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Experimental Results of Hydrogen Distillation at
the Deuterium Removal Unit of the MuCAP
Experiment

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Экспериментальные результаты очистки водорода на установке для удаления дейтерия в MuCAP эксперименте

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Аннотация

Рассматривается криогенная разделительная колонна внутренним диаметром 2.2см и высотой 155см, являющаяся основным элементом установки для удаления дейтерия из водорода для циркуляционной системы сверхвысокой очистки водорода (CHUPS). Приводятся основные рабочие параметры и возможности колонны при работе в разных режимах. Получены результаты работы колонны при работе с нормальным водородом и протием с содержанием дейтерия около 2ppm: задержка на насадке колонны, падение давления в колонне и чистота конечного газа. Для определения эффективности колонны использовались хроматографические измерения разделения орто- и пара-водорода, из-за отсутствия оперативных методов измерения малого содержания дейтерия в водороде.

Abstract

The cryogenic distillation column of 2.2 cm inner diameter and 155 cm packing height, which is the main element of a new unit (named the Deuterium Removal Unit) of the Circulation Hydrogen Ultrahigh Purification System, has been described in details. Fundamental operating parameters and column performance at different operating modes have been measured on the column. Using both normal hydrogen and hydrogen with residual deuterium content about 2 ppm data have been gathered and given here on liquid holdup in the column packing, on pressure drop across the column, and on product purity. Ortho-para hydrogen analysis by gas chromatography was used for the column performance evaluation because of the inefficacy of low deuterium content hydrogen analysis.

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I. INTRODUCTION

MuCAP (Muon Capture on the Proton) experiment [1] has been carried out during 1998-2006 at Paul Sherrer institute (PSI), Switzerland. The goal of this experiment is measuring the rate of the basic electroweak process of muon capture with hitherto unachievable high precision (1%). This measurement will provide an estimation of the nucleon charged form factor g_p with all-time high accuracy $\sim 7\%$.

The method needs a very precise measurement of the muon lifetime in the μ^+p system in comparison with the lifetime of the free μ^+ . To achieve this precision, ultra pure and deuterium depleted hydrogen gas (so called protium) must be used. It is necessary to avoid transfers of μ^+ to impurities or deuterium nuclei. These transfers lead to spoiled time spectra [2]. A new experimental technique based on high pressure time projection chamber (TPC) filled with protium was used for the measurements.

After preliminary studying, basic purity requirements for working gas were proposed. The concentration of additional impurities, such as nitrogen, oxygen, water, hydrocarbons and other matters must be strongly limited. Desirable overall part of contaminants is about 10^{-8} . As for the isotopic purity, the concentration of deuterium (2H/1H ratio) must not exceed $1 \cdot 10^{-6}$ (1 ppm) [2] but desirable level is 0.1 ppm. Average concentration of this heavy isotope in natural water is 156 ppm.

Circulating Hydrogen Ultrahigh Purification System (CHUPS) intended to provide the permanent purification of protium from the foreign contaminants during an entire statistics run (up to 2 months of continuous operation) [3]. This system was recently appended by the Deuterium Removal Unit (DRU) – device for manufacturing of ultra pure protium from hydrogen obtained by electrolysis of previously “depleted” water or even from natural hydrogen. A principle of hydrogen cryogenic distillation was used as a basis for the unit design.

The rectification method is prevailing and its principles are well-known. This method uses the difference in saturation vapour pressure of separating species above the surface of the mixture. It can be considered as a multi-step distillation with the use of a column filled with special facility for increasing a phase contact surface. This facility can be either a set of perforated plates or particular packing. The packing is more suitable for a column with a relatively small inner diameter. On the top of the column a condenser is placed which condenses vapour and returns it into the column in the form of liquid. This liquid is called reflux. The vapour from the top of the column can be partially taken away from the column as a product of the process. The reflux drains down along the column moistening the packing. An amount of the liquid suspended on the packing is a column holdup. Lower end of the column is equipped by reboiler. A

separated mixture boils in the reboiler forming the vapour. The vapour rises upward along the column and interacts with the counterflow of draining reflux: the liquid is being saturated by the high-boiling component, and the gas – by low-boiling.

In our case of hydrogen-deuterium separation a separating mixture is the mixture of regular hydrogen (H_2) and “deuterohydrogen” (HD). D_2 molecules are extremely rare and should not be considered. Besides, there is a division of the H_2 molecules into two groups, one of which has its protons aligned in the same direction (referred to as orthohydrogen) and the other in opposite directions (parahydrogen). Ordinarily, transition between ortho and para forms are relatively rare, so H_2 can be considered as a mixture of two distinct components. The ratio between the ortho and para forms is about 3:1 at standard temperature and pressure, but the para form dominates at low temperatures.

Separation factor, α , defines of an elementary separation effect achieved at one contact of liquid and vapour hydrogen. Ideal separation factor is a ratio of saturation vapour pressures above pure components, it depends on temperature: the higher temperature the lower separation factor. But separation factor for deuterium-protium is much larger than for ortho-para hydrogen at the same temperature. Throughout the contacting tower, liquid and vapour are brought into repeated contacts that multiply elementary separation effect. Thus the flowing down liquid hydrogen becomes steadily enriched in deuterium and ortho-hydrogen while the rising vapour becomes steadily depleted. According to the concept of Equilibrium Theoretical Plate, Height Equivalent to a Theoretical Plate (HETP) is the main performance characteristic of a separation column. HETP is the height of a part of a column left by liquid and gas flows which are in equilibrium to each other, (i.e., for example, deuterium concentration in the liquid is α times higher than in the gas). The lower HETP value the higher column's separation power.

The column was designed to be operated so as to separate the initial mixture injected into the column through a feed port withdrawing pure H_2 (with deuterium content at least 30 times lower than in feeding gas) as distillate product and discarding the HD component in the reboiler stream.

For the safety reasons and to select the most suitable operating mode a number of studies and verifications were carried out. They include checks of the mechanical design and integrity and investigations of the pressure drop, the liquid holdup and column performance at different modes. Also, these investigations are very important to evaluate the accuracy of the measurements. The results of these studies are reported here.

