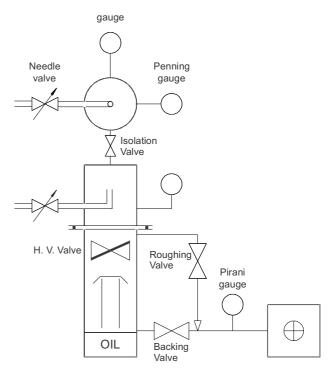


Figure 1: Schematic diagram of the diffusion-pump high-vacuum system used for the laboratory experiments

Slika 1: Shema visokovakuumskega črpalnega sistema z difuzijsko črpalko, ki se uporablja za laboratorijsko delo

we reserve some of the available time for a detailed visit to all the different vacuum systems and components, which are in operation in our research laboratories, under the topic of vacuum science and technology applications in science and industry. Along this line, visits to industrial facilities using large vacuum systems (e.g., metal-film deposition, freeze drying, vacuum furnaces, and vacuum transport), are also offered at the end of the course. In particular, the close collaboration with an agent/supplier of vacuum equipment and a vacuumsystem designer/manufacturer ², which has an overview of industrial and research facilities, provides the students with a closer insight into the applications of vacuum technology and the possibility for further collaboration and training in this area.

3 THEORETICAL LECTURES

The syllabus for the first part of the course is outlined in **Table I** and detailed in the following paragraphs. The classes start with a brief overview of the definition of vacuum, the different vacuum ranges and references to different applications of vacuum technology to industry and research, in particular to those present at national level.

The kinetic theory of gases is used to describe the physical quantities with interest to vacuum (e.g., molecular density, mean velocity and energy of molecules in a gas volume, rate of molecular incidence on a surface, pressure, molecular mean free path and the time of formation of a molecular monolayer on a clean surface) and its dependence on pressure and/or temperature. The relevant parameters to gas flow through vacuum systems (pumping speed, gas throughput, conductance of pipes and other elements) are then defined and the different gas-flow regimes are discussed, pointing out situations that are likely to be present in standard vacuum systems.

The ideal vacuum system is then discussed in terms of the pumping speed in different parts of the system and to the pressure dependence on the pumping time. The effects of real and virtual leaks are discussed, as well as the concept of the ultimate pressure reached in a vacuum chamber. The limitations present in real vacuum systems are highlighted, by discussing the different sources of gas that subsist in the vacuum chamber, detailing the surface out-gassing, the volume diffusion, the permeation, the vaporization and the gas back-streaming from the pump. Some examples are given for the effect of out-gassing rate and permeability on the ultimate pressure achieved in vacuum chambers, or along the vacuum system, and its dependence on the pumping time. In this section we introduce a discussion on the characteristics of the different materials suitable for vacuum, their advantages and limitations.

It is in the following topics – vacuum production and pressure measurement – that the extensive use of the different manufacturers' catalogues and equipment manuals is implemented. Detailed descriptions of the

- Vacuum Fundamentals
 - Kinetic theory of gas
 - Gas flow in vacuum systems
- Ideal vacuum systems
 - Pumping speed in vacuum systems
 - Pressure dependence on pumping time
 - Real and virtual leaks, ultimate pressure
- Real vacuum systems
 - Sources of gas inside vacuum systems & out-gassing
 - Pressure dependence on pumping time
 - Materials for vacuum systems
- Vacuum production
 - Pumps and accessories
 - Valves and fittings
- Pressure measurement
 - Pressure gauges
 - Gauge calibration & standards
- Leak detection

– Residual gas analysers

- Laboratorial practice

– Vacuum applications to science and industry

operating principles, characteristics and limitations are found in books, and these are the most important technical characteristics needed for the selection and comparison of the commercially available pumps and gauges found in the catalogues. Pump accessories, such as oils, filters and traps, their usefulness and drawbacks are also included in this topic.

Particular attention is given to mechanical, roots and diffusion pumps, due to their extensive use in industry because of their simple operation, easy maintenance and the possibility to achieve large pumping speeds at low cost. In addition, the different pumping combinations, advantages and limitations, are discussed.

