

JAERI - M
88-253

HYDROGEN ISOTOPE SEPARATION STUDY WITH TSTA
CRYOGENIC DISTILLATION SYSTEM
—SINGLE COLUMN EXPERIMENT WITH D-T SYSTEM—

December 1988

Toshihiko YAMANISHI, Hiroshi YOSHIDA, Taisei NAITO*¹

Shingo HIRATA*², R.H. SHERMAN*³, J.R. BARTLIT*³

and J.L. ANDERSON*³

日 本 原 子 力 研 究 所
Japan Atomic Energy Research Institute

JAERI-Mレポートは、日本原子力研究所が不定期に公刊している研究報告書です。
入手の問合わせは、日本原子力研究所技術情報部情報資料課（〒319-11茨城県那珂郡東海村）あて、お申しこしください。なお、このほかに財団法人原子力弘済会資料センター（〒319-11 茨城県那珂郡東海村日本原子力研究所内）で複写による実費頒布をおこなっております。

JAERI-M reports are issued irregularly.

Inquiries about availability of the reports should be addressed to Information Division
Department of Technical Information, Japan Atomic Energy Research Institute, Tokai-mura, Naka-gun, Ibaraki-ken 319-11, Japan.

©Japan Atomic Energy Research Institute, 1988

編集兼発行	日本原子力研究所
印刷	楳高野高速印刷

Hydrogen Isotope Separation Study with TSTA
Cryogenic Distillation System
- Single Column Experiment with D-T System -

Toshihiko YAMANISHI, Hiroshi YOSHIDA, Taisei NAITO^{*1}
Shingo HIRATA^{*2}, R.H. SHERMAN^{*3}, J.R. BARTLIT^{*3}
and J.L. ANDERSON^{*3}

Department of Thermonuclear Fusion Research
Naka Fusion Research Establishment
Japan Atomic Energy Research Institute
Naka-machi, Naka-gun, Ibaraki-ken

(Received November 21, 1988)

An experimental study of cryogenic distillation was performed with deuterium-tritium (60 g of tritium) by using one column in the TSTA isotope separation system. The overall HETP values were 4 ~ 5.7 cm for the total reflux mode and 5 ~ 5.5 cm for the total recycle mode. The values for both operation modes increased slightly with the vapor velocity. No effect of the reflux ratio on the overall HETP was observed. The HETP depended little on the column height. The dynamic column behavior predicted by mathematical simulation modeling was in qualitative agreement with the experimental observation.

Keywords: Cryogenic Distillation, Deuterium-Tritium System, HETP,
Pressure Drop, Liquid Holdup, Total Reflux, Total Recycle,
Vapor Velocity, Reflux Ratio

*1 Mitsubishi Atomic Power Industries, Inc.

*2 Kawasaki Heavy Industries, Ltd.

*3 Los Alamos National Laboratory

T S T A 深冷蒸留システムを用いた水素同位体分離に関する研究
- D - T による単塔実験 -

日本原子力研究所那珂研究所核融合研究部

山西 敏彦・吉田 浩・内藤 大靖^{*1}・平田 慎吾^{*2}

R. H. SHERMAN^{*3}・J. R. BARTLIT^{*3}・J. L. ANDERSON^{*3}

(1988年11月21日受理)

核融合炉燃料サイクル技術開発に関する原研/ロスアラモス国立研究所のT S T A 共同試験の一環として、T S T A 同位体分離システムにおける蒸留塔によるD - T 系深冷蒸留実験(T₂ インベントリ-60g)を行った。総括のH E T P は、全還流操作の場合4~5.7 cm、抜き出し循環操作の場合5~5.5 cmであることが判明した。H E T P は、いずれの操作条件においても塔内蒸気速度に伴って増加した。一方、還流比がH E T P に及ぼす影響並びにH E T P の塔高への依存性は認められなかった。なお、数学的シミュレーションモデルによる蒸留塔の動特性の計算は、実験結果と定性的には一致することがわかった。

那珂研究所：〒311-01 茨城県那珂郡那珂町大字向山801-1

*1 三菱原子力工業㈱

*2 川崎重工業㈱

*3 ロスアラモス国立研究所

Contents

1. Introduction	1
2. Experimental	1
3. Mathematical simulation procedure	2
3.1 Algorithm of the simulation codes	2
3.2 Analysis of experimental data	4
4. Results and discussion	6
4.1 Liquid holdup in packed section	6
4.2 Pressure drop across column	7
4.3 Effects of vapor velocity and reflux ratio on overall HETP	7
4.4 Composition distribution within the column	9
4.5 Dynamic column behavior	10
5. Conclusion	11
Acknowledgement	11
References	12

目 次

1. 緒 論	1
2. 実 験	1
3. 数学的シミュレーション手法	2
3.1 シミュレーションコードの演算法	2
3.2 実験データの解析手法	4
4. 結果と考察	6
4.1 充填部の液ホールドアップ	6
4.2 塔内の圧力損失	7
4.3 塔内蒸気速度と還流比が統括HETPに及ぼす影響	7
4.4 塔内の組成分布	9
4.5 蒸留塔の動的挙動	10
5. 結 論	11
謝 辞	11
文 献	12

1. INTRODUCTION

As part of a research program to establish and verify design and operating methods for the cryogenic distillation system, it was planned in the JAERI/LANL-DOE collaboration on Fusion Fuel Technology that the single and two column experiments would be performed using the TSTA isotope separation system.⁽¹⁾ In accordance with this program, the authors performed H-D distillation experiments on a single column, and reported the data obtained in Refs. (1) and (5).

The hydrogen isotopes differ appreciably in physicochemical properties such as density, viscosity and surface tension. In the case where a large amount of tritium exists in the column, the decay heat may affect column performance. Hence, the information obtained from the H-D distillation experiment should be verified using D-T. Several workers have published experimental data for columns separating D-T⁽²⁾⁽³⁾, however, there are some differences between the results obtained. For these reasons, further experimental studies are required.

The present paper summarizes the results of the single column experiment with the D-T system (60 g of tritium) performed at TSTA. The principal objectives of the present study are 1) to measure fundamental engineering data of the column such as the HETP, pressure drop and liquid holdup and 2) to examine the validity of mathematical simulation procedures by comparing experimental observations with calculational results.

2. EXPERIMENTAL

Uranium tritide beds (UTB-3 and 4)^{*1} were used to supply D-T gas (with a little H) into the column. The D-T mixture charged into the column was circulated with transfer pumps through an

1. INTRODUCTION

As part of a research program to establish and verify design and operating methods for the cryogenic distillation system, it was planned in the JAERI/LANL-DOE collaboration on Fusion Fuel Technology that the single and two column experiments would be performed using the TSTA isotope separation system.⁽¹⁾ In accordance with this program, the authors performed H-D distillation experiments on a single column, and reported the data obtained in Refs. (1) and (5).

The hydrogen isotopes differ appreciably in physicochemical properties such as density, viscosity and surface tension. In the case where a large amount of tritium exists in the column, the decay heat may affect column performance. Hence, the information obtained from the H-D distillation experiment should be verified using D-T. Several workers have published experimental data for columns separating D-T⁽²⁾⁽³⁾, however, there are some differences between the results obtained. For these reasons, further experimental studies are required.