Inefficacy of low deuterium content analysis in protium and the fact that possible residual deuterium content of the depleted product can be lower than the

detection limit complicate the verification of separating power. It made us find another approach. The use of gas chromatography method for ortho-para hydrogen analysis was found as a relatively fast and simple way of column performance evaluation. Also, low deuterium probes were measured on the small accelerator built specially for isotope analyses with special ion source giving extremely low backgrounds of hydrogen ions.

II. THE MECHANICAL DESIGN OF THE DEUTERIUM REMOVAL UNIT

The simplified layout of the DRU is presented in Fig. 1. Separation column of 2.2 cm inner diameter and 155 cm overall packing height is the main part of the unit. It consists of two sections connected by ISO CF40 flange coupling and is cased in a vacuum jacket. The 2.5 m vacuum jacket fixed on tripod (Fig. 2) is a basis for the setup. All working parts of the device are placed inside the jacket and its upper and lower attachments (so called adaptors). The jacket and the adaptors comprise a common vacuum volume.

The column has a condenser and a reboiler connected with its upper and lower part. A COOLPOWER 140T (Leybold vacuum GmbH [4]) cryogenerator was used for the cold operations, with the maximal cooling power of 20 W at 20 K.

A conical liquid distributor is fixed between the upper and lower sections of the column. Both sections of the column are filled with random packing. The liquid feed stream (see comments below) is introduced into the bottom of the upper section of the column. The distributor is intended to collect the liquid trickling down from the upper section and guide it to the centre of the lower section. It is essential to prevent the effect of “adhesion” of the liquid to the column’s wall.

The control system provides the algorithms necessary for column’s operation in all modes. It operates two mass-flow controllers (MFC4 and MFC5) and two heaters (H10 and H11) and measures temperature and pressure sensors.