Concerning pressure measurements, besides a discussion of the different processes and the different gauges available, attention is given to the precision of such measurements and the requirements in both research and industry. Particular attention is also given to gauge calibration, the dependence on the residual gas present in the vacuum system, absolute gauges and the importance of calibration methods, as standards.

The leak-detection topic starts by highlighting the differences between real and virtual leaks and system out-gassing. Any of these contributions could be responsible for the ultimate pressure present in the vacuum system and/or for the pressure rise when the pumping action is stopped. The sequential localization of a leak by monitoring the pressure reading of the system gauges, while isolating different sections of the vacuum system, and/or taking advantage of the different sensitivity of the gauges to different gases, is discussed. The different leak-detection methods and leak-detector

systems are then discussed. Residual gas analysers are discussed, not only as a part of a leak-detector system, but also as a powerful tool that gives important information about the gas species that are being injected into the vacuum volume, due to either leaks or system out-gassing.

Typical texts used for the course lectures are those presented in the books of O'Hanlon ³ and Weston ⁴.

4 LABORATORY PRACTICES

The laboratory experiments performed by the students are listed in **Table II**. All the experiments involve quantitative measurements of the characteristics of vacuum pumps, the conductance of different components and the material properties. A detailed description of these experiments and some illustrative results obtained by students are presented in Ref. ⁵.

For most of the experiments we use a comprehensive high-vacuum system for laboratory teaching ⁶, **Figure 1**. An ISO test dome ^{7.8} is connected to a NW63 oil diffusion pump (135 l/s) backed by a 0.75 m³/h rotary pump. This vacuum chamber has several ports needed for gas inlet, vacuum gauges and connection to other equipment. A combined Pirani/Penning controller is used, allowing continuous pressure monitoring in the high-vacuum chamber, in the 10^5 to 10^{-5} Pa range, and a rough-vacuum pressure reading at the rotary pump inlet.

The particular use of a rough-vacuum gauge in the high-vacuum chamber ^{1,9} enables an evaluation of the gas throughput that enters the chamber volume, through the pressure-rise rate method ¹⁰, without the need for any further equipment or system as a flowmeter. Real leaks, fed through a needle valve, are used for pumping-speed measurements ^{11,12}, and the out-gassing rate of the materials placed inside the chamber is directly obtained from the gas load flowing into the chamber as a result of the out-gassing of those materials ¹³.

The rough vacuum experiments are performed with the diffusion pump off and isolated. Rotary-pump speed

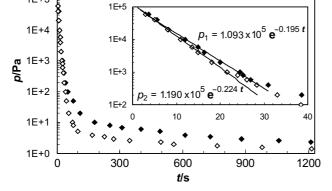


Figure 2: Pressure evolution as a function of time in the high-vacuum chamber, $p_1(\bullet)$, and at the rotary-pump inlet, $p_2(\diamond)$

Slika 2: Nastajanje vakuuma v odvisnosti od časa v visokovakuumski komori $p_1(\bullet)$, in vhodu v rotacijsko črpalko $p_2(\diamond)$

Table II: Laboratorial experiments**Tabela II:** Laboratorijski eksperimenti

- Rough vacuum experiments
 - Rotary pump speed measurement by the constant volume method
 - Rotary pump speed measurement by the constant pressure method
 - Roughing line conductance measurement
- High vacuum experiments
 - Diffusion pumping-speed measurement
 - Vor and Viton out-gassing rate measurements
- Leak detector operation
- Permeability constant measurement of Kapton thin foils
- Mass quadrupole spectrometer operation
- Isotope abundance measurements
- Diffusion pump unit maintenance
- Thin-film thermal evaporation

measurements are done using both constant-volume and constant-pressure methods ⁵. For the constant-volume method, the pumping speed can be measured in both places of the vacuum system, in the high-vacuum chamber and at the rotary pump inlet. **Figure 2** shows both pressures as a function of the pumping time. The effective pumping speed, S, is calculated using

$$S = -V \,\mathrm{d}(\log_{\mathrm{e}} p)/\mathrm{d}t \tag{1}$$

where V is the volume being pumped and p the associated pressure. For pressures above 10^3 Pa, the data can be fitted to a straight line with slope -(S/V), i.e., S is independent of p. For lower pressures, S decreases due to the finite compression ratio of the pump and due to out-gassing. The results obtained by the different students for the pumping speed at the pump inlet are within 6 % and agree with the value given by the manufacturer, while those obtained in the chamber are within 7 %. This experiment allows the students to observe the effect of the roughing-line conductance on the pumping speed and on the ultimate pressure, p_u , achieved in the chamber.