The present paper summarizes the results of the single column experiment with the D-T system (60 g of tritium) performed at TSTA. The principal objectives of the present study are 1) to measure fundamental engineering data of the column such as the HETP, pressure drop and liquid holdup and 2) to examine the validity of mathematical simulation procedures by comparing experimental observations with calculational results.

2. EXPERIMENTAL

Uranium tritide beds (UTB-3 and 4)^{*1} were used to supply D-T gas (with a little H) into the column. The D-T mixture charged into the column was circulated with transfer pumps through an

equilibrator to make its composition the equilibrium state at room temperature. This operation was performed for about 4 hrs before the distillation. Total amount of the gases charged into the column was 20.1 mol, and the atomic percentages of the three isotopes were 0.59%, 52.52% and 46.89%, H, D, and T respectively.

In the four column cascade of the TSTA isotope separation system, the lead column was used in the present study. The experimental apparatus and procedure were already described in Refs. (1) and (5). The experiment was performed in total reflux and total recycle modes as shown in Fig. 1, and the dynamic variation of composition distributions within the column were measured by gas chromatography. In the present experiment, the packed section of the column was slightly refrigerated by the helium gas to remove the decay heat of tritium.

*1 The gas in UTB-3

Amount : 6.8 mol

Atomic percentages : 0.27% of H, 30.53% of D and 68.75% of T

The tritium gas added to the UTB-3 from shipping containers

Amount : 3.3 mol

The gas in UTB-4

Amount : 10 mol

Atomic percentages : 0.97% of H, 82.84% of D and 12.95% of T

3. MATHEMATICAL SIMULATION PROCEDURE

3.1 Algorithm of the simulation codes

Several simulation codes for the cryogenic distillation column have been developed in JAERI. Among these codes, Crydis-₁⁽⁶⁾, ₂⁽⁵⁾ and D⁽⁷⁾ are used to analyze the experimental data. Heat balances and nonideality of hydrogen isotope mixtures are

equilibrator to make its composition the equilibrium state at room temperature. This operation was performed for about 4 hrs before the distillation. Total amount of the gases charged into the column was 20.1 mol, and the atomic percentages of the three isotopes were 0.59%, 52.52% and 46.89%, H, D, and T respectively.

In the four column cascade of the TSTA isotope separation system, the lead column was used in the present study. The experimental apparatus and procedure were already described in Refs. (1) and (5). The experiment was performed in total reflux and total recycle modes as shown in Fig. 1, and the dynamic variation of composition distributions within the column were measured by gas chromatography. In the present experiment, the packed section of the column was slightly refrigerated by the helium gas to remove the decay heat of tritium.

*1 The gas in UTB-3

Amount : 6.8 mol

Atomic percentages : 0.27% of H, 30.53% of D and 68.75% of T

The tritium gas added to the UTB-3 from shipping containers

Amount : 3.3 mol

The gas in UTB-4

Amount : 10 mol

Atomic percentages : 0.97% of H, 82.84% of D and 12.95% of T

3. MATHEMATICAL SIMULATION PROCEDURE

3.1 Algorithm of the simulation codes

Several simulation codes for the cryogenic distillation column have been developed in JAERI. Among these codes, Crydis-1⁽⁶⁾, 2⁽⁵⁾ and D⁽⁷⁾ are used to analyze the experimental data. Heat balances and nonideality of hydrogen isotope mixtures are

not incorporated into the codes. The above simple models used are sufficient to determine the overall HETP values.

3.1.1 Crydis-1

This code simulates the composition distribution of the column at steady state in the total recycle mode. The successive substitution approach of the tridiagonal matrix method is used for achieving convergence of temperatures and overall material balances. Input data are the flow rates of the vapor and liquid streams within the column, compositions and flow rates of the feeds, and column pressure. The calculation is performed by assuming the number of total theoretical stages and the feed stage number.

3.1.2 Crydis-2

This code simulates the composition distribution at steady state in the total reflux or total recycle modes. If the concentrations of components at either the top or bottom stream are given, the composition distribution can be calculated using the vapor liquid relations in a similar manner to the Thiele-Geddes method⁽⁸⁾.

3.1.3 Crydis-D

This code simulates the dynamic behavior of the column. The integration algorithm used is the one developed by Ballard and Brosilow⁽⁹⁾. Input data are the compositions and flow rates of the feed, flow rates of the vapor and liquid streams, liquid holdups, column pressure, and initial composition distribution within the column. The calculation is performed by assuming the total number of theoretical stages and the feed stage numbers.

3.2 Analysis of experimental data

3.2.1 Determination procedure of the HETP values

The overall HETP values are determined from the packed height of the column and the number of total theoretical stages which gives the best fit to experimental observations at steady state. Variation of the HETP with the column height can also be discussed by comparing the experimental observation with calculated results for the composition distribution within the column.

(i) Total reflux mode

The steady state simulation can be performed by the Crydis-2. In the case where the composition at the top or bottom of the column could not be measured in the experiment, the overall HETP value was determined from the calculated and experimental results for the middle part of the column.

(ii) Total recycle mode

In the experiment, only dominant molecular species were detected in the top and bottom streams of the column. The steady state simulation in the total recycle mode can be performed by Crydis-1 as a function of the total number of theoretical stages and the feed stage number, however, the calculated results for the dominant components at the top and bottom streams vary little with the above parameters. Hence, the number of total theoretical stages and the feed stage number of the actual column are determined by using the Crydis-1 and 2 in the following manner.

- (1) Assume the total number of theoretical stages and the feed stage number.

- (2) Calculate the composition distribution of the column by Crydis-1.
- (3) Recalculate the composition distribution by Crydis-2. For trace components, the calculated values by Crydis-1 are used.
- (4) Determine the total number of theoretical stages and the feed stage number of the actual column by comparing the calculated result of Crydis-2 with experimental observation.
- (5) Return to step (1), and repeat steps (2)~(4) until the following equation is satisfied :
The total number of theoretical stages and the feed stage number determined at step (4) = those of step (1).

3.2.2 Evaluation of dynamic column behavior

For step changes of the operating parameters such as the vapor velocity and the reflux ratio, it was observed that the liquid holdup in the reboiler varied rapidly and reached a steady state value within a few minutes. The dynamic behavior of the column is predicted by Crydis-D, however, the variation of the liquid holdups can not be incorporated into the simulation code. Therefore, the authors consider the variation of the liquid holdup, using the following assumptions :

- (1) The dynamic variation of the liquid holdup is very rapid in comparison with that of the composition distribution.
- (2) The liquid holdup on every theoretical stage in the packed section decreases or increases uniformly. The liquid holdup in the reboiler varies by the decrement or increment of that in the whole packed section.

4. RESULTS AND DISCUSSION

4.1 Liquid holdup in packed section

It is expected that the liquid holdup of the condenser is negligible in comparison with that in the packed section. Hence, the liquid holdup in the packed section was evaluated by subtracting the measured value for the reboiler and the calculated vapor holdup within the whole column from the total amount of gas charged into the column.

