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HYDROGEN ISOTOPE SEPARATION STUDY WITH THE  
TSTA CRYOGENIC DISTILLATION SYSTEM

— TWO-COLUMN EXPERIMENT WITH H-D-T —

December 1988

Toshihiko YAMANISHI, Hiroshi YOSHIDA, Hiroshi FUKUI\*<sup>1</sup>  
Taisei NAITO\*<sup>2</sup>, Shingo HIRATA\*<sup>3</sup>, R. H. SHERMAN\*<sup>4</sup>  
K. M. GRUETZMACHER\*<sup>4</sup>, J. R. BARTLIT\*<sup>4</sup>  
and J. L. ANDERSON\*<sup>4</sup>

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Hydrogen Isotope Separation Study with the TSTA  
Cryogenic Distillation System  
- Two-Column Experiment with H-D-T -

Toshihiko YAMANISHI, Hiroshi YOSHIDA, Hiroshi FUKUI<sup>\*1</sup>  
Taisei NAITO<sup>\*2</sup>, Shingo HIRATA<sup>\*3</sup>, R. H. SHERMAN<sup>\*4</sup>  
K. M. GRUETZMACHER<sup>\*4</sup>, J. R. BARTLIT<sup>\*4</sup> and J. L. ANDERSON<sup>\*4</sup>

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

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Cryogenic distillation cascade experiments were performed using two columns in the TSTA isotope separation system with H-D-T. Both columns, differing in packed height and inner diameter, confirmed the overall HETP values were approximately 5 cm and relatively constant within the column. The dynamic behavior of the cascade was also discussed, and a basic control method was proposed.

Keywords : Cryogenic Distillation, Two-Column Cascade, Blanket Tritium Processing, Overall HETP, Packed Height, Column Diameter, Dynamic Behavior, Control Method

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\*1 Mitsubishi Heavy Industry, Ltd.

\*2 Mitsubishi Atomic Power Industries, Inc.

\*3 Kawasaki Heavy Industries, Ltd.

\*4 Los Alamos National Laboratory

TSTA 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  
— H-D-T系による2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・福井裕<sup>\*1</sup>・内藤大靖<sup>\*2</sup>・平田慎吾<sup>\*3</sup>

R. H. SHERMAN<sup>\*4</sup>・K. M. GRUETZMACHER<sup>\*4</sup>

J. R. BARTLIT<sup>\*4</sup>・J. L. ANDERSON<sup>\*4</sup>

(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共同試験の一環として、TSTA同位体分離システムにおける2本の塔を用いたH-D-T系深冷蒸留カスケード実験を行った。この実験により、充填部の内径及び高さがおのおの2.8 cm $\phi$ 、412 cmH及び2.5 cm $\phi$ 、320 cmHと異なる両塔共に、総括のHETPが約5 cmであること、更にHETPの値は塔の高さ方向においてほぼ均一であることが判明した。本研究では、カスケードの動的特性についても考察しあわせてその基本的な制御手法を提示した。

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那珂研究所：〒311-01 茨城県那珂郡那珂町大字向山801-1

- \*1 三菱重工業㈱
- \*2 三菱原子力工業㈱
- \*3 川崎重工業㈱
- \*4 ロスアラモス国立研究所

## Contents

1. Introduction .....	1
2. Experimental .....	2
3. Results and discussion .....	4
3.1 Pressure drops across the columns .....	4
3.2 Separation performance of the columns .....	5
3.3 Control method of the cascade .....	7
4. Conclusion .....	9
Acknowledgement .....	9
References .....	10
Appendix Experimental results of all runs .....	18

## 目 次

1. 緒 論 .....	1
2. 実 験 .....	2
3. 結果と考察 .....	4
3.1 塔内の圧力損失 .....	4
3.2 塔の分離性能 .....	5
3.3 カスケードの制御方式 .....	7
4. 結 論 .....	9
謝 辞 .....	9
文 献 .....	10
付録 全実験データ .....	18

## 1. INTRODUCTION

As part of the JAERI/LANL-DOE collaboration on Fusion Fuel Technology, the authors performed H-D and D-T distillation experiments on a single column of the TSTA isotope separation system<sup>(1)</sup>, and reported the data obtained in Refs. (2), (3) and (4).

Since the above studies produced useful engineering data for a single column, the next subject to be studied is the separation characteristics of column cascades. To date, several cryogenic distillation column cascades have been proposed for the main stream fuel circulation<sup>(1)(5)</sup> and the blanket tritium processing system<sup>(6)</sup> in fusion reactors. In addition, cryogenic distillation column cascades are being studied and tested as attractive methods for recovering tritium from heavy water<sup>(7)</sup> and from glove-box atmospheres<sup>(8)</sup>. However, there are few experimental studies reporting on the separation characteristics of the column cascades. The authors<sup>(9)</sup> have reported experimental data for the column cascade of the TSTA isotope separation system. The above study dealt briefly with static and dynamic behaviors of the cascade composed of four interlinked columns. Further experimental studies are desirable for cascades which have different configurations.

