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Canadian Fusion Fuels
Technology Project

ITER TASK T48 (1994)
LOW-INVENTORY CRYOGENIC DISTILLATION TESTS

CFFTP G-9504
January, 1995

K. Woodall[1], J. Robins[1], D. Bellamy[1], R. Gnoyke[1], S. Sood[2], C. Fong[2], and P. Gierszewski[3]
Ontario Hydro Technologies[1], Ontario Hydro Nuclear[2], Canadian Fusion Fuels Technology Project[3]

CFFTP GENERAL

The Canadian Fusion Fuels Technology Project represents part of Canada's overall effort in fusion energy research. The focus for CFFTP is tritium technology and remote handling. The Project is funded by the Government of Canada and Ontario Hydro Technologies. Ontario Hydro Technologies administers the Project.

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1. EXECUTIVE SUMMARY

Previous work at Ontario Hydro Technologies (OHT) had shown that small cryogenic columns could be stably controlled and designed to much lower inventories than had been previously thought possible. Among the results were measurements of Height-of-Equivalent-Theoretical-Plate (HETP) versus holdup for Heli-Pak A and B in columns up to 20 mm diameter. ITER cryogenic distillation column designs suggest that the final high-tritium columns could be 30-70 mm diameter. The objective of this ITER task was to design and construct a column section for demonstration of scale-up of low inventory cryogenic distillation.

The experiments were to be carried out in an upgraded Cryogenics Distillation Laboratory at OHT, in the facility used for previous low-inventory column tests. The ITER scaled-up test system has the following characteristics: 55 W condenser capacity; 30-mm diameter column loaded with Helipak B; 1500 mm packed height.

The first task was to design and build the scaled-up test facility. Most of the changes were straightforward. In order to reduce costs, it was necessary to use existing 30-35 W helium refrigerators. Therefore, to provide 60-W duty to the scaled-up column, the two refrigerators had to be well-coupled thermally, but not mechanically, since the refrigerator cold heads have very thin shells. The solution was to attach the column firmly to one cold head, and indirectly to an adjacent cold head through flexible copper braid. Several iterations were required to obtain the desired good heat transfer with flexible mechanical connection. This facility is now operational and ready to begin measurements on the 30-mm column.

Also during 1994, the Princeton Tritium Processing System (TPS) was installed and commissioned. The results from this experience are relevant to the ITER distillation system.

The implications of the 1994 results are:
- Staged diffusers provide efficient, clean and low-inventory separation of hydrogen suitable for cryogenic distillation feed;
- Column wetting procedures can be applied to multicolumn cryogenic distillation systems in order to minimize HETP;
- The FLOSHEET code has been further benchmarked;
- Multiple columns can be stably controlled using inventory control;
- Small columns should not be closely coupled to large columns, in order to improve the stability of the smaller column.

Future experimental work should:
- Complete characterization of 30-mm column, including measurement of HETP versus inventory over a range of operating conditions;
- Test OHT improved low-inventory packing (potentially 20% improvement);
- Test the interaction between the upper (large diameter) and lower (small diameter) section of a column with an intermediate reboiler, as an option for reducing the ITER tritium inventory.

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2. TASK OBJECTIVES

The objective of this ITER task was to design and construct a column section for demonstration of scale-up of low inventory cryogenic distillation.

The anticipated results are:
1. Determine smallest column diameter that can handle full boilup of 60 W with Helipak;
2. Measure D and T inventory in this column;
3. Measure HETP for this column as a function of column inventory and column boilup;
4. Combine these measurements with previous from small diameter columns to provide reference estimates of HETP versus column diameter, and extrapolate the results to ITER reference conditions to indicate minimum expected ITER tritium inventory.

3. THEORY

Minimizing the tritium inventory in a cryogenic distillation system requires attention to several factors:
- optimum combination of column diameter, packing and operating conditions to minimize holdup per stage;
- column operating conditions and configuration (position of equilibrators) to create tritium concentration profile that is pushed to the bottom of the final column;
- reboiler design that has the minimum liquid inventory necessary for stable operation;
- tapered cascade (multiple columns or tapered column).

Some of these parameters must be empirically determined, and then an optimized design (or operating scheme) can be found with the assistance of computer codes such as FLOSHEET [1].

Previous work at Ontario Hydro Technologies (OHT) had shown that small cryogenic columns - as low as 4.6-mm diameter - could be stably controlled and designed to much lower inventories than had been previously thought possible. Among the results were measurements of Height-of-Equivalent-Theoretical-Plate (HETP) versus holdup for Heli-Pak A and B in columns up to 20 mm diameter and 30-35 W condenser duty. These results were used to design and build the Princeton Tritium Purification System, with a predicted tritium inventory of < 1 g [2] The Princeton purity requirement is 99%, but there should be sufficient plates to produce 99.9% T.

ITER cryogenic distillation column designs suggest that the final high-tritium columns could have 70-200 W condenser duty, with 30-70 mm diameter. Therefore, in order to extend our previous results to ITER with confidence, tests on larger diameter columns were required.

4. EXPERIMENT DESCRIPTION

The experiments are to be carried out in the Cryogenics Distillation Laboratory at OHT, in the facility used for previous low-inventory column tests. The cold box was enlarged, and the larger column and cooling system assembled. The control system was upgraded. Existing mechanical helium refrigerators are used.

Figure 1 illustrates the experimental setup (note that feed and sample points are interchangeable). The system has the following characteristics:
- Approx 55 W condenser capacity;
- 30-mm diameter column loaded with Helipak B;
- approx. 1500 mm packed height.

The instrumentation is as follows:
- 8 temperature diodes located on the cooling system, condenser, mid-column and reboiler;
- 4 absolute pressure gauges;
- 2 differential pressure gauges (packing liquid inventory and reboiler liquid level).

The control is provided by:
- 4 flow controllers (flow into and out of the column, and in tritium sampling system);
- 4 feed, withdrawal or sampling points along the column;
- 486 computer with Labview software for data acquisition, analysis and control, with sampling frequency of about 0.5 Hz.

The column will be fed with a D_2/DT mixture at a feed rate up to 13 mol/hr. Under these conditions, the reflux ratio will vary up to 17. The DT concentration will be restricted to trace levels in the feed, but tritium will be injected at above background levels in order to reduce the time required to setup the tritium profiles in the column.

The number of theoretical plates in the column will be determined as follows. A gas sample is withdrawn from the column at the product end and oxidized to water. This water is trapped in a scintillation vial, weighed, scintillation cocktail added, and then the tritium is counted. Each water sample is collected for about 1.5 hours to ensure representative results. The product tritium level is thus determined to within 3%. Since the DT concentration in the column changes by about 20% per theoretical plate, accurate estimates for the number of plates are obtained by comparing experimental results with FLOSHEET simulations.