Figure 2 shows the variation of liquid holdup in the packed section with the vapor velocity. The values are from 11 to 16 mol, and increase with the vapor velocity. The dimensions of the packed section of the column used were approximately 2600 cm^3 ($2.84 \text{ cm} \phi \times 412 \text{ cm}$).⁽¹⁾⁽⁴⁾ The liquid holdup in the packed section corresponds to 10~15% of the superficial volume. Sherman et al⁽⁴⁾ measured the liquid holdup in the packed section for the column used in the present study, using pure H_2 or D_2 under the conditions of total reflux. Their experimental data for pure D_2 shown in Fig. 2 are slightly smaller than those of the present study. The above-mentioned difference in the liquid holdup may be related to the experimental procedure used (whether in reflux mode or recycle mode).

Figure 3 shows the variation of the liquid level in the reboiler during the experiment. The measured values fluctuate appreciably when the vapor velocity is large ($\sim 13 \text{ cm/sec}$). The liquid holdup in the packed section may be subject to fluctuation owing to the large vapor velocity. Another result observed from Fig. 3 is that the liquid holdup in the reboiler decreases continuously during each run. The output of the reboiler heater increases slightly during each run as shown in Fig. 4, so that the liquid holdup in the packed section should gradually

increase.

4.2 Pressure drop across column

Figure 5 shows the variation of the pressure drop across the column during the experiment. The liquid holdup in the packed section is one of the major parameters which affect the pressure drop across the column. The pressure drop shown in Fig. 5 fluctuates when the vapor velocity is high (~ 13 cm/sec), and increases continuously during each run. These experimental results support the discussions made for the liquid holdup in the packed section.

Figure 6 shows the relation between the pressure drops and vapor velocity. The values are proportional to the vapor velocity to the power 1.5~1.7, and neither the flooding point nor the loading point is observed. It can be concluded that the present experiments were performed in a velocity range adequately far from flooding. The pressure drop of the column used in the present study previously reported by sherman et al.⁽⁴⁾ are also presented in Fig. 6. The above experimental data were measured with pure D_2 under the condition of total reflux, and they are in good agreement with those of the present study. It appears that deuterium-tritium mixtures behave similarly to pure D_2 .

4.3 Effects of vapor velocity and reflux ratio on overall HETP

The overall HETP values were measured as functions of the vapor velocity and reflux ratio. Figure 7 shows the effect of vapor velocity on the overall HETP values. The values for the total reflux mode are from 4 to 5.7 cm, and those for total recycle are 5~5.5 cm. Except the case where the vapor velocity is large (~ 13 cm/sec), the overall HETP values for the total recycle mode are larger than those of the total reflux mode.

The flow disturbance in the column caused by the recycle operation may result in the decrease of vapor/liquid interfacial area.

Another significant result observed from Fig. 7 is that the overall HETP values increase slightly with the vapor velocity for both the operation modes. The overall HETP value can be expressed by⁽¹⁰⁾

$$\text{overall HETP} = z/(N-2), \quad - (1)$$

$$(y_{i,1} - y_{i,N}) = n_i/V_j dz, \quad - (2)$$

where n_i = mass transfer rate for component i ($\text{molm}^2/\text{m}^3\text{hr}$)

N = number of total theoretical stages of the column (-)

V_j = flow rate of vapor stream leaving j -th stage (mol/sec)

$y_{i,j}$ = mole fraction of i -th component in the liquid stream leaving the j -th stage (-)

z = packed height of the column (m).

The mass transfer rate n_i can be regarded as a function of the vapor velocity and the column height. If n_i increases proportionally to the vapor velocity, the overall HETP value should have no dependence on the vapor velocity. The present experimental results show that the mass transfer rate for the hydrogen isotope distillation system is proportional to less than the first power of the vapor velocity.

The effect of the reflux ratio on the overall HETP is shown in Fig. 8 under conditions that the vapor velocity and the flow rate of the bottom stream are almost constant. The overall HETP values are not affected by the reflux ratio. The reflux ratio and the vapor velocity are suitable manipulated parameters of the distillation column to control purities of top and bottom products at desirable values.⁽¹¹⁾ The stage model used in the

present study has the disadvantage that it is difficult to consider the variation of the number of total theoretical stages with operating conditions when dynamic behavior of the columns is simulated. However, the constancy of the overall HETP against the reflux ratio and its slight dependency on the vapor velocity indicate that the simulation results by the stage model can well be extended to actual column behavior even for the case where the above operating parameters are varied to control products purities.

4.4 Composition distribution within the column

Figures 9 and 10 show the composition distribution at steady state for representative runs. The calculated lines are also drawn in the figure under the assumption that HETP is constant with column height. Table 1 shows the experimental and calculational conditions of the runs. The general trends observed from the figures are as follows :

- (1) At the middle part of the column, the calculated results differ from the experimental observations in the concentrations of DT and T_2 .
- (2) The calculated results are in rough agreement with the experimental observations at the upper and lower parts of the column.

Figure 2 shows the steady state composition distribution obtained from the H-D experiments. The calculated result is in agreement with the experimental observation within the whole column. Table 2 shows mole fractions and their equilibrium compositions at room temperature for the experimental observations and calculated results of Run B. The experimental observations at the middle part of the column are roughly equal to the equilibrium compositions at room temperature. The above

phenomena might show that the isotope equilibrium reaction proceeds in sampling lines owing to the existence of tritium, especially at the middle part of the column. Since the volumes of sampling lines at the middle part of the column are larger than those of other parts, a longer time was required to purge memory of previous sample gases. The longer residence time in the sampling lines may give more time for the equilibration reaction.

Except for concentrations of DT and T_2 at the middle part of the column, the composition distribution calculated is in rough agreement with experimental observation. It appears that the HETP depends little on the column height. This experimental result is important for column design : positions of feed and side-cut streams of actual columns can be well estimated by the stage model.

4.5 Dynamic column behavior

The dynamic behavior of the column after a step change of the reflux ratio was observed in the total recycle mode. The pressure of the column alone was controlled by the temperature of the helium refrigerant gas. Figure 12 shows the dynamic variation of D_2 and DT concentrations in the top stream of the column. Calculated results with the Crydis-D are also presented in the figure. Two calculated lines are not in quantitative agreement with the experimental observations, however, they simulate the dynamic variation of components qualitatively. Since the lead column in the TSTA isotope separation system was used in the experiment, there are three columns acting as dead volume in the experimental apparatus (see Fig. 13). Considering this configuration of the experimental apparatus, the present

simulation procedure appears to be roughly valid for prediction of the dynamic column behavior.

5. CONCLUSION

The present experimental study was performed by using the actual-size column in the TSTA isotope separation system. The inventory of tritium within the column was about 60 g. Some significant conclusions are recapitulated here.

- (1) The liquid holdup in the packed section was approximately 10~15% of its superficial volume, and increased with vapor velocity.
- (2) In the range of vapor velocity from 5 to 13 cm/sec, there was no evidence that loading or flooding occurred.
- (3) The overall HETP values measured were from 4 to 5.7 cm for the total reflux mode and from 5 to 5.5 cm for the total recycle mode. The overall HETP increased slightly with vapor velocity and showed no dependence on the reflux ratio. The HETP appeared to be relatively constant within the column.
- (4) The dynamic column behavior predicted by the mathematical simulation procedure was in qualitative agreement with the experimental observation.
- (5) The stage model is well suited for the simulation of actual column behavior and performance.