The present paper summarizes the experimental results of a two-column cascade using H-D-T (26g of tritium). The principal objectives of the present study are 1) to measure separation characteristics of the cascade and 2) to examine control schemes. The cascade selected simulates a basic

configuration of those proposed by the authors (5) for the blanket tritium processing system, and gives preliminary experimental data for the above system. In addition, the information obtained in the present study is useful for other cascades, since the two-column cascade can be regarded as a basic component of those composed of several columns.

## 2. EXPERIMENTAL

The configuration of the two-column cascade is shown in Fig. 1. The bottom stream of the lead column is fed to the second column. The output streams of the cascade are recycled as the feed. Columns I and T of the TSTA isotope separation system<sup>(9)</sup> were used in the present study as the lead and the second columns, respectively. Specifications of both the columns are presented in Table 1.

The experimental procedure is summarized as follows. After hydrogen isotope gases were charged into the cascade from uranium beds, the mixture within the cascade was circulated through the equilibrators to make its composition the equilibrium state at room temperature before the distillation. The columns were individually operated in the total reflux mode until steady state was achieved. The columns were then put into cascade operation.

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The separation characteristics of the cascade were measured by varying the vapor velocities and the reflux ratios of the columns (flow rates of output streams and outputs of reboiler heaters). To reach the steady state, approximately 2 h was needed for the total reflux mode, and 4 h for the cascade operation.

The total amount of gases charged into the cascade was about 35 mol, and the atomic percentages of the three isotopes were 11.5%, 76.0% and 12.5%, H, D and T, respectively. As described in the preceding section, one of the objectives of the present study is to obtain preliminary experimental data for the cascades proposed by the authors for the blanket tritium processing system. In the blanket tritium processing system, either  $H_2$  or  $D_2$  is added to the helium purge gas to decrease the tritium inventory in the blanket and to reduce the production of tritium oxide. The above-mentioned percentages of three isotopes are roughly equal to the calculated values for the holdup in the cascade proposed for the case where the tritium is diluted with  $D_2$  by two orders of magnitude<sup>(5)</sup>.

### 3. RESULTS AND DISCUSSION

#### 3.1 Pressure Drops across the Columns

Figure 2 shows the relation between the pressure drops across the columns and the vapor velocity. The measured values are roughly proportional to the vapor velocity for both columns, and neither flooding nor loading is observed. Although the packed height of Column T is smaller than that of Column I (see Table 1), the pressure drops for Column T are larger than those of Column I. The data are too limited to establish the reason for the difference between the columns. Two possible explanations are suggested. The dominant molecular species within Column I (the lead column of the cascade) are  $H_2$ , HD, HT,  $D_2$  and DT, and those of Column T (the second column of the cascade) are  $D_2$ , DT and  $T_2$  (see Appendix). Hence, the physicochemical properties of the fluid (e.g. density and viscosity) may affect the pressure drop<sup>(3)</sup>. Another explanation is the effect of the inner diameter. Sherman et al.<sup>(10)</sup> have reported the pressure drops of Columns I and T. The measured value of Column T with  $D_2$  in the total reflux mode was slightly larger than that for Column I. The difference in inner diameter between the columns is small (see Table 1). However, the experimental results obtained by Sherman et al. and the authors may suggest that the inner diameter of the packed section is one of the factors affecting the pressure drop.

In Fig. 2, the pressure drops for Column I obtained in our previous work<sup>(3)</sup>, which were measured with H-D in single column

operation, are also shown for comparison. The present experimental data are slightly smaller than those of our previous work, however, no significant difference is observed. The pressure drop obtained in the single column operation can be used to deal with the cascade operation.

### 3.2 Separation Performance of the Columns

Simulation models used for analysis of experimental data (i.e. determination of the overall HETP values and calculation of composition distributions within the columns) does not incorporate the heat balances and the nonideality of the isotope solution. The simple models used are sufficient for the above-mentioned objectives. The basic equations of the simulation models and the analysis procedure of the experimental data were described in Refs. (3), (4) and (11).

In the single column experiment previously performed by the authors (3)(4), the overall HETP values of Column I were 4~6 cm and the HETP depended little on the column height. The four-column experiment of the authors (9) gave the information that the overall HETP value of 5 cm was roughly valid for evaluation of the separation performance of the columns. Fig. 3 shows the composition distribution of Column I in the cascade operation for

a representative run. The calculated lines are also drawn in the figure under the assumptions that the overall HETP value is 5 cm and the HETP is constant with column height. The experimental and calculational conditions of the run are shown in Table 2. The calculated results are in rough agreement with the experimental observations. For Column I, we can thus confirm the engineering data obtained in the single column experiment.