5. RESULTS

5.1 30-mm Column Design/Fabrication

The first task was to design and build the scaled-up test facility. Most of the changes were straightforward. However, the refrigeration system design was more complex. In order to reduce costs, it was necessary to use existing 30-35 W helium refrigerators. Therefore, to provide 60-W duty to the 30-mm column, two refrigerators had to be well-coupled thermally to the column condenser. But since the refrigerator cold heads have very thin shells, it was not possible to have them rigidly coupled to eachother (the thermal contraction down to cryogenic temperatures alone would be sufficient to destroy them). The solution was to attach the column firmly to one cold head, and indirectly to an adjacent cold head through flexible copper braid. Several iterations were required to obtain the desired good heat transfer with flexible mechanical connection.

The present design of thermal connection is based on several layers of high-conductivity copper braid, interlaid with indium foil, and precompressed together tightly to form the connections at the cold head and column condenser. Tests indicate that this system is able to provide 55 W condenser duty. This is sufficient for present tests, although we believe that some further improvements are still possible.

General drawings of the frame and cold box, top flange, and column are provided in Figures 2-4.

5.2 30-mm Column Tests

In the range of column diameters expected for the ITER high-tritium column, Heli-Pak B would be our recommended low-inventory packing, based on our small-diameter column hydrogen data and on the manufacturer's published hydrocarbon data. Figure 5 summarizes this hydrocarbon data and our previous measurements with Heli-Pak B. Figure 5 also illustrates our assumed values of HETP for FLOSHEET modelling purposes. Specifically, we use a H_2 HETP of 2.5 cm for columns below about 25 mm diameter, and expect that the H_2 HETP at 50-mm diameter would have increased slightly to 2.5-3 cm. The present experiments will verify these assumptions up to 30-mm diameter column. This corresponds to the expected smallest practical diameter column for 60 W condenser duty.

The reference 30-mm column tests will be based on the following nominal conditions:

- 900 torr pressure;
- 5 L/min feed rate;
- 55 W condenser power;
- total reflux;
- Heli-Pak B;
- D_2 feed with trace DT.

A FLOSHEET simulation of a possible experiment is attached (Figure 6). The simulation shows a 60-plate column fed 15 plates below the condenser. The column is operated with 5 L/min feed, and 1 mL/min bottom drawoff, with 55 W condenser power. Note that the bottom DT concentration is enriched by almost 4,000 while the top DT concentration is depleted by about a factor of 5, with most of the DT residing in the bottom of the column.

Development of the control systems were carried out on a 4.6-mm column at OHT, in preparation for the 30-mm column tests. Also in other commissioning tests, procedures for wetting the 4.6-mm column were tested. Figure 7 illustrates the effect of startup procedure on the packing performance. In the initial set of experiments, the column was brought to equilibrium at its normal operating conditions. The tritium and total Q_2 (liquid) inventory (measured by column pressure drop) are shown in Figure 7 in arbitrary units. In the second set of experiments, the Q_2 inventory in the column was deliberately increased to completely wet the packing. During this phase, the tritium product concentration increased - indicating that the column was operating with more theoretical plates (or that the HETP had decreased). In the third and fourth set of experiments, the Q_2 inventory was reduced, but the tritium product concentration continued to increase (i.e., HETP continued to drop). Lowering the Q_2 inventory back to the original operating conditions, the column continued to produce - in steady-state - a tritium product concentration 1.6 times larger than before the packing treatment. Note that lowering the Q_2 inventory further finally starts to lower the product tritium concentration, presumably because the packing is starting to dry out. This process enables us to obtain the maximum number of theoretical plates from the column while minimizing the tritium inventory per plate. This (and other) processes were also applied successfully during commissioning the Princeton TPS system in 1994, and will be tested on the 30-mm ITER column at OHT.

5.3 Princeton TPS Four-Column System

During 1994, the Princeton Tritium Processing System (TPS) was installed and commissioned. The results from this experience are relevant to the ITER cryogenic distillation system, and are briefly summarized here. Figures 8 and 9 are flow schematics for the Princeton system permeator and cryogenic distillation systems.

The Princeton system uses a staged diffuser configuration to provide clean hydrogen feed to the cryogenic distillation system. This configuration is similar to that proposed for the ITER fuel cleanup system front-end. It has worked very well, with performance near its theoretical limit. For example, in commissioning tests with 120 NL/hr of 33% N_2, 50% D_2 and 17% H_2 at 1400 torr, the waste stream was essentially pure N_2, with only 1400 ppm Q_2.

For the reference commissioning case described above, the H_2 product from Column 1 contained < 0.1% HD, and the D_2 product with Column 3 contained < 0.1% HD (the purity of the product could not be read to greater accuracy).

Commissioning was largely as expected, with some minor problems that have been solved. For example, thermal radiation onto the small capillary return line from CD3 to CD1 has reduced its capacity, but this will not limit the performance for Princeton conditions.

One important observation is that inventory control provides stable control of the system, including the fourth small (4.6-mm diameter) high-tritium column CD4. To optimize performance and minimize tritium inventory, the Q_2 inventory in each column must be controlled (the Q_2 level is measured from the pressure drop across the column). The Q_2 inventory in CD1 is controlled by adjusting the sum of the draw-off rates for the H_2 (top of CD1) and D_2 (top of CD3) streams. The split between H_2 and D_2 drawoff is controlled so that a constant D_2 concentration is maintained in the center of CD1 (measured by the temperature and pressure in the center of CD1). If the D_2 level is too high, the D_2 drawoff rate is increased and the H_2 rate is decreased. Inventory control in columns 2,3 and 4 is simpler. If the column pressure drop signal is above the set point, indicating too much Q_2 inventory, the condenser temperature is raised by the control system. This increases the pressure in the column and increases the flow out of the column. (In the Princeton TPS system, the feed-forward flows are pumped, but the feed-backward flows are controlled only by the pressure differecne between the two columns.) Figure 10 shows an example of stable four-column control during commissioning tests at Princeton.

The final small column CD4 is now operating more smoothly than during the previous commissioning tests at OHT. Part of this improvement is because it has been found better to provide some degree of isolation between the small column and the other larger ones. This has not been an issue on Darlington-scale cryogenic distillation columns, but - at Princeton system scale - pressure fluctuations in column CD2 were translated into much larger oscillations in column CD4. These prevented Column CD4 from running as close to its optimal point as desired. The solution in the Princeton system was to reduce the diameter of the return line from CD4 to CD2 to provide a pressure drop, which also meant that CD4 operated at a higher pressure than CD2 (approx 1100 torr rather than 900 torr).