ACKNOWLEDGEMENT

The present study was supported by the US Department of Energy, office of Fusion Energy, and the Japan Atomic Energy Research Institute.

simulation procedure appears to be roughly valid for prediction of the dynamic column behavior.

5. CONCLUSION

The present experimental study was performed by using the actual-size column in the TSTA isotope separation system. The inventory of tritium within the column was about 60 g. Some significant conclusions are recapitulated here.

- (1) The liquid holdup in the packed section was approximately 10~15% of its superficial volume, and increased with vapor velocity.
- (2) In the range of vapor velocity from 5 to 13 cm/sec, there was no evidence that loading or flooding occurred.
- (3) The overall HETP values measured were from 4 to 5.7 cm for the total reflux mode and from 5 to 5.5 cm for the total recycle mode. The overall HETP increased slightly with vapor velocity and showed no dependence on the reflux ratio. The HETP appeared to be relatively constant within the column.
- (4) The dynamic column behavior predicted by the mathematical simulation procedure was in qualitative agreement with the experimental observation.
- (5) The stage model is well suited for the simulation of actual column behavior and performance.

ACKNOWLEDGEMENT

The present study was supported by the US Department of Energy, office of Fusion Energy, and the Japan Atomic Energy Research Institute.

simulation procedure appears to be roughly valid for prediction of the dynamic column behavior.

5. CONCLUSION

The present experimental study was performed by using the actual-size column in the TSTA isotope separation system. The inventory of tritium within the column was about 60 g. Some significant conclusions are recapitulated here.

- (1) The liquid holdup in the packed section was approximately 10~15% of its superficial volume, and increased with vapor velocity.
- (2) In the range of vapor velocity from 5 to 13 cm/sec, there was no evidence that loading or flooding occurred.
- (3) The overall HETP values measured were from 4 to 5.7 cm for the total reflux mode and from 5 to 5.5 cm for the total recycle mode. The overall HETP increased slightly with vapor velocity and showed no dependence on the reflux ratio. The HETP appeared to be relatively constant within the column.
- (4) The dynamic column behavior predicted by the mathematical simulation procedure was in qualitative agreement with the experimental observation.
- (5) The stage model is well suited for the simulation of actual column behavior and performance.

ACKNOWLEDGEMENT

The present study was supported by the US Department of Energy, office of Fusion Energy, and the Japan Atomic Energy Research Institute.

REFERENCES

- (1) Yamanishi, T. et al. : JAERI-memo, Japan Atomic Energy Research Institute, Private Communication (1988).
- (2) Bartlit, J. R., Denton, W. H., Sherman, R. H.: Proc. 3rd Topical Mtg. on Technol. of Controlled Nuclear Fusion, Santa Fe, NM, May 9-11, 1978.
- (3) Wilkes, W. R.: MLM-2502, p. 10 (1978).
- (4) Sherman, R. H., Bartlit, J. R., Veirs, D. K.: Fusion Technol., 6, 625 (1984).
- (5) Yamanishi, T., Yoshida, H., Hirata, S., Naito T., Naruse Y., Sherman R. H., Gruetzmacher K. M., Bartlit, J. R., Anderson J. L.: Proc. 3rd Topical Mtg. on Tritium Technol. in Fission, Fusion and Isotopic Applications, Toronto, Canada May 1-6, 1988 (in press).
- (6) Kinoshita, M. and Naruse, Y.: JAERI-M 9871 (1981).
- (7) Kinoshita, M.: Fusion Technol., 9, 492 (1986).
- (8) Thiele, E. W. and Geddes, R. L.: Ind. Eng. Chem., 25, 289 (1933).
- (9) Ballard, D. M., and Brosilow, C. B.: Proc. 71st Annual Mtg. American Institute of Chemical Engineers, Miami, Florida, November 12-16, 1978, No. 42a.
- (10) Yamanishi, T. and Kinoshita, M.: J. Nucl. Sci. Technol., 21, 853 (1984).
- (11) Kinoshita, M., Bartlit, J. R., Sherman, R. H.: Nucl. Technol./Fusion, 5, 30 (1984).

Table 1 Experimental and calculational conditions

	Run A	Run B	Run C
Operation mode	total recycle	Total recycle	Total reflux
System	D-T	D-T	H-D
Pressure (Torr)	709	770	680
Flow rate (mol/sec)			
top stream	--	1.49×10^{-3}	--
bottom stream	--	1.68×10^{-3}	--
Vapor velocity (cm/sec)	13.3	9.9	9.2
Liquid holdup of reboiler (mol)	2.58	2.43	0.88
Number of total theoretical stages	77 ^{*1}	76	83 ^{*2}
Feed stage number	--	53	--

*1 The total number of theoretical stages was determined except the portion from the top of the column to 84.3 cm.

*2 The number of total theoretical stages was determined except the portion from the bottom of the column to 38.1 cm.

Table 2 Comparison between experimental observation and calculated results for Run B

Sampling point	Experimental observation (%)	Equilibrium composition at room temperature of experimental observation (%)	Calculated results (%)
Top stream	H ₂ : 0.0 HD : 4.47 HT : 2.05 D ₂ : 62.65 DT : 30.73 T ₂ : 0.0	H ₂ : 0.134 HD : 5.29 HT : 0.972 D ₂ : 64.58 DT : 26.21 T ₂ : 2.81	H ₂ : 0.0 HD : 4.47 HT : 2.05 D ₂ : 62.65 DT : 30.73 T ₂ : 0.0
322.7 cm from bottom	H ₂ : 0.0 HD : 0.44 HT : 0.32	H ₂ : 0.0 HD : 0.476 HT : 0.280	H ₂ : 0.0 HD : 1.01 HT : 0.61
Upper part of packed section	D ₂ : 25.34 DT : 68.71 T ₂ : 5.18	D ₂ : 36.21 DT : 46.94 T ₂ : 16.09	D ₂ : 31.75 DT : 67.18 T ₂ : 0.0
226.1 cm from bottom	H ₂ : 0.0 HD : 2.39 HT : 1.64	H ₂ : 0.0 HD : 2.48 HT : 1.44	H ₂ : 0.0 HD : 1.00 HT : 0.60
middle part of packed section	D ₂ : 33.55 DT : 49.15 T ₂ : 13.27	D ₂ : 35.43 DT : 45.29 T ₂ : 15.30	D ₂ : 30.29 DT : 67.74 T ₂ : 0.35
99.1 cm from bottom	H ₂ : 0.0 HD : 0.0 HT : 0.0	H ₂ : 0.0 HD : 0.0 HT : 0.0	H ₂ : 0.0 HD : 0.62 HT : 0.44
middle part of packed section	D ₂ : 16.70 DT : 58.58 T ₂ : 24.72	D ₂ : 21.49 DT : 48.99 T ₂ : 29.51	D ₂ : 26.50 DT : 66.38 T ₂ : 6.05
38.1 cm from bottom	H ₂ : 0.0 HD : 0.0 HT : 0.0	H ₂ : 0.0 HD : 0.0 HT : 0.0	H ₂ : 0.0 HD : 0.0 HT : 0.0
lower part of packed section	D ₂ : 5.26 DT : 79.06 T ₂ : 14.68	D ₂ : 20.40 DT : 48.78 T ₂ : 30.82	D ₂ : 2.62 DT : 80.37 T ₂ : 16.93
Bottom stream	H ₂ : 0.0 HD : 0.0 HT : 0.0 D ₂ : 3.18 DT : 62.69 T ₂ : 34.14	H ₂ : 0.0 HD : 0.0 HT : 0.0 D ₂ : 12.20 DT : 44.64 T ₂ : 43.16	H ₂ : 0.0 HD : 0.0 HT : 0.0 D ₂ : 0.79 DT : 61.50 T ₂ : 37.66