In the constant-pressure method an air leak is allowed to flow into the chamber through the needle valve and the equilibrium pressure is taken for both Piranis, p_{eq1} and p_{eq2} . The gas throughput, Q, flowing into the chamber is obtained through the pressure-rise rate method, after the high-vacuum chamber has been isolated, **Figure 3**:

$$Q = V \,\mathrm{d}p_1/\mathrm{d}t,\tag{2}$$

The throughput measurement, **Figure 3** a, and the equilibrium pressure readings in both places allow not only a determination of the rotary pump speed, at these places, as a function of pressure in the chamber, **Figure 3** b,

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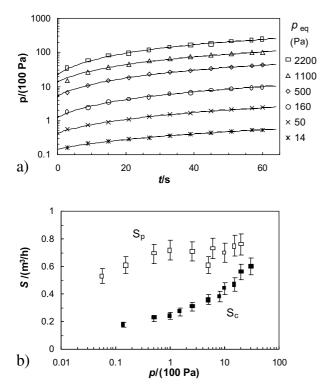


Figure 3: (a) Pressure evolution in the high-vacuum chamber as a function of time, after the rotary pump has been stopped (at t = 0), for different air leaks through the needle valve. The solid lines represent linear fittings to the different data sets. p_{eq} is the equilibrium pressure reached in the chamber while the pumping action is on. (b) Rotary-pump pumping speed as a function of pressure, at the pump inlet, S_p (\blacksquare), and in the high-vacuum chamber, S_c (\square), taken from the data of **Figure 3a** and from the respective values of p_{eq} .

Slika 3: (a) Nastajanje vakuuma v visokovakuumski komori v odvisnosti od časa po zaustavitvi rotacijske črpalke (pri t = 0) za različno intenziteto puščanja igelnega ventila. Cele črte so linearni približki podatkom. p_{eq} je ravnotežni tlak v komori med delovanjem črpalke. (b) Hitrost črpanja rotacijske črpalke v odvisnosti od tlaka pri vstopu v črpalko, S_p (\blacksquare) in v visoko-vakuumsko komoro, S_c (\square) po podatkih s **slike 3** a in vrednostih za p_{eq} .

$$S = Q/(p_{\rm eq} - p_{\rm u}) \tag{3}$$

but also an evaluation of the roughing-line conductance, C,

$$C = Q/(p_{eq1} - p_{eq2}) \tag{4}$$

as a function of the average pressure, $p_{av}=(p_{eq1}+p_{eq2})/2$, **Figure 4**. This last measurement shows an approximately linear trend, denoting the fact that the air flow through the roughing line is predominantly laminar for the studied pressures.

Students can compare the pumping speed values obtained with the different methods and discuss their precision. In addition, the difference between real and virtual leaks can be established by monitoring the pressure rise in the high-vacuum chamber when the leak valve is closed: the pressure in the chamber rises towards an equilibrium pressure, instead of the linear rise obtained in the leak throughput measurements.

The high-vacuum experiments include the pumpingspeed measurement of the diffusion pump as a function

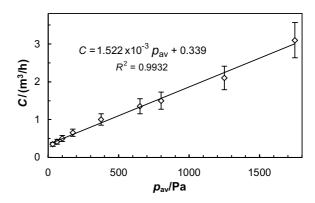


Figure 4: Roughing line conductance, C, as a function of the average pressure at the ends of the line, p_{av} . The solid line represents a linear fitting to the data