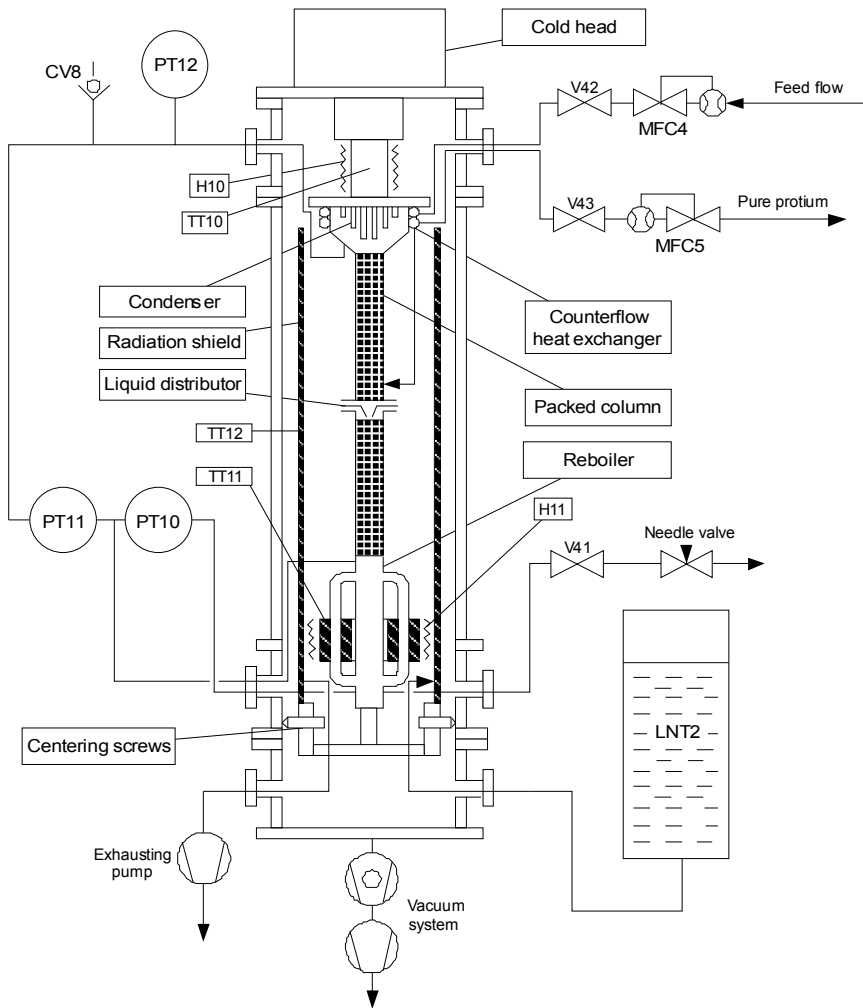


Fig. 1. DRU simplified flowchart

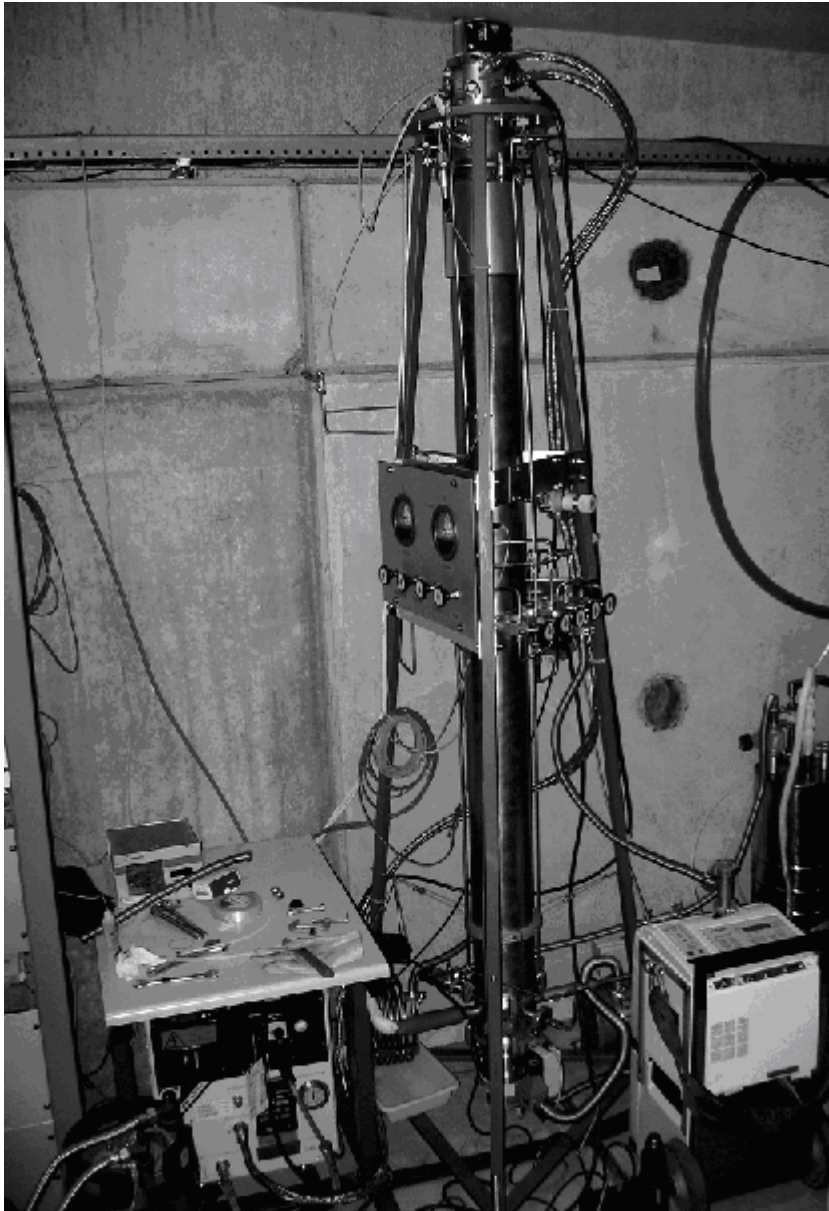


Fig. 2. Deuterium removal unit

Condenser

The condenser is a closed conical bimetallic volume. Its flat upper part is made of 10 mm copper plate. Inner part of this plate is processed in a form of a row of lamellas to develop the heat transfer surface area. Vertical orientation of the lamellas provides easy sliding of hydrogen condensed drops downward. The conical part has a flange for connecting to the column and welded to the copper plate by electron-beam. The condenser has two connections (tubes with 1 mm internal diameter) for conducting product hydrogen and its pressure to an upper point of the differential manometer PT11.

The cold head tightly contacts with outer surface of the copper plate through an indium foil. The cooled part of the condenser is mounted in the upper adaptor and that way in the common vacuum insulation of the column.

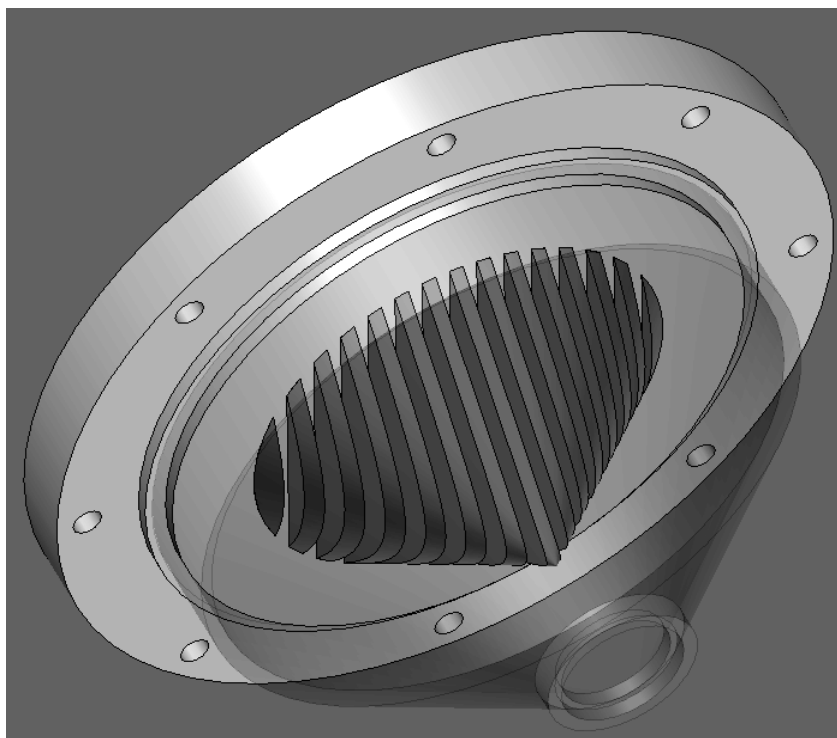


Fig. 3. Condenser

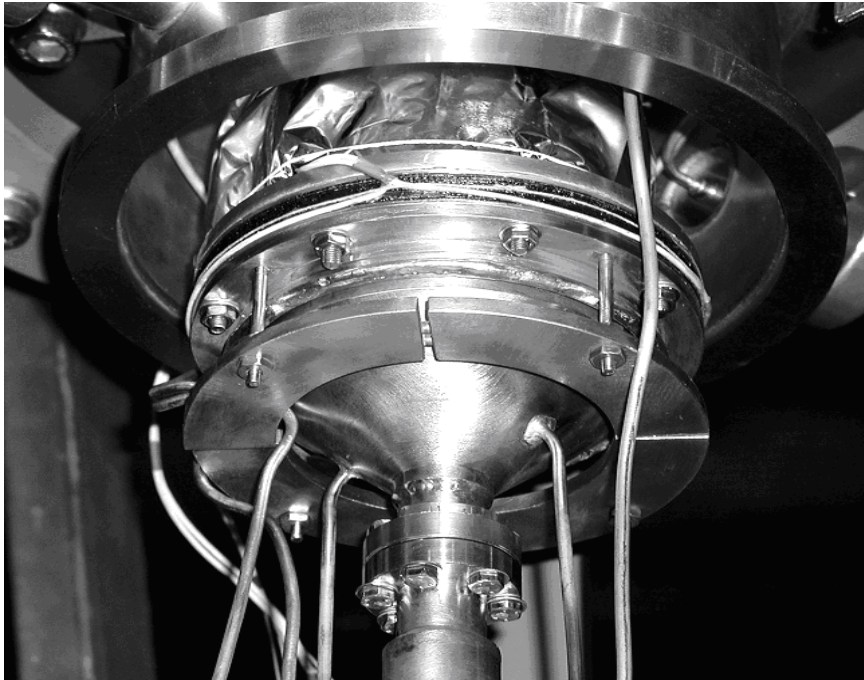


Fig. 4. Condenser with counterflow heat-exchanger

A counterflow heat exchanger is mounted around the condenser and fixed on its upper flange. The assembly of the heat exchanger consists of two copper rings fixed one under another (Fig. 4). The lower ring is cut to four segments to tear the heat flow along the heat exchanger's tubes. Upper one is tightly screwed to the cover flange of the condenser.