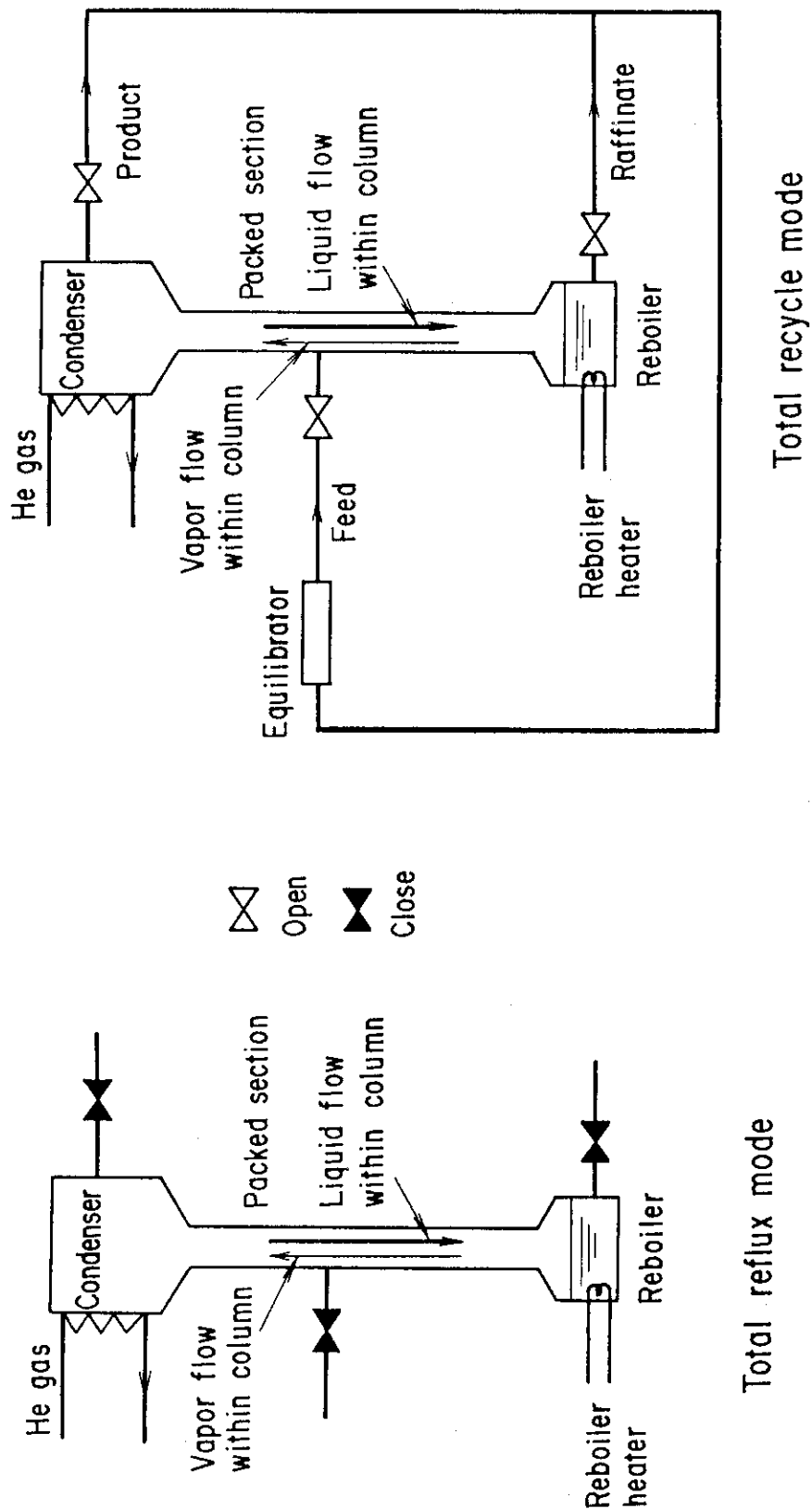


Fig. 1 Operation modes used in present study

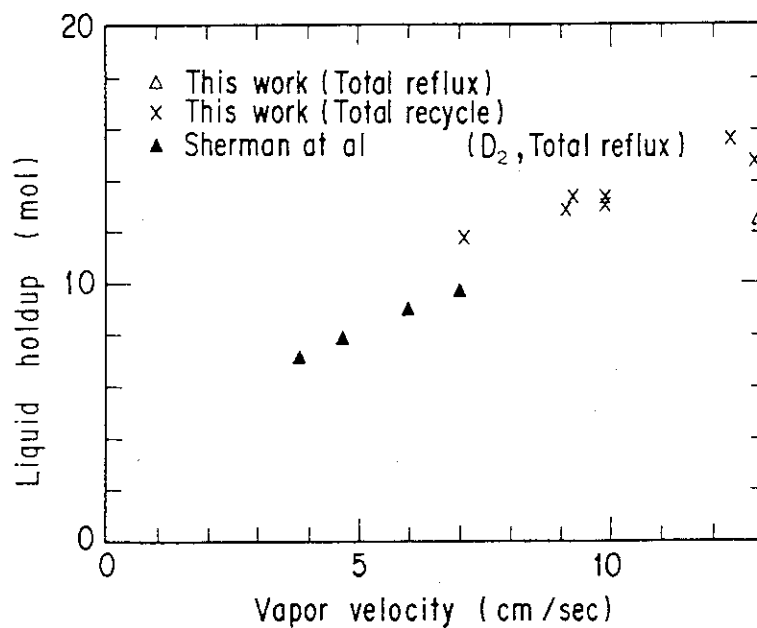


Fig. 2 Variation of liquid holdup in packed section with vapor velocity

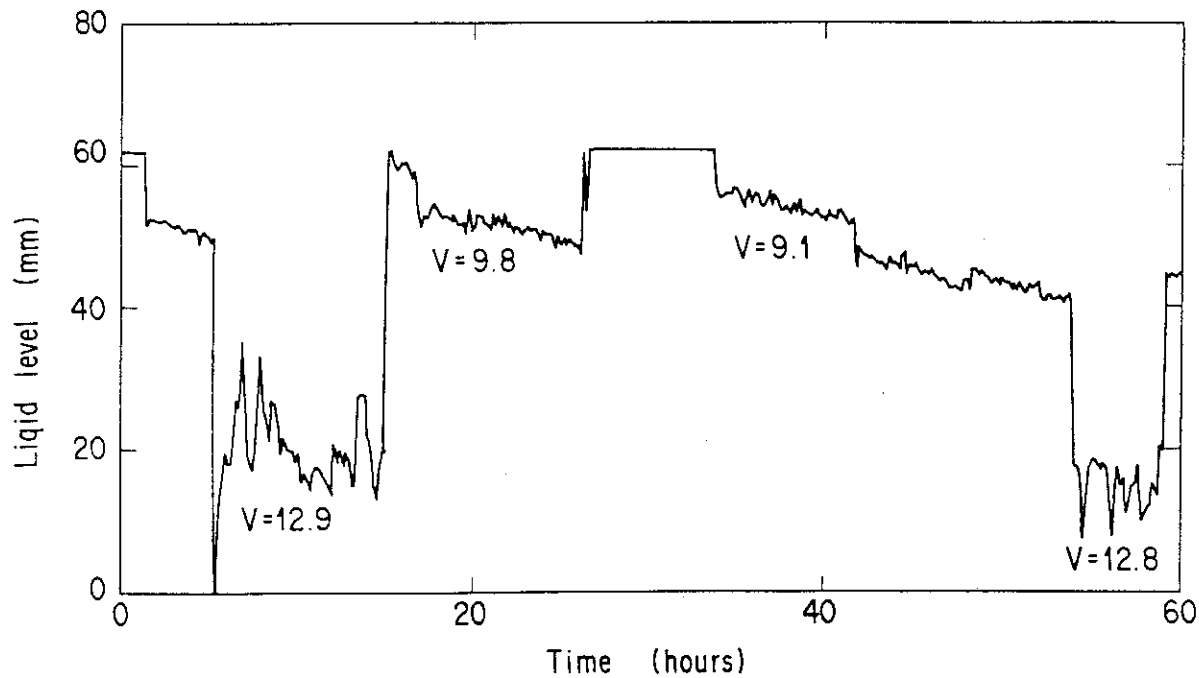


Fig. 3 Dynamic variation of liquid level in reboiler during experiments V = Vapor velocity (cm/sec)