Slika 4: Prevodnost predčrpalne linije C v odvisnosti od povprečnega tlaka na koncu linije p_{av} . Cela črta je linearni približek podatkom

of pressure at its inlet and the measurement of the out-gassing rates of Vor and Viton. The pumping speed is obtained through the constant pressure method, being the gas throughput flowing into the high-vacuum chamber determined by the pressure-rise reading on the Pirani, after the equilibrium pressure in the chamber has been reached and the high-vacuum valve has been closed, Figure 5. Out-gassing rates are determined by measuring the gas throughput flowing into the highvacuum chamber due to out-gassing of the samples placed under high-vacuum, using the pressure-rise reading on the Pirani after the high-vacuum valve has been closed. The out-gassing can be taken as a virtual leak: after the high-vacuum valve has been closed, the pressure in the high-vacuum chamber will rise to an equilibrium pressure. However, for pressures much lower than this equilibrium, a nearly linear rise is observed and the gas throughput due to out-gassing can be estimated, Figure 6. The out-gassing throughput is obtained using equation (2) and the sample out-gassing rate $(q_t, where t is the time that the sample has been$ under vacuum) is obtained from

$$Q_{\rm t} = (Q - Q_0)/A \tag{5}$$

where Q_0 is the background out-gassing throughput and A is the surface area of the sample. The background out-gassing from the high-vacuum chamber surfaces is calculated by the same process. To reduce this contribution we have used an independent, small sample chamber connected to the high-vacuum chamber through an isolation valve. In this way, only the surfaces of this small chamber are exposed to air during the sample handling, while the high-vacuum chamber is always kept under vacuum. As an example, for Vor, the results obtained by the different students extended through almost a decade, with a relative standard deviation of about 60 %. Nevertheless, the obtained results are in reasonable agreement with those reported in the literature ¹⁴.

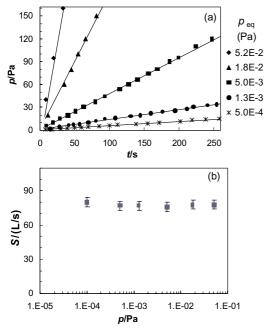


Figure 5: (a) Pressure rise in the high-vacuum chamber as a function of time, after the high-vacuum valve has been closed (at t = 0), for different air leaks through the needle valve. The solid lines represent linear fittings to the different data sets. (b) Diffusion-pump pumping speed as a function of pressure in the chamber, as obtained from the data in **Figure 5 a**.

Slika 5: (a) Povečanje tlaka v visokovakuumski komori v odvisnosti od časa po zaprtju visoko vakuumskega ventila (pri t = 0) za različno intenziteto puščanja igelnega ventila. Cela črta je približek podatkom. (b) Hitrost črpanja difuzijske črpalke v odvisnosti od tlaka v komori, kot je določeno iz podatkov na **Slika 5 a**.

In the last two experiments the students have the opportunity to operate two important pieces of vacuum equipment: a He-leak detector and a mass spectrometer. These experiments are assisted by a graduate student acquainted with the operation of these pieces of equipment.

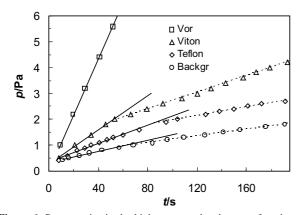


Figure 6: Pressure rise in the high-vacuum chamber as a function of time, after the high-vacuum valve has been closed (at t = 0), for different samples present inside the sample chamber

Slika 6: Povečanje tlaka v visokovakuumski komori v odvisnosti od časa po zaprtju visoko vakuumskega ventila (pri t = 0) za različne vzorce v komori z vzorci The He-leak detector is used to measure the He throughput flowing through a Kapton foil due to its permeability ¹⁵, which can be taken as a real leak. The use of a calibrated leak will allow absolute throughput measurements. The Kapton foil separates two volumes, one connected to the leak-detector and the other to the He-line. Knowing the film area, the thickness and the He-pressure differential, the Kapton permeability constant can be calculated using

$$Q = K \left(\frac{A}{d} \right) \Delta p \tag{6}$$

where A is the area of the material through which the permeability is being measured, d is its thickness and Δp is the differential pressure between the two sides of the material. Students can assess the dependence of the He throughput on the foil area, on foil thickness, **Figure** 7, and on the He pressure differential. The results obtained for the Kapton permeability constant are in good agreement with those reported in the literature ⁴.