6. DISCUSSION

Relative to the task objectives (Section 2) , we have carried out the following:

1. Designed, built and commissioned a 30-mm cryogenic column test facility;
2. Combined measurements from OHT and from PPPL TPS experience to provide reference estimates of HETP versus column diameter for Helipak B;
3. Applied these results, with FLOSHEET, to optimize the ITER design and reduce the tritium inventory (from 180 g to 130 g, based on the Sept 1994 reference design).

We have not yet measured the inventory and holdup versus HETP for the 30-mm ITER test column. Commissioning tests for other cryogenic distillation columns has also provided relevant information on diffusers and on column design.

The implications of the 1994 results are:

- Staged diffusers provide efficient, clean and low-inventory separation of hydrogen suitable for cryogenic distillation feed;
- Column wetting procedures can be applied to multicolumn cryogenic distillation systems in order to minimize HETP;
- The FLOSHEET code has been further benchmarked;
- Multiple columns can be stably controlled using inventory control;
- Small columns should not be closely coupled to large columns, in order to improve the stability of the smaller column.

Future experimental work should:

- Complete characterization of 30-mm column, including measurement of HETP versus inventory over a range of operating conditions;
- Test OHT improved low-inventory packing (potentially 20% improvement);
- Test the interaction between the upper (large diameter) and lower (small diameter) section of a column with an intermediate reboiler, as an option for reducing the ITER tritium inventory;
- Further optimize the reference ITER cryogenic distillation system design based on this data.

The new OHT packing has been shown in scoping tests with water (for water distillation purposes) to have a surprisingly low HETP, and therefore should be tested with hydrogen also. The test of a column with an intermediate reboiler is based on our observation, during commissioning of the Princeton TPS, that closely coupling a large and small column creates a less stable environment in the small column. A split column with intermediate reboiler should be equivalent to such a large-small column pair, and there have been no tests of split hydrogen distillation columns on an ITER-relevant scale. Appropriate tests of both the new packing and of a split column can be carried out in the present OHT Low-Inventory Cryogenic Distillation test facility.

7. REFERENCES

1. A. Busigin and S.K. Sood, "FLOSHEET - A computer program for simulating hydrogen isotope separation systems", Fusion Technology 14 (1988) 529.

2. A. Busigin, C. Busigin, F. Adamek, K. Woodall, J. Robins et al., "Control system implementation for a complex low-inventory cryogenic distillation system for Princeton TFTR", Proc. 18th SOFT, Karlsruhe (1994).

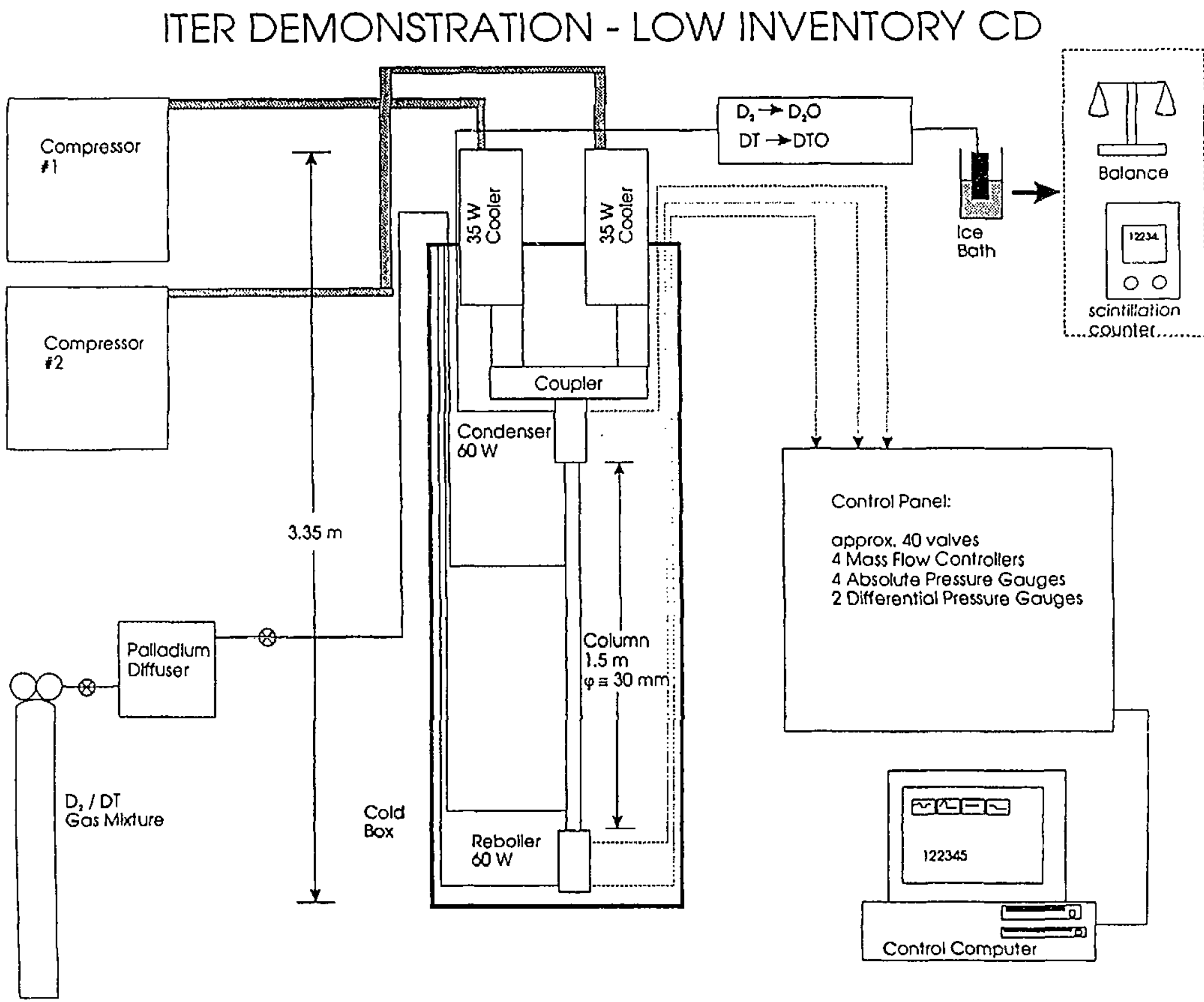


Figure 1. Schematic diagram of Low-inventory Cryogenic Distillation test facility at Ontario Hydro Technologies.