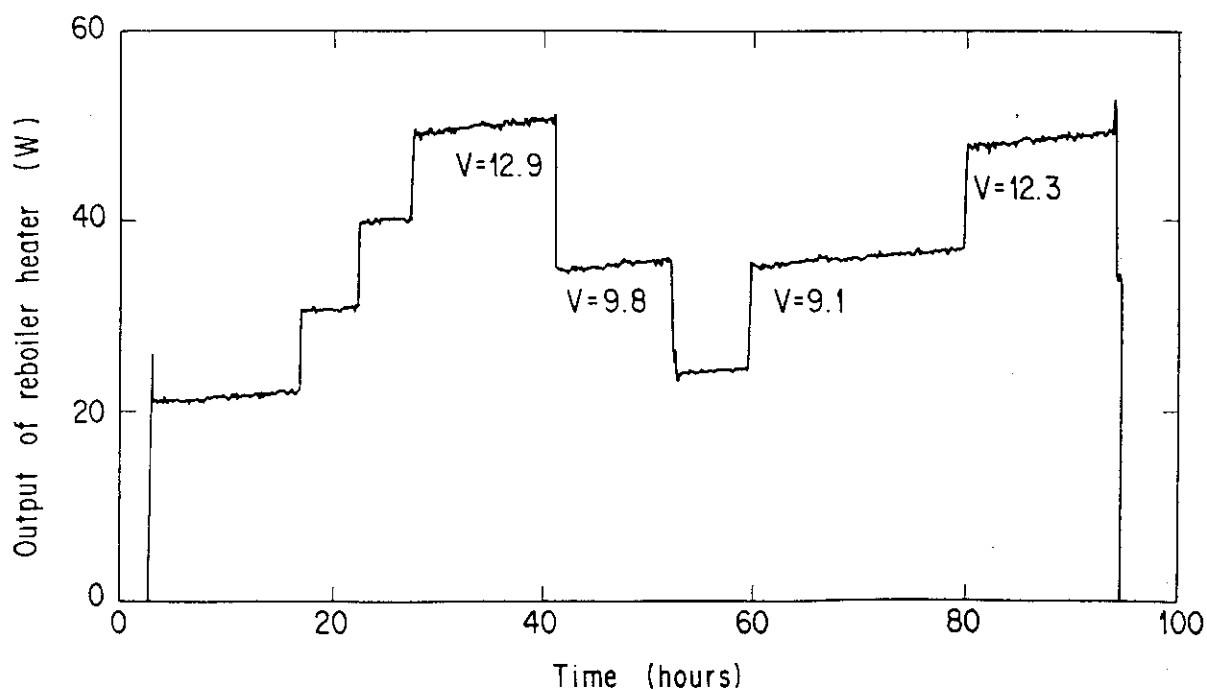


Fig. 4 Dynamic variation of output of reboiler heater during experiments V = Vapor velocity (cm/sec)

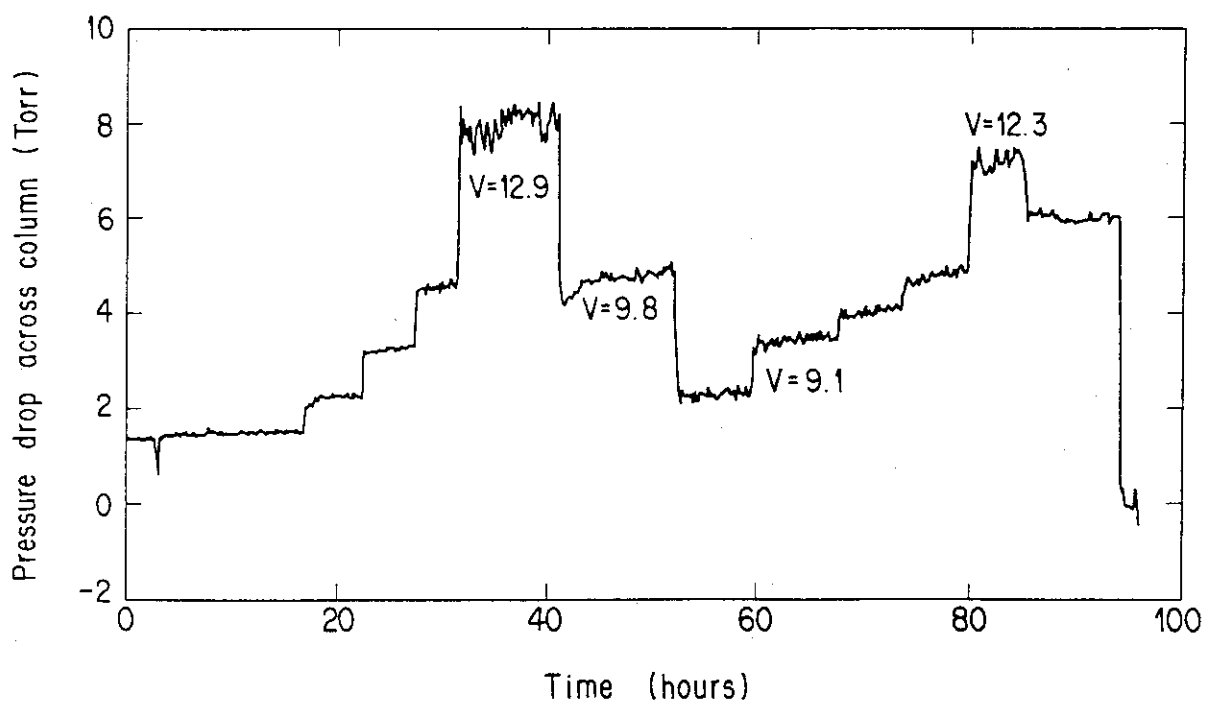


Fig. 5 Dynamic Variation of pressure drop of column during experiments V = Vapor velocity (cm/sec)