The rings are spread by 40 mm by four steel screws and that way thermally unbounded. Both of heat exchanger's tubes are silver-soldered into the lower segmented ring. Outlet tube of the heat exchanger is either soldered into the upper ring.

The heat exchanger cools down the feed flux flow (inlet flow) by the deuterium depleted product flux (outlet flow). This equilibration occurs in the lower ring. Further the feed flux leaves the segmented lower ring and is finally cooled and liquefied in the upper ring by the cooling power of the cryogenerator transmitted through the condenser's cover.

Reboiler

The reboiler (Fig. 5) is essential to supply evaporation of the separating mixture. It has rather complicated arrangement to provide steady controllable boiling. This unit consists of central pipe, copper muff, upper and lower collectors and several connecting tubes. The central pipe keeps the most of liquid hydrogen collected in the bottom of the column. Both of collectors are welded to the pipe. The lower collector is also connected to the boiler by the short connecting tube.

The massive copper muff has a large central hole. Several longitudinal apertures are drilled in the muff around the big hole. Their outlets are opened into common upper and lower grooves which are closed by stainless steel covers. The covers are welded to the boiler's body by electron-beam. Bimetallic structure allows the boiler to have very good heat conductivity owing to the copper body and therewith solid connection with other steel parts through the steel covers.

The boiler is equipped by electric heater coiled around its body and PT100 thermometer (TT11) to control its temperature. Liquid hydrogen boils in the longitudinal apertures and its vapour moves upward through four long connecting tubes. The short connecting tube provides a circulation between the lower collector and the boiler.

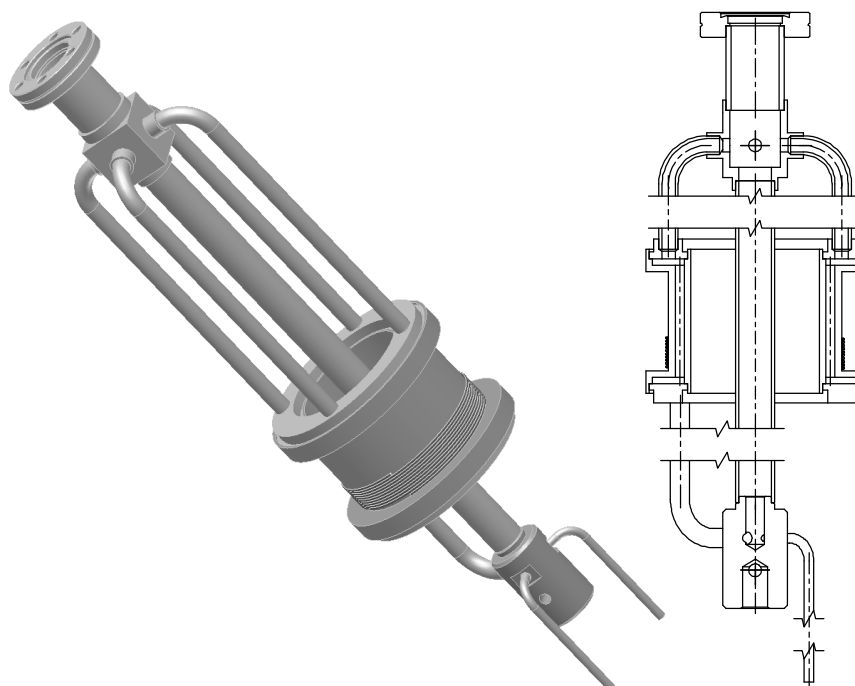


Fig. 5. Reboiler

The advantage of the considered assembling is separation of main amount of liquid hydrogen from the boiling section. Because of this separation, we obtain a steady level of liquid hydrogen inside the central pipe, which is measured by a differential manometer PT10. The low pressure point of this manometer is connected to lower part of the column and the high pressure point – directly to the lower collector.

ISO CF16 and CF40 connections are used for coupling of column parts. This type of connection is characterized by excellent vacuum tightness and good temperature cycling reliability.

Packing

The column is filled with specially designed spiral prismatic random packing made from 0.2 mm stainless steel wire (Fig. 6). The packing is intended to provide a maximal surface of phases contact. At the size of the prismatic springs of 2×2 mm and total volume of packing in the column 560 ml it gives

surface of 1.95 m^2 . The choice of stainless steel as a material is caused by purity conditions of hydrogen. The packing surface was pickled by nitric acid to develop its roughness and improve wettability by liquid hydrogen.

It was the first test of the packing in a cryogenic distillation column. This type of packing (but bigger size – $3 \times 3 \text{ mm}$) was earlier tested in PNPI's water distillation columns of 80 mm inner diameter and showed good separation characteristic (HETP=2.5-3 cm) [5]. The similar packing made from stainless steel (Heli-Pak and Coil Pack) tested in cryogenic distillation column of small diameter gives HETP value about 5-6 cm [6]. That is why the value of HETP equal to 5 cm was put in the design according to conservative approach.



Fig. 6. Packing

III. EXPERIMENTS AND RESULTS

In April – May 2006 the DRU was tested during a few long (up to 7 days) periods of operation. The total amount of pure protium produced by the column in this run was 1300 L. Almost all tests of columns performance were carried out at so called “total reflux mode”, with zero feed flow and correspondingly withdrawal flows from the column top and bottom.