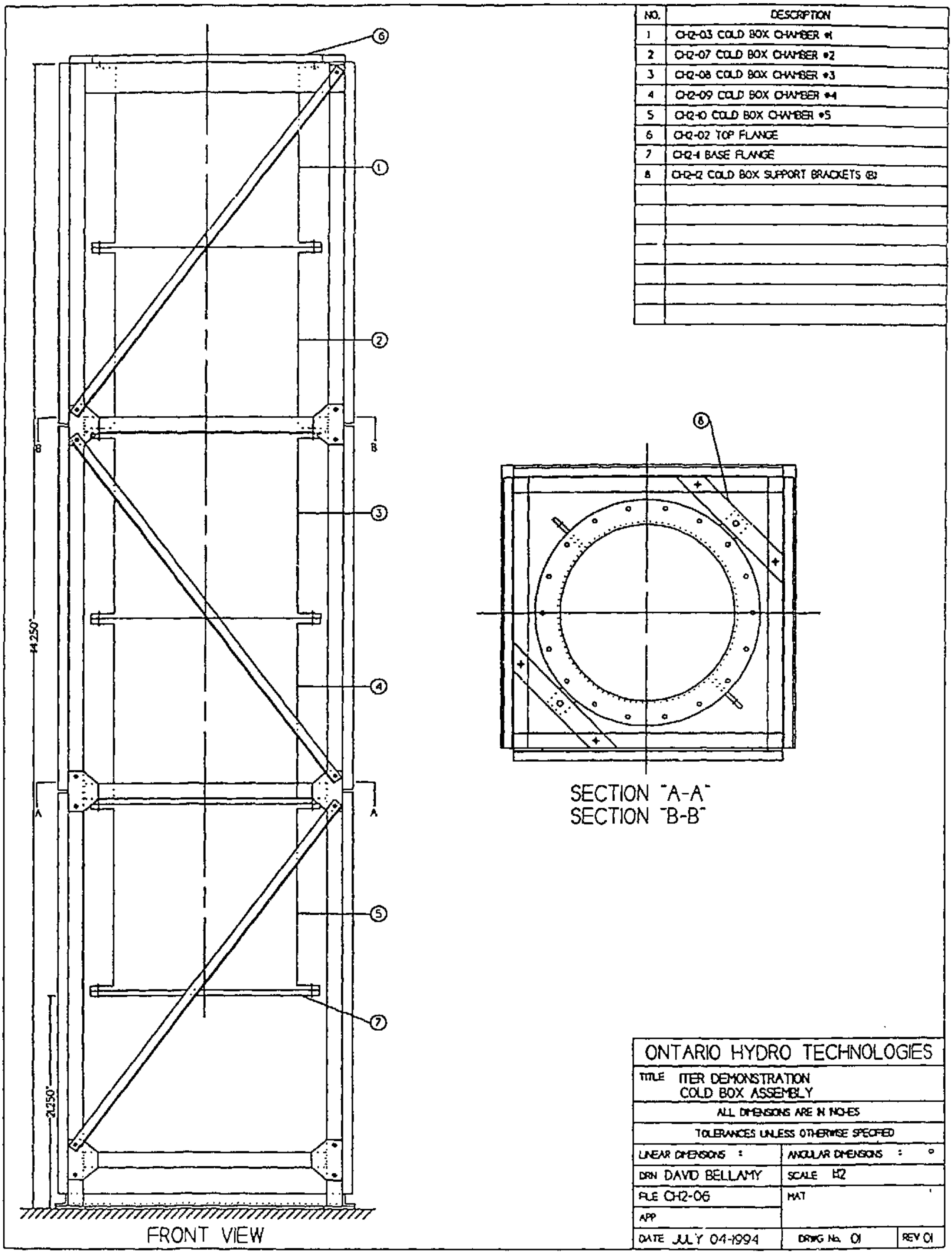


Figure 2. Drawing of cold box and frame.

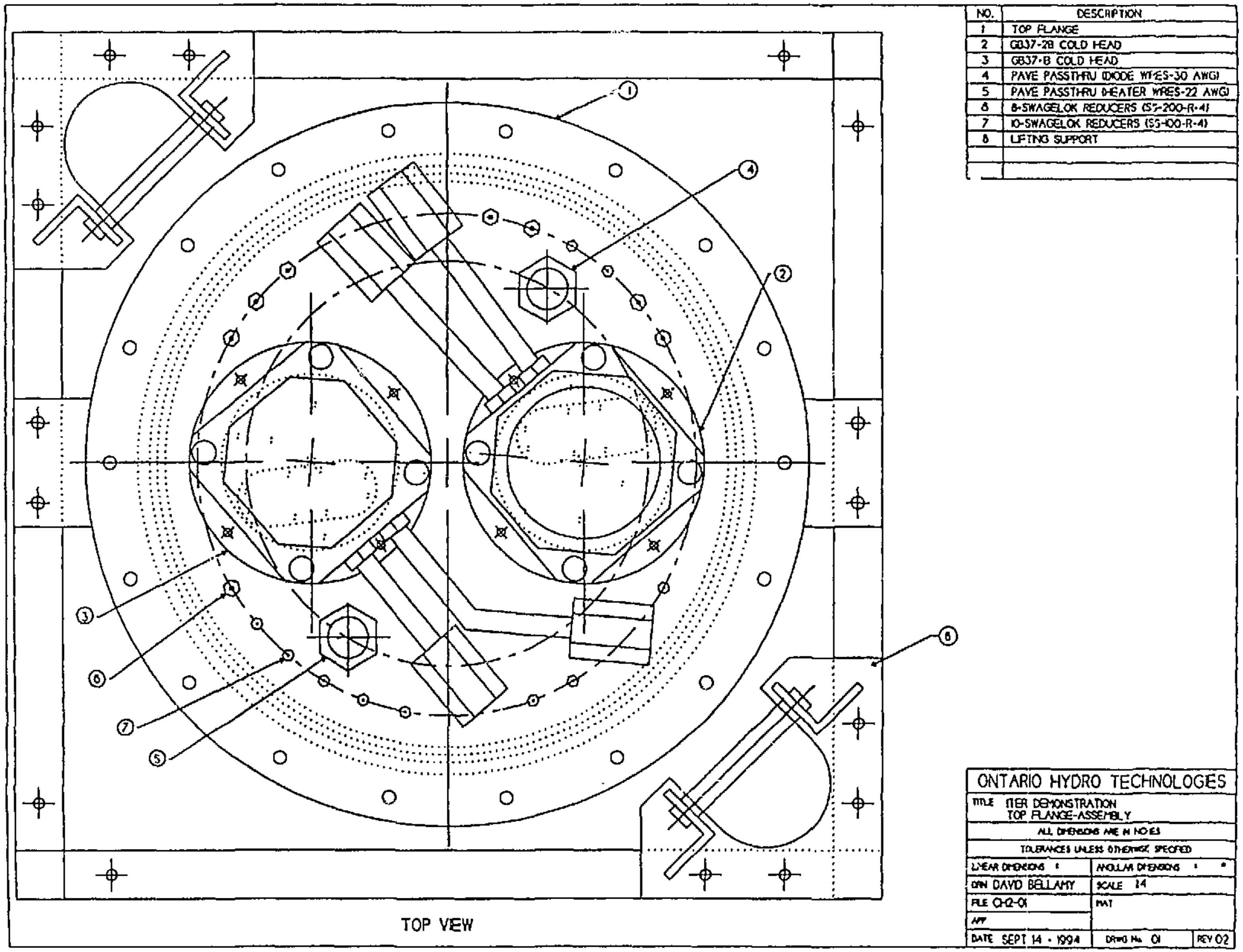


Figure 3. Drawing of the cold box flange.