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The operation of the mass spectrometer (a quadrupole mass spectrometer, in our case), not only aims to get students acquainted with the residual gas analysis and with cracking patterns, but also to measure the natural abundances of the different isotopes of elements like xenon, krypton and argon ¹⁶. The mass spectrometer's sensitivity and response changes with its operating conditions and history 3,4 but, for isotope analysis, only the relative performance is needed. Given the small mass range and the fact that the chemical species of the isotopes is the same, the mass spectrometer sensitivity is constant for all isotopes. In addition, as the isotope abundance is obtained through the relative partial pressure to the total sum of the partial pressures of all isotopes, the obtained experimental results are independent of the mass spectrometer's operating conditions and agree well with the tabulated results (e. g. ³). Figure 8 depicts typical scans, obtained by the

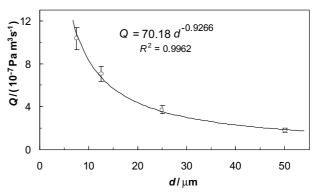


Figure 7: Helium leak-rate due to permeability, Q, through 1-cm diameter Kapton foils as a function of their thickness, d, for a 1-bar helium-pressure differential. The solid line is the best fit of a power curve to the experimental data.

Slika 7: Hitrost puščanja helija zaradi permeabilnosti L skozi Kapton-folije s premerom 1 cm v odvisnosti od njihove debeline d pri razliki tlaka helija 1 bar. Cela črta je približek potenčne krivulje eksperimentalnim podatkom.

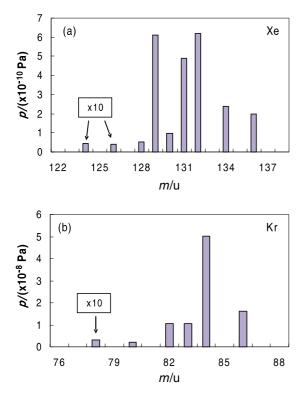


Figure 8: Typical partial pressures, as read by the mass spectrometer, in the range of (a) 122 to 138 u, and (b) 76 to 88 u, for a xenon and a krypton leak, respectively, introduced in the vacuum chamber **Slika 8:** Tipičen parcialni tlak, kot je določen v masnem spektrometru v območju (a) 122 u do 138 u in (b) 76 u do 88 u za puščanje ksenona

in kriptona iz vakuumske komore.

mass spectrometer for xenon and krypton leaks introduced in the vacuum chamber.

In addition to the above experiments, time is reserved for students to perform a full maintenance of a diffusion pump system, including diffusion pump disassembling, cleaning and system assembling and performance check. This maintenance has been performed using one of the two systems available for the laboratory classes or alternatively, using other systems needing maintenance from various research groups.

5 CONCLUDING REMARKS

The increasing use of vacuum technology in research and industrial environments in large areas of activities requires a corresponding response at university level by providing undergraduate students with the teaching of such curricula. At the Physics Department of the University of Coimbra, an introductory course on vacuum science and technology is available for students majoring in Physics, Physics Engineering and Material Science Engineering, and has been taught during the past decade.

The contents of the course allows teaching the students an appreciation of the basic concepts and, also, provides a first contact and training with modern vacuum equipment, while supplying the theory behind it. A comprehensive set of basic experiments have been put together, which allows the students to perform actual measurements of the characteristics of different vacuum components or material properties, during the course of the experimental activities. The result of this effort will be better-trained graduates to meet the demands of both research laboratories and industry.

The close collaboration with an agent/supplier of vacuum equipment and a vacuum-system designer/ manufacturer, which has an overview of industrial and research facilities, as well as visits to industrial facilities and to research laboratories are important tools in providing the students with a good insight into the applications of vacuum technology and to open up the possibility of further collaboration and training for those willing to undertake additional studies in this area at an engineering level.

Acknowledgements

This work was carried out in the Atomic and Nuclear Instrumentation Group of the Instrumentation Center (Unit 217/94) of the Physics Department of University of Coimbra. Support is acknowledged from Fundação para a Ciência e a Tecnologia, FCT (Lisbon), and from FEDER.

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