Check on operational capability and heat leakage measurements

After assembling of the setup with serial checking of all hermetic seals on vacuum it was important to test cooling power capability. At first for the exhausted column we measured the steady-state temperature at the top (TT10) corresponding to the given load of the upper heater H10. Then the same was done with two heaters (H10 and H11) and the column filled with hydrogen (Fig. 7). The difference between the curves shown on the figure is explained by the heat leakage and the calculated value is 1.7 Watt. This level of heat leakage was found quite acceptable. It is indicative that the first curve passes through the point (20 K, 20 W) which exactly corresponds to COOLPOWER 140T performance.

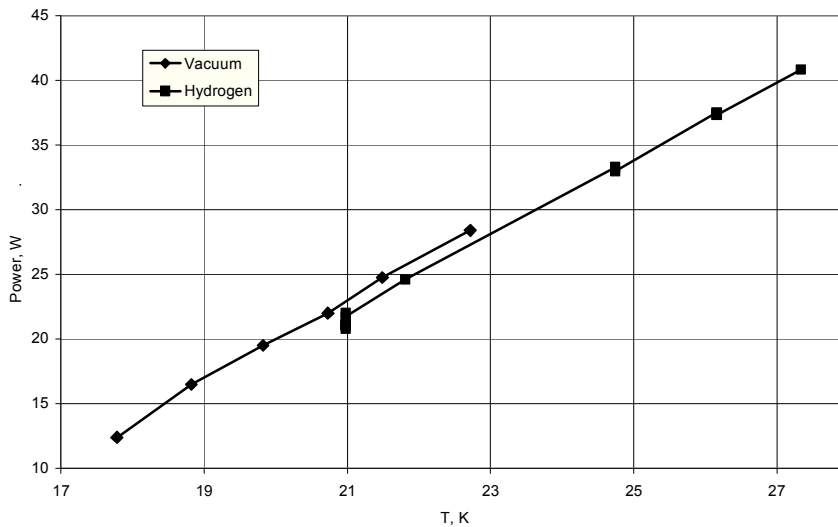


Fig. 7. Heat leakage measurements

Analysis

The analysis of low deuterium content in hydrogen is not prompt and besides the residual deuterium content of the depleted product can be lower than the detection limit. Nevertheless, a method for fast estimation of column performance was strongly needed. A chromatographic analysis of ortho-para isomers of hydrogen was proposed as a method of indirect estimation of the column separation power.

For the analysis the same chromatographic device was used as for analysis of nitrogen traces [7]. However there are some changes introduced into the device for this specific method of analysis. The first distinction was the use of Al_2O_3 -filled chromatographic column immersed into liquid nitrogen. The liquid nitrogen temperature is essential to provide the separation of the isomers that have temperate difference in adsorption affinity. The other essential difference is the direct injection of the sample into the carrier gas without the preliminary accumulation of impurities. The accumulation is unnecessary because of relatively high concentration of the separated components. The third serious property of this method was the use of neon as the carrier gas. It was used to provide more difference in thermo conductivity between the separated species and carrier gas in comparison with conventionally used helium.

Measurement of the concentration of ortho- and para- isomers in the top and bottom parts of the column makes it possible to estimate its separation performance and recalculate the concentration profile for deuterium. An instance of chromatogram for samples taken from the top and bottom part of the column is shown in Fig. 8.

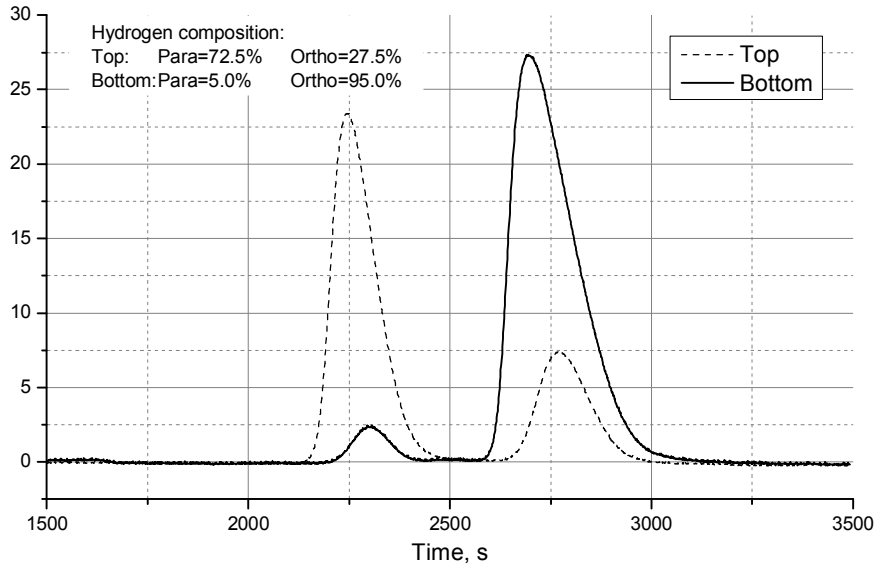


Fig. 8. Chromatogram of separated ortho- and para- hydrogen.
 (Column pressure = 1.2 bar; Reboiler power = 10W;
 Operating mode: total reflux)

Separation ratio is the ratio of relative concentration of a component of interest at the bottom to the top of the column:

$$SR = \frac{X_{Bottom} / (1 - X_{Bottom})}{X_{Top} / (1 - X_{Top})} \quad (1)$$

If separation factors are known for the total reflux operating mode the recalculation of ortho-para hydrogen separation ratio (SR) into deuterium-protium separation ratio can be done easily with the help of Fenske equation:

$$SR = \alpha^N, \quad (2)$$

where N – the number of theoretical plates. A separation factor α is the value of elementary separation effect. It can be presented as a ratio between saturation vapour pressures for pure components. For ortho-para separation it can be shown as:

$$\alpha_{o-p} = \frac{P_{sat.Para}}{P_{sat.Ortho}} \quad (3),$$

where α_{o-p} – ideal separation factor for ortho-para hydrogen system, $P_{sat.Para}$ and $P_{sat.Ortho}$ – saturation vapour pressures above pure para- and ortho-hydrogen, respectively.

Thus, for the deuterium-protium case:

$$\alpha_{D-H} = \frac{P_{sat.H_2}}{P_{sat.HD}} \quad (4),$$

where α_{D-H} – ideal separation factor for system of protium and deuterohydrogen, $P_{sat.H_2}$ and $P_{sat.HD}$ – saturation vapour pressures for pure normal H_2 and pure HD, respectively.