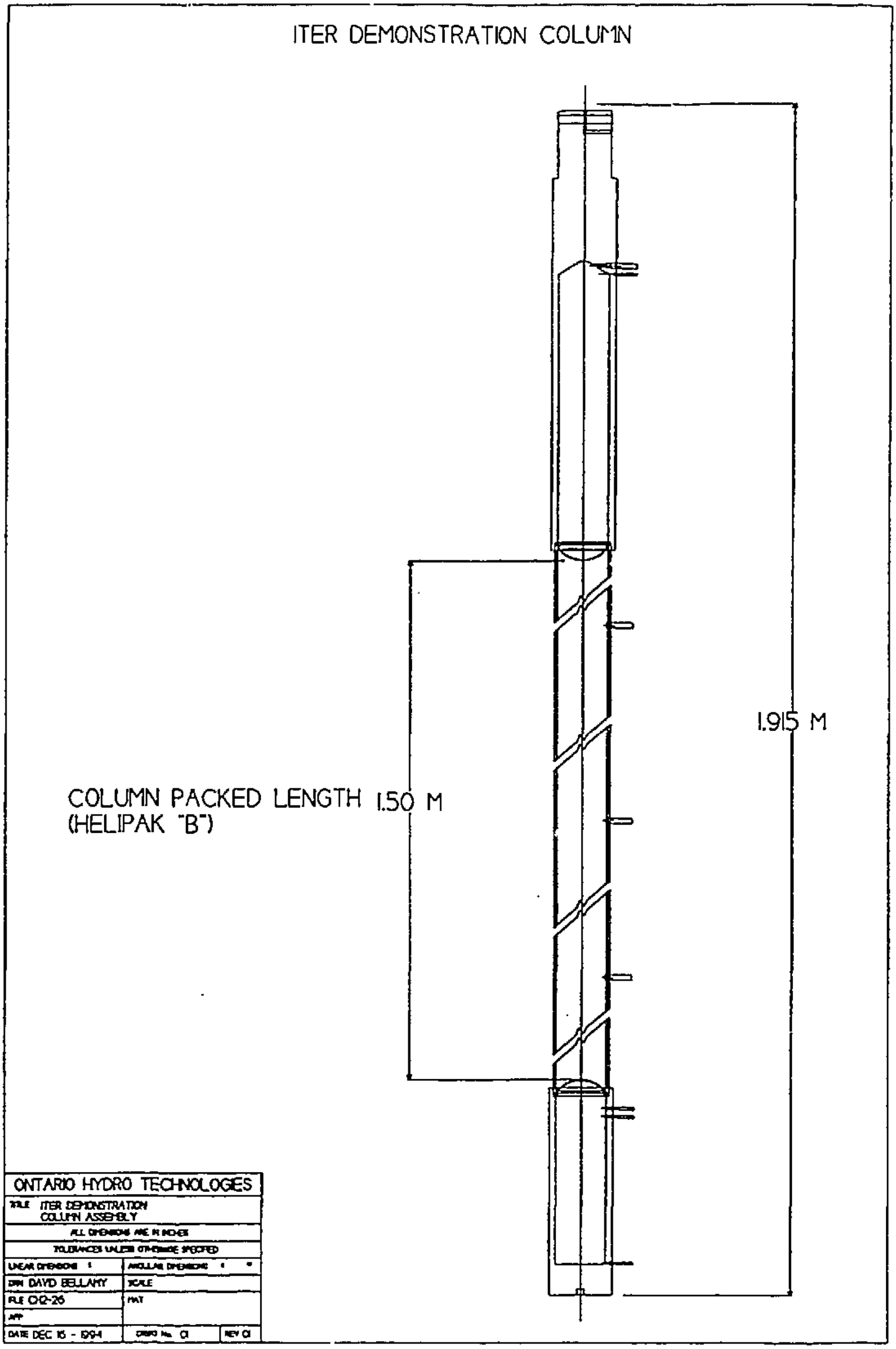


Figure 4. Drawing of column.

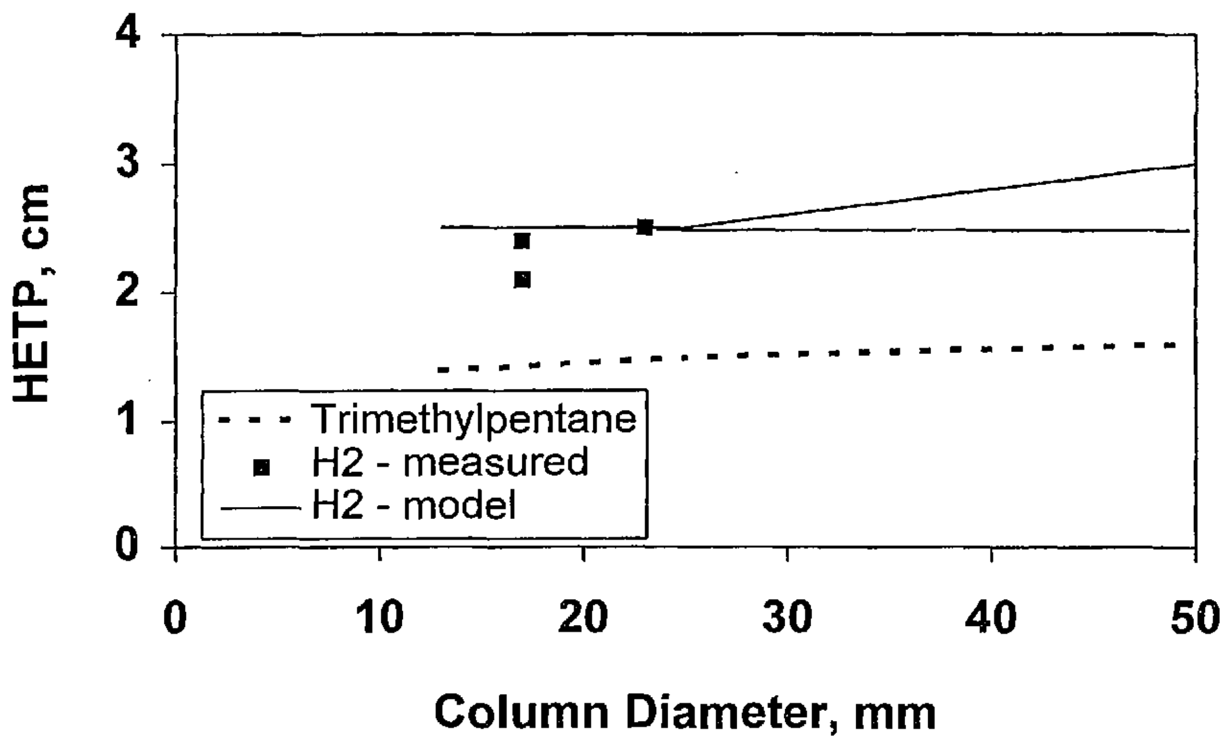


Figure 5. Summary of HETP versus column diameter estimates for Heli-Pak B.