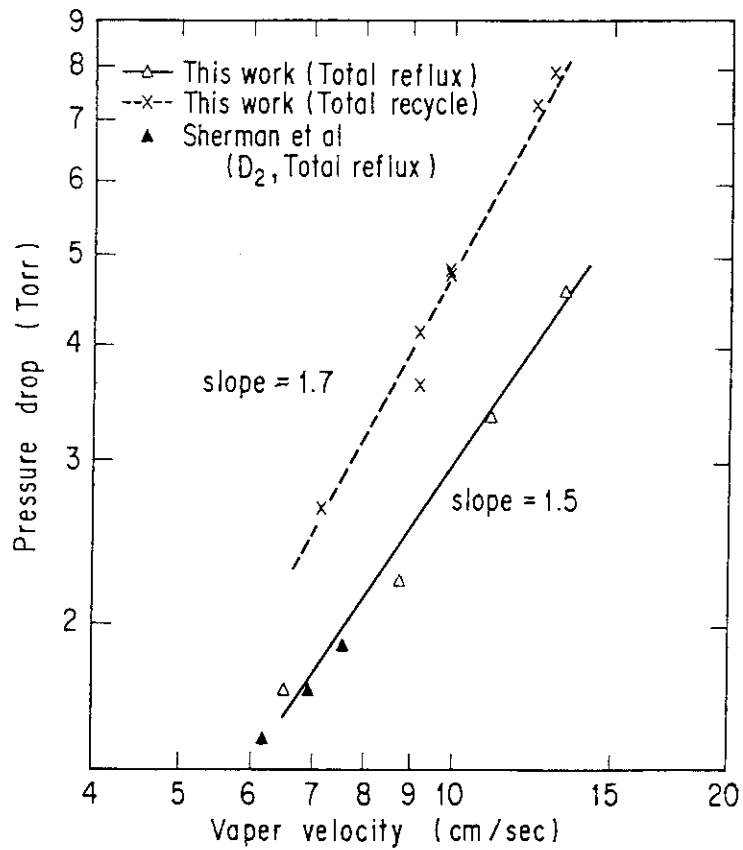


Fig. 6 Variation of pressure drop across column with vapor velocity

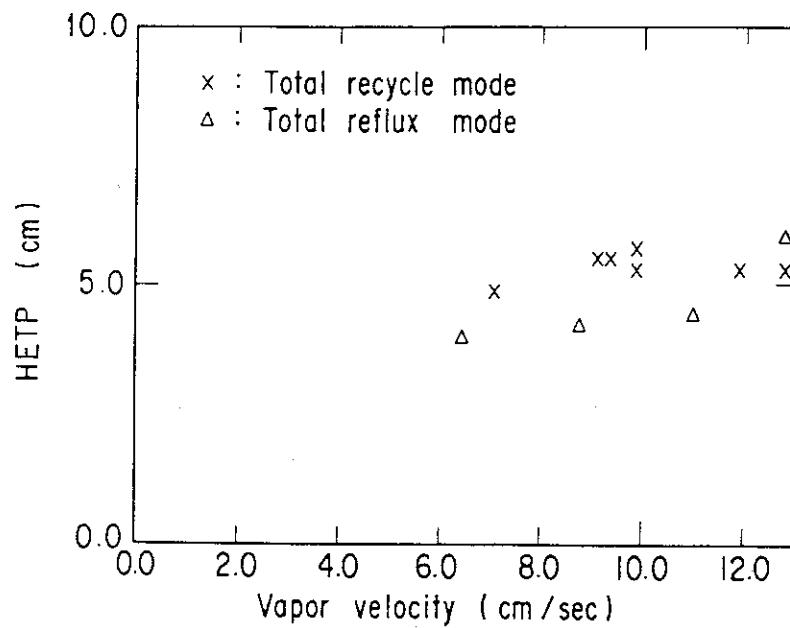


Fig. 7 Effect of vapor velocity on overall HETP

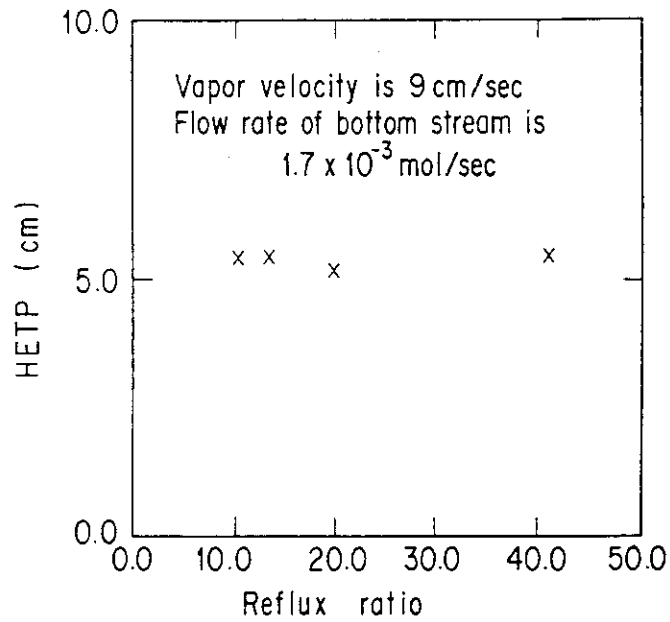


Fig. 8 Effect of reflux ratio on overall HETP

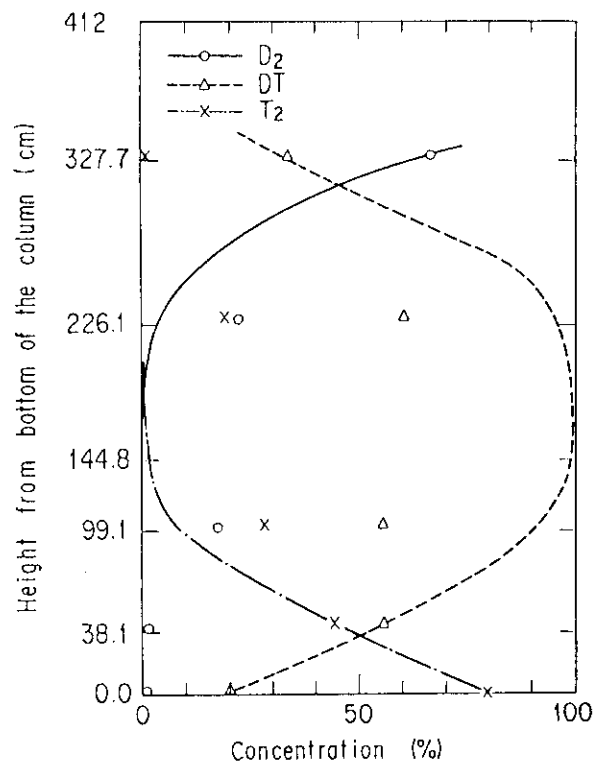


Fig. 9 Composition distribution within column for Run A