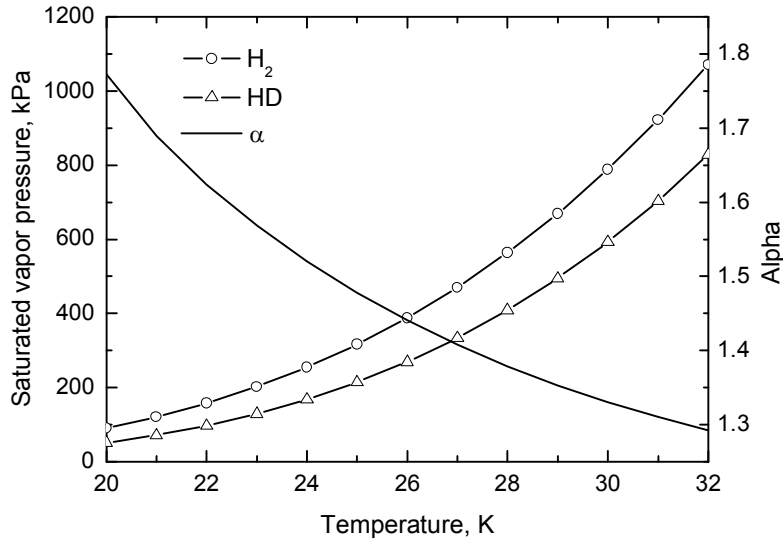


Fig. 9. Temperature dependencies of saturated vapour pressures and separation factor for H_2 -HD system

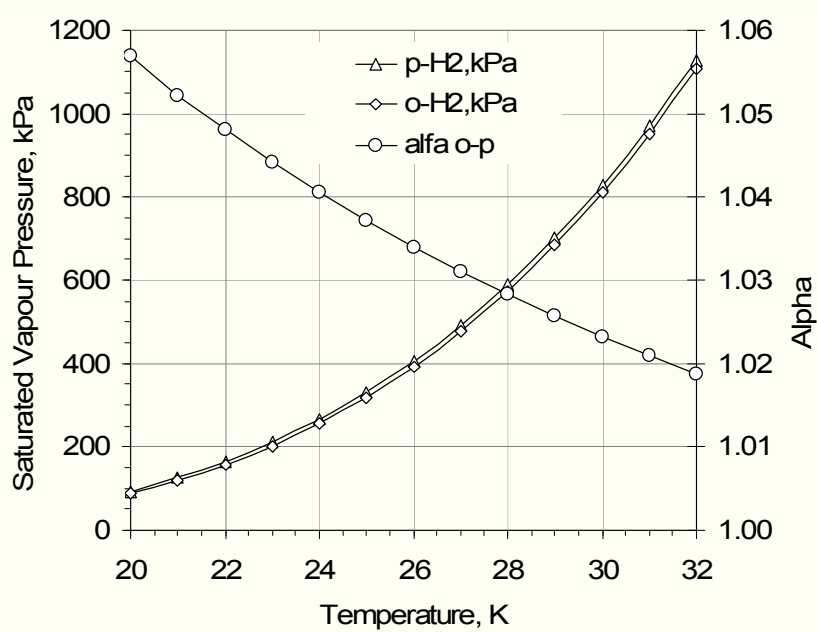


Fig. 10. Temperature dependencies of saturated vapour pressures and separation factor for ortho-para hydrogen system

Dependencies of separation factors for HD-H₂ and ortho-para hydrogen system on temperature are shown on Fig. 9 and Fig. 10. The significant difference between the separation factors according to Fenske equation (2) results in much larger difference between the separation ratios for a large number of theoretical plates in the column (or height of the column: $N=H/HETP$).

Height Equivalent to a Theoretical Plate (HETP) is the main characteristics of column's performance. The higher separation power corresponds to greater number of theoretical plates (N) and less HETP value.

Results of natural gas separation at total reflux mode

First DRU run was carried out with the natural hydrogen with 126.7 ppm of Deuterium. The column was filled by liquid hydrogen in large excess, and after 1 hour operation excess gas (84 liters) was withdrawn from the column bottom. This explains low final deuterium concentration in the bottom (Table 1). Withdrawn gas was sufficiently enriched to change largely the average concentration in the column. Initial gas volume was 181.6 liters.

Negative deuterium concentration in the column top (-1.9 ppm) indicates the accuracy of the measurement. The method consists in extrapolation of sample measurement from deuterium concentration in natural hydrogen and gives low accuracy nearby zero concentration.

Table 1. Natural hydrogen run results

Probe location	Deuterium, ppm	Ortho, %
Original gas	126.7	75
Column top	-1.9	14.6
Middle	-	32.2
Column bottom	56.2	85
Average concentration calculated from the mass balance	7.7	53.5

The experimental data for the ortho-para separation was fitted by the Fenske equation (Fig. 11). The average HETP calculated on the basis of tests with various vapor flow rates was 2.2 cm (Fig. 12), which is comparable with the best world' s results [6].