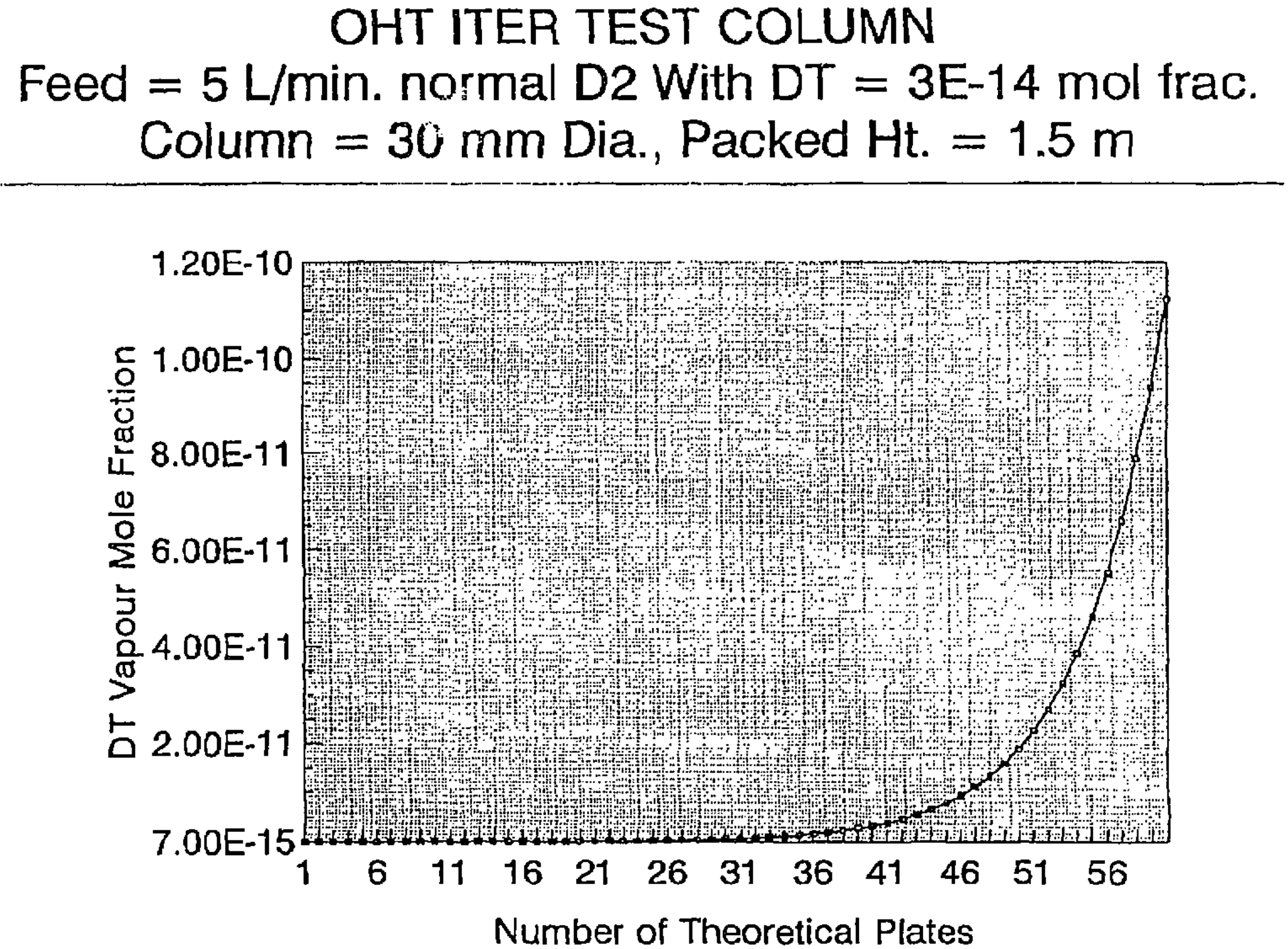


Figure 6. Example DT concentration profile in 30-mm column test (from FLOSHEET).