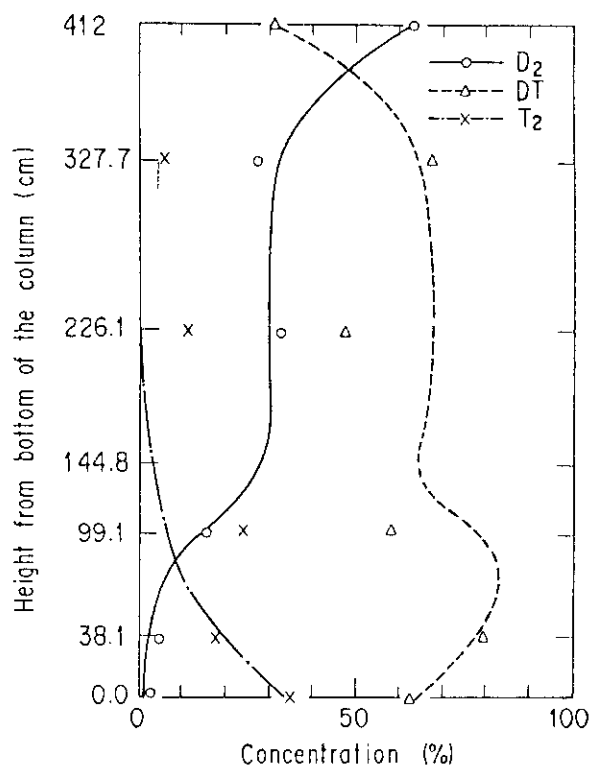


Fig. 10 Composition distribution within column for Run B

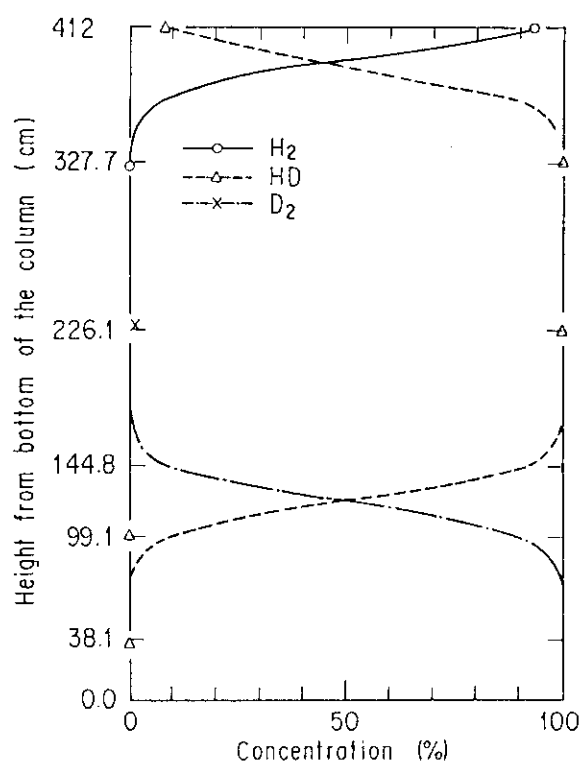


Fig. 11 Composition distribution within column for Run C

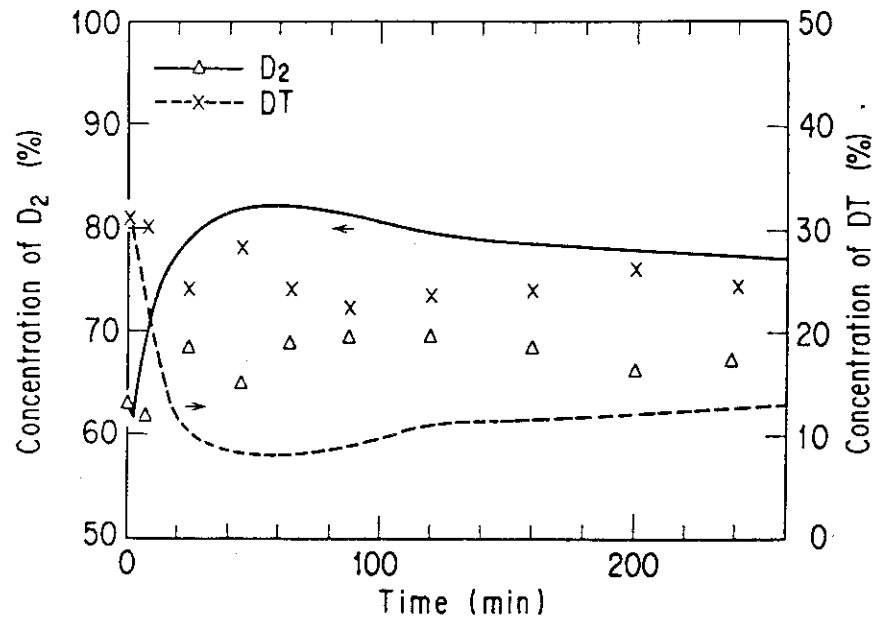


Fig. 12 Dynamic variation of compositions of top stream

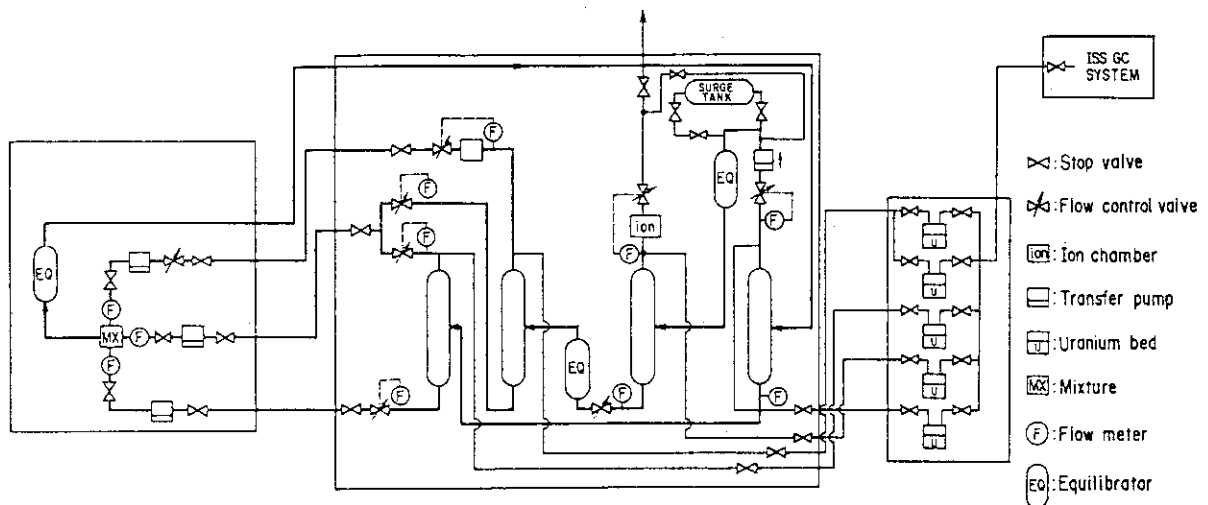


Fig. 13 Flow diagram of experimental apparatus