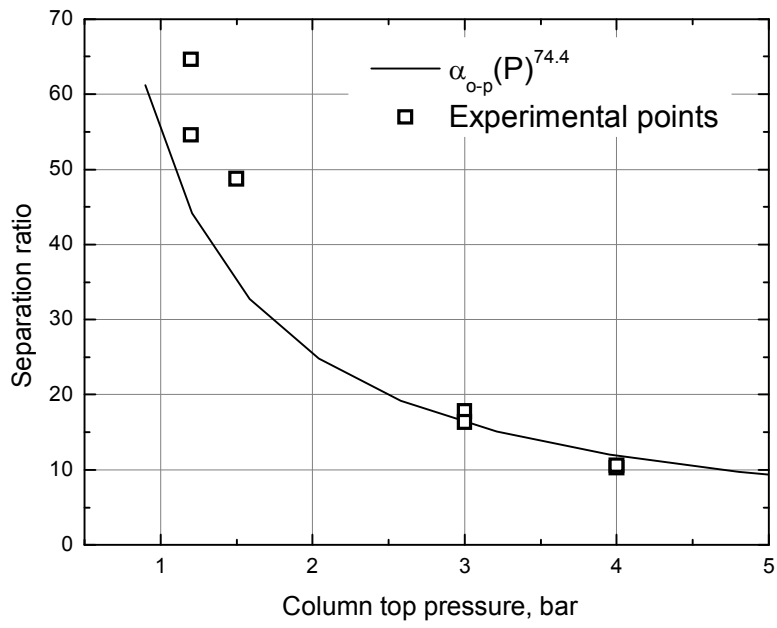


Fig. 11. Using of Fenske equation for total reflux mode

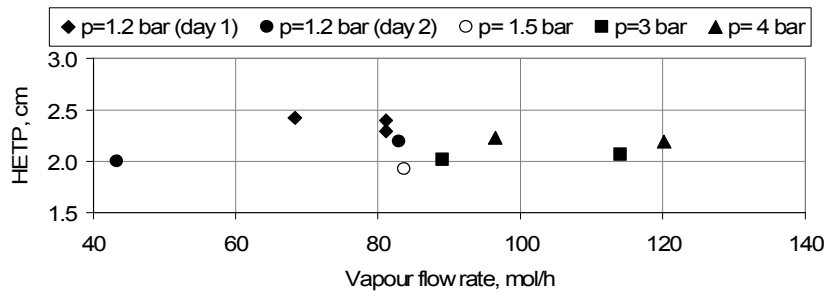


Fig. 12. HETP dependence on vapour flow rate

Studies on the pressure drop and the liquid holdup

Fig. 13 shows the relation between pressure drop across the column and vapour flow rate for the tested mass exchange packing. Unfortunately we did not reach flooding due to relatively large inner column diameter. Original design value was 16 mm, but according to the number of circumstances the inner diameter of 22 mm was chosen, this means that packing was working at relatively low load. We had some anxieties in this respect but fortunately they were relieved due to the excellent packing performance. Packing holdup presented in Fig. 14 had an expected high value. In spite of this the time of reaching a steady state by the column is short – about 1 hour.

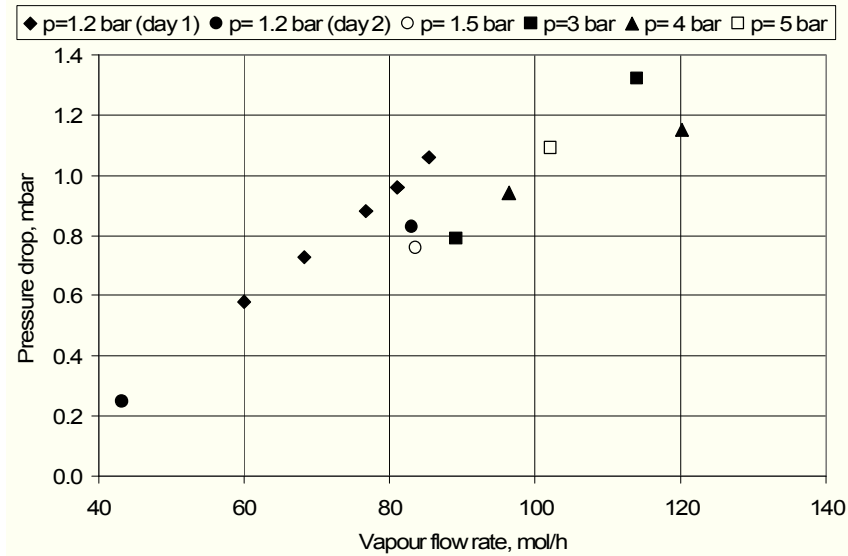


Fig. 13. Pressure drop across the column

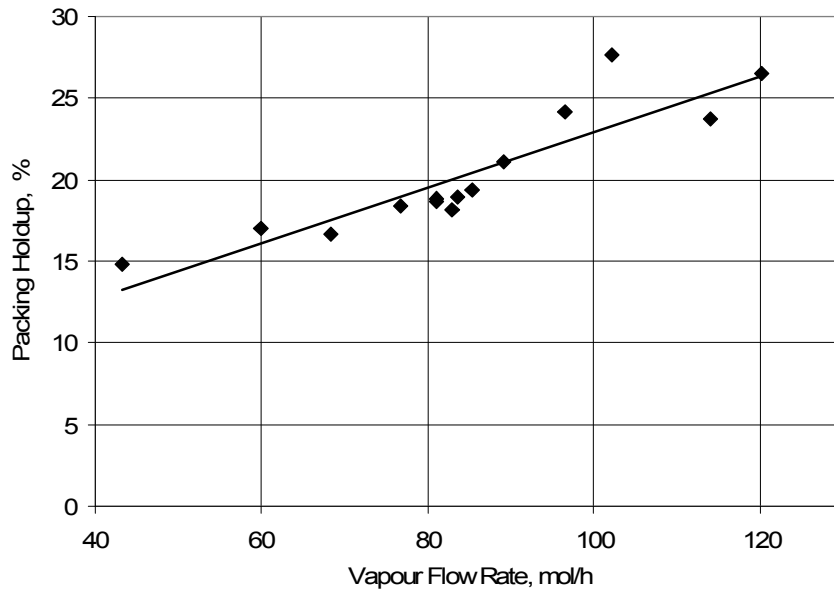


Fig. 14. Packing holdup as a function of flow rate

Production of Deuterium Depleted Protium

There are several possible DRU operation modes for the deuterium depleted protium production. Four modes were tested during experiments: “feed through” with purging, “feed through” without purging, continuous circulation through CHUPS and so-called Raleigh depletion.

During the Raleigh depletion the column was totally flooded by liquefied hydrogen. After the flooding, the hydrogen was discharged through the product line. Natural hydrogen was used only for preliminary tests. The accumulating of required product was carried out from pre-depleted protium.

Mass-flow controllers of CHUPS system were used to operate the feed (MFC5) and for the product flow (MFC4). The rate of the purging flow was adjusted by manual needle valve with the help of bubble flow meter.

The results of the cleaning runs are shown in the Table 2.