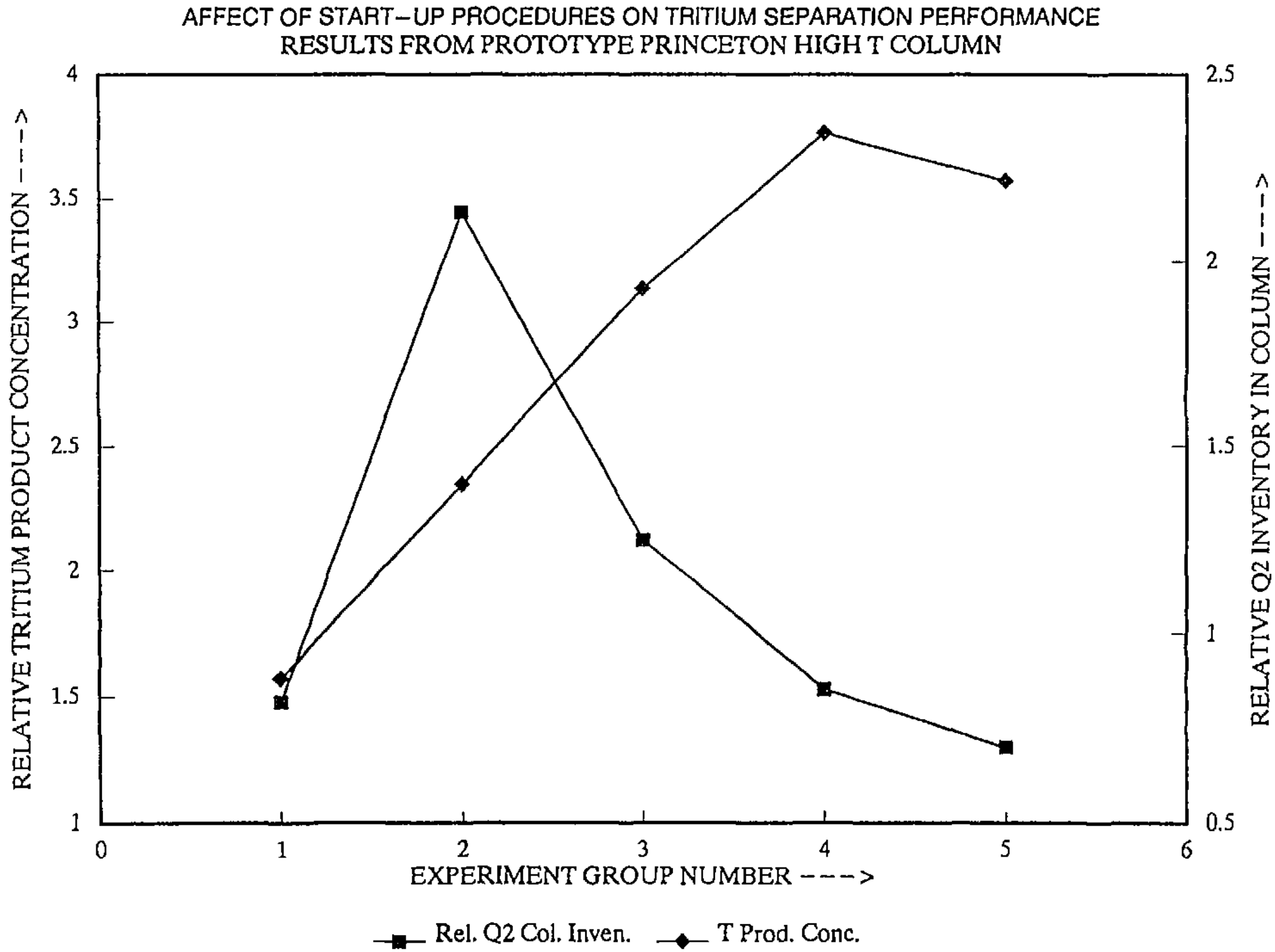
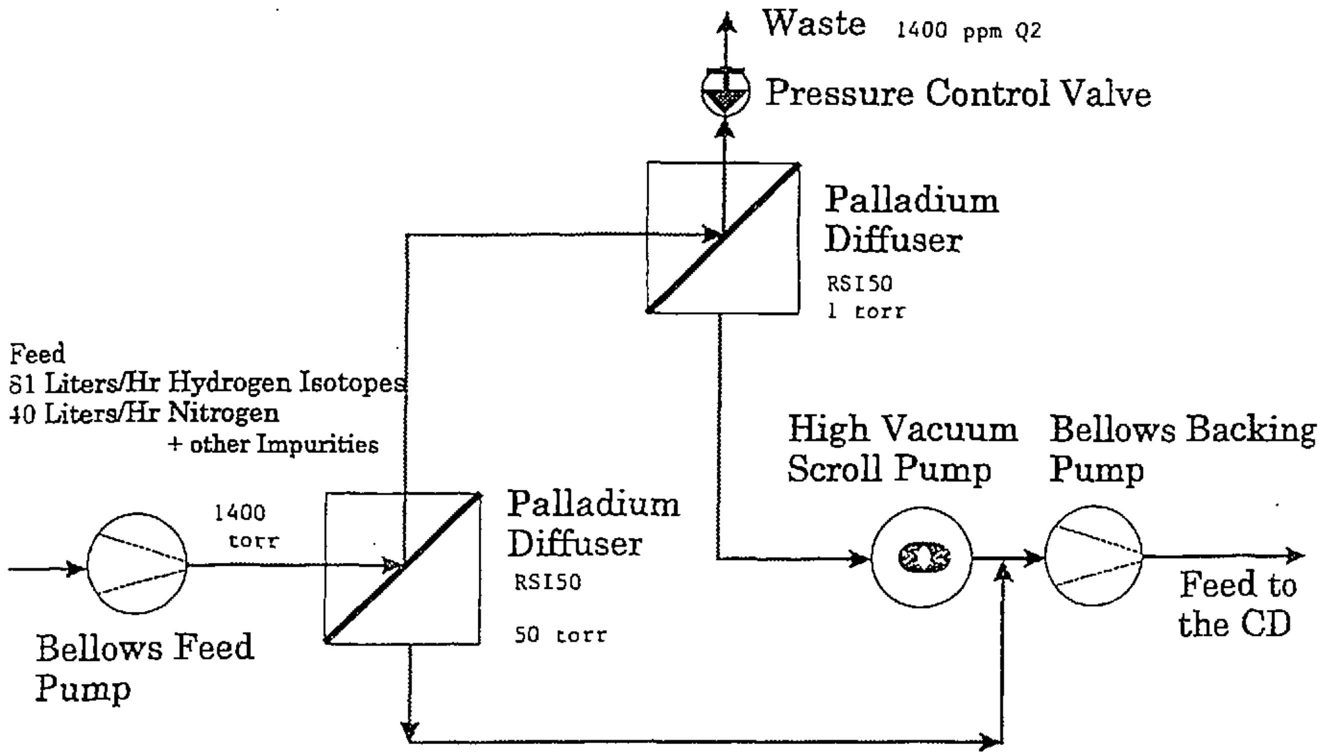


Figure 7. Effect of start-up procedure on tritium separation performance (results from 4.6-mm column tests).



Tritium Processing System
Princeton Plasma Physics Laboratory
Feed Treatment System

Figure 8. Flow schematic of PPPL TPS front-end staged diffuser system.