Table 2. Results of the cleaning

Mode	"Feed through" no purging	"Feed through" with purging	Rayleigh's depletion	CHUPS Circulation	
Reboiler power, W	20.5	19.0	12.0	20.0	
Pressure, bar	1.5	1.5	1.5	2.0	
Feed flow, l/min	0.81	0.81	1.25	0.55	
Ortho, %	Top	67.8	69.4	-	64.8
	Middle	69.2	74.6	94.2	69.3
	Bottom	96.4	98.5	-	98.2
HD initial, ppm	3	3	10	<3	
HD expected, ppm	Top	< 0.01	< 0.01	0.09	< 0.01
	Bottom	123	~ 200	370	-
HD probe	№ 50	№ 57	№ 61	№ 67	
Amount of gas, SL	572	630	390	420	
Bottom probe HD measured, ppm	57.5 ± 1.2	76.9 ± 1.6	probe lost	16.2 ± 0.5	

In addition, a number of deuterium depleted probes were collected (so called zero protium samples). Expected HD concentrations were calculated using the special program "CD-2004" on the basis of the column's model with known HETP and total height.

The measurements of the probes were performed on the new 200 kV Tandem accelerator built for isotope analysis in Zurich. A special ion source was constructed giving extremely low backgrounds of hydrogen ions from walls, etc. The walls are continuously sputtered to keep the background low allowing measurements during 2 hours. The existence of the zero samples from the DRU system turned out to be crucial, since the accelerator gives a different background if the ion source is not fed with hydrogen gas. First zero sample measurement gave zero deuterium concentration at 0.14 ppm sensitivity. Thus, it contains less than 0.14 ppm of deuterium.

The difference in expected HD concentration and measurement for probes 50 and 57 is most likely caused by wrong assumption about initial HD concentration, which was measured with extrapolation method and has large error. According to probe 50 measurement, the initial HD concentration should be 1.40 ± 0.03 ppm that corresponds to the initial hydrogen measurement on the accelerator (1.44 ± 0.13 ppm).

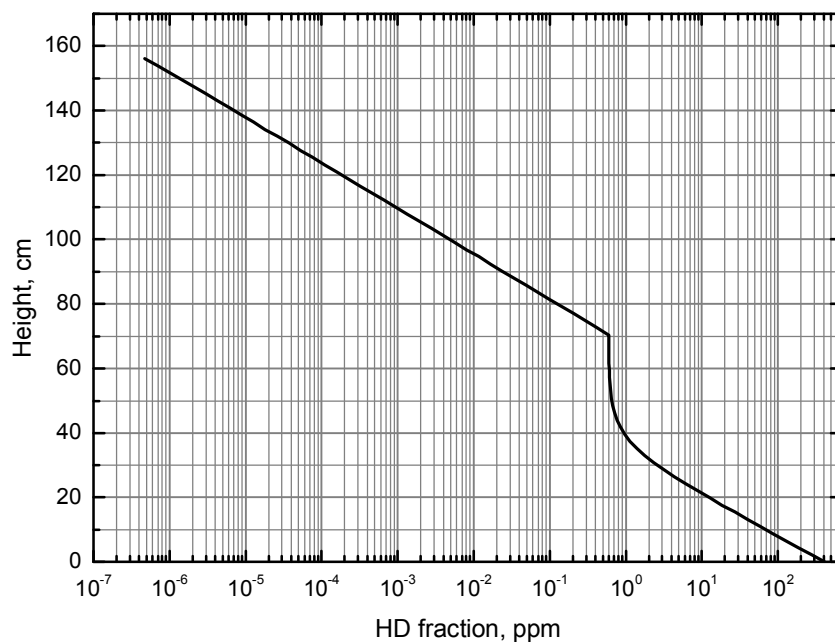


Fig. 15. Expected HD concentration profile.
 Feed flow = 1 L/min; pressure = 1.5 bar; purging flow = 0.015 L/min;
 HETP = 2.2 cm; initial deuterium atomic fraction = 3 ppm.

Deuterium concentration profile in the column, calculated on the base of the ortho- and para-hydrogen separation, gives very low concentration in the top of the column (Fig. 15). Such a low concentration could not be proved by direct measurement because of relatively low sensitivity of the mass spectroscopy.

The additional direct analysis of zero samples using accelerator mass spectrometry in Zurich gave a better result for depleted hydrogen from the top of the column: less than 60 ppb at 60 ppb sensitivity. This is ten times better than technical requirements of MuCAP experiment.

Conclusions

The Deuterium Removal Unit was designed and manufactured in PNPI during the fall and winter of 2005-2006. The final installation, tests and protium manufacturing were carried out in spring, 2006 in PSI. The separation characteristics of the column were studied in details. Due to the absence of direct prompt method of deuterium measurements, the indirect method of chromatographic analysis for ortho- and para- isomers of hydrogen was used to estimate the column's separation efficiency. Some interesting data were obtained.

The height equivalent to a theoretical plate (HETP) for the column is 2.2 cm. It is one of the best ever obtained results for columns of low and medium cryogenic power. For the present column, HETP value is almost constant in a wide range of vapour flow rate.

The column has the separation power in excess for the goal to be sought. Output deuterium concentration lower than 0.1 ppm almost does not depend on the initial concentration (natural hydrogen can be used!).

The pure orthohydrogen useful for some experimental physics applications can be produced by the setup as well as protium.

After the selection of column's operating parameters and the development of its operating algorithm the protium production run was performed. Several modes of isotope purification were used. As a result about 1.3 m³ of deuterium depleted hydrogen was obtained. CHUPS system was put into the mode of continuous hydrogen circulation through the column to remove deuterium.

The productivity of the system in the "feed through" mode is 500 L/day. In the mode of circulation through CHUPS system it comes to 150 L/day. The mode of Rayleigh depletion is considered to be inefficient because of small initial amount of hydrogen in the column.

The method of measurement of low deuterium content in hydrogen (1 ppm and less) is suggested. It is based on deuterium enrichment by a factor of 40 (and even more) in the reboiler.

This work is also of great importance for Detritiation Plant of PIK reactor as in accordance with a new developing design of the plant the cryogenic column with close parameters is needed. A good packing was developed and tested, successful experience was gained.

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