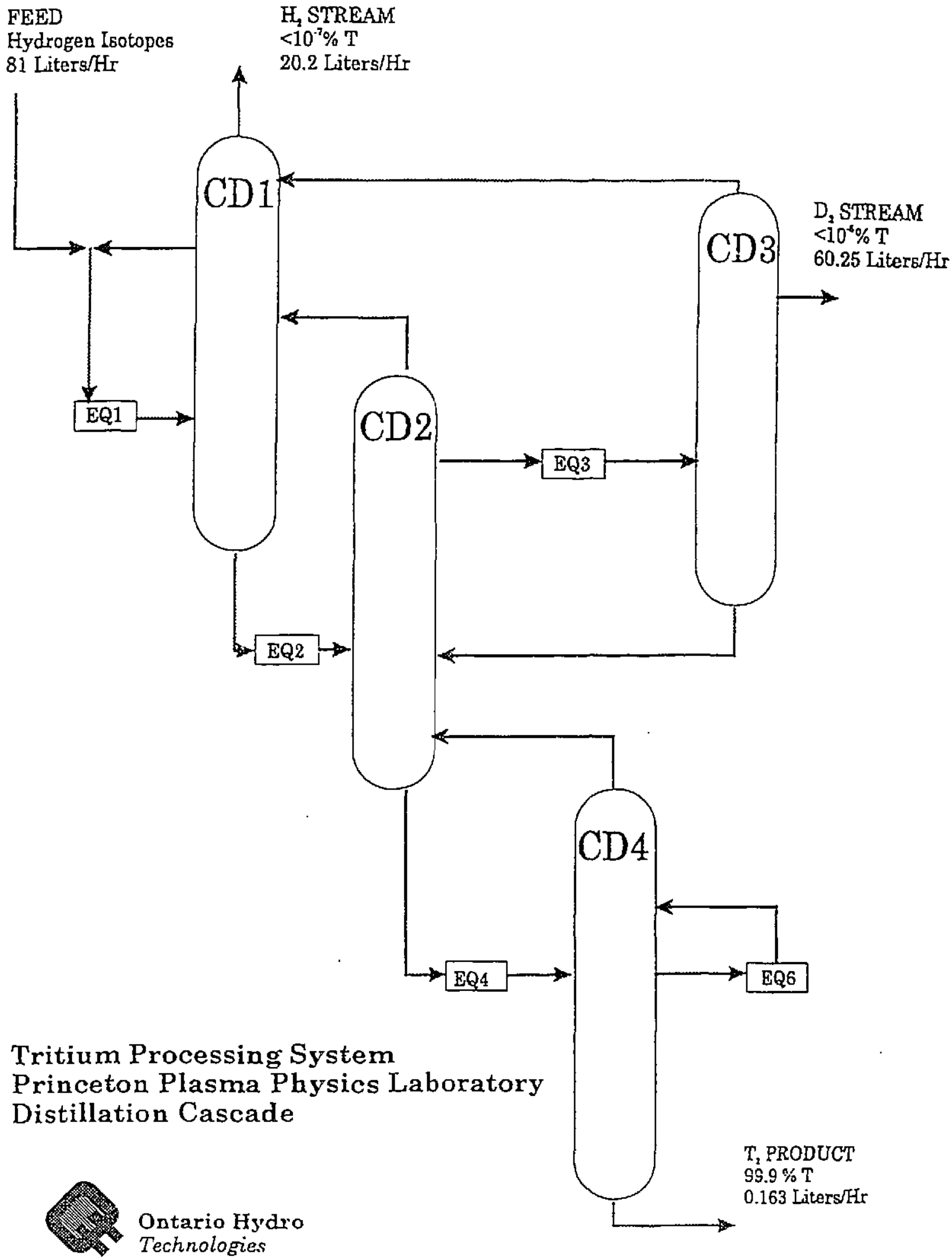


Figure 9. Flow schematic of PPPL TPS cryogenic distillation system.

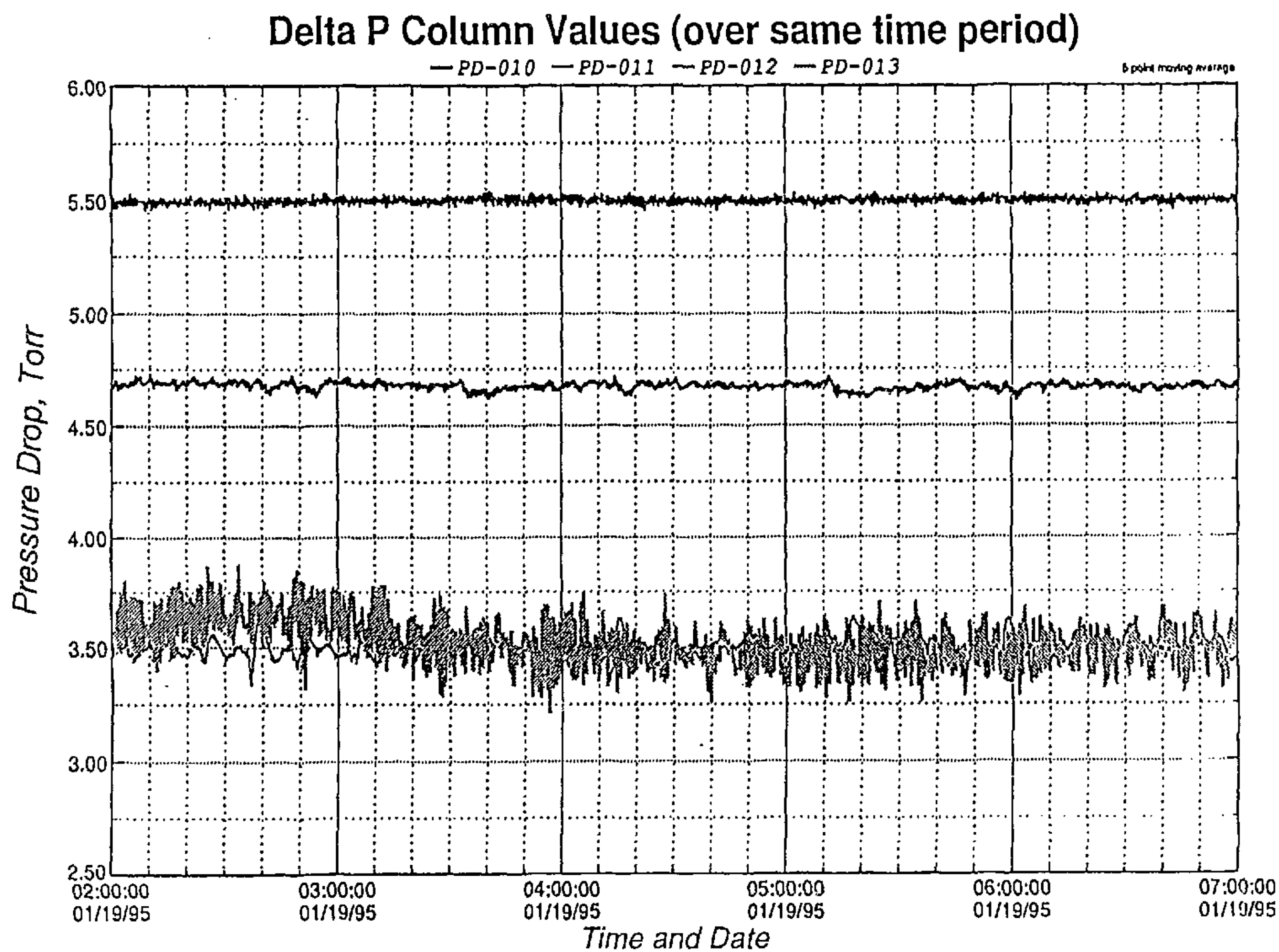


Figure 10. Column pressure drop signals from PPPL commissioning tests showing stable operation of four-column system.