Columns I and T differ in height and inner diameter of packed section, which may possibly affect the separation performance. Table 3 shows the overall HETP values of Column T for various vapor velocities and reflux ratios. The measured values are about 4~5 cm, and no significant difference is observed in comparison with those for Column I obtained in our previous work. The differences between the columns in packed height and inner diameter are not large. The packing materials used are the same (see Table 1). For the above reasons, no effect of the inner diameter and the packed height is observed on the separation performance of the columns. Figure 4 shows an example of comparison between experimental observations and calculated results for the composition distribution within Column T in the cascade operation. The experimental and calculational conditions are presented in Table 2. The calculated results are in close agreement with the experimental observations, and it can be confirmed for Column T also that the HETP is constant within the column.

### 3.3 Control Method of the Cascade

The control scheme of the columns in the present experiment is illustrated in Fig. 5. For Column I, the pressure is controlled by the condenser load (temperature of the refrigerant helium gas), and the output of the reboiler heater is manipulated to control the liquid level in the reboiler. The liquid level in the reboiler of Column T is controlled by the output of the reboiler heater. The flow rates of output and input streams of the cascade are maintained constant. Hence, the liquid holdups in the reboilers of both columns vary with the flow rate of the bottom stream of Column I (feed stream of Column T). For instance, the liquid holdup of Column I in the reboiler increases with decreasing the flow rate of bottom stream, while that of Column I decreases. The flow rate of the bottom stream of Column I depends on the difference of pressures between the columns. Accordingly, to maintain the liquid holdups in the reboilers of both the columns, the pressures of the columns have to be stabilized.

Figure 6 shows an example of dynamic variation of the pressures and the liquid levels in the reboilers when the reflux ratios are changed stepwise as presented in Table 4. The pressure of Column I was successfully controlled. For Column T, the pressure was maintained constant by adjusting the condenser load. The dynamic behavior of the liquid levels in the reboilers indicates that the liquid holdups within the column vary rapidly and reach steady values within 20 min even for the actual size columns used. Since the pressures of the columns were almost

maintained constant, the liquid holdups in the reboilers were stable after the above rapid variation.

The most critical task of the two-column cascade simulated in the present experiment is to obtain a desired product, which is high purity tritium, from the bottom of the second column<sup>(6)</sup>. As discussed above, the condenser loads of the columns would be key parameters for stable operation of the cascade. To obtain high purity tritium from the product of the cascade, the flow rates of the bottom streams should be chosen as the manipulated variables<sup>(12)</sup>. The liquid levels in the reboilers can be controlled by the flow rates of the top streams. To cope with fluctuation of flow rates of the feed streams, the output of the reboiler heaters are manipulated<sup>(12)</sup>. The control method thus proposed for the cascade is illustrated in Fig. 7.

## 4. CONCLUSION

- (1) The pressure drop of Column T was larger than that of Column I in spite of the smaller packed height of Column T. This phenomenon may result from the differences in the physicochemical properties of the fluid within the column and the inner diameter of the packed section.
- (2) It was verified in the cascade operation that the overall HETP values of Column I were about 5 cm. For Column T, the overall HETP values of 4~5 cm were obtained. No effect of the difference between the columns in the packed height and the inner diameter was observed on the separation performance. It was also confirmed for both the columns that the HETP depended little on the column height.
- (3) A basic control method of the present cascade was proposed. Pressure increases are minimized by adjusting the condenser loads for both the columns. The flow rates of bottom streams are manipulated to control the product purity.

## ACKNOWLEDGEMENT

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## REFERENCES

- (1) J. R. Bartlit, R. H. Sherman, R. A. Stutz, and W. H. Denton, : Cryogenics, 19, 275 (1979).
- (2) T. Yamanishi et al. : JAERI-memo, Japan Atomic Energy Research Institute, Private Communication (1988).
- (3) T. Yamanishi, H. Yoshida, S. Hirata, T. Naito, Y. Naruse, R. H. Sherman, K. M. Gruetzmacher J. R. Bartlit, and J. L. Anderson, : 3rd Topical Meeting, Tritium Technology in Fission, Fusion and Isotopic Applications--May 1-6, 1988--Toronto, Ontario, CANADA (in press).
- (4) T. Yamanishi, H. Yoshida, T. Naito, S. Hirata, R. H. Sherman, J. R. Bartlit, and J. L. Anderson, : JAERI-M, (in press) (1988).
- (5) M. Kinoshita, : Fusion Technol., 6, 629 (1984).
- (6) M. Kinoshita, H. Yoshida, and H. Takeshita, : Fusion Technol., 10, 462 (1986).
- (7) D. Legar, G. Dirian, and E. Roth, : Energ. Nucl. (Paris), 12, 2, 135 (1970) (in French).
- (8) W. R. Wilkes, : MLM-2502, p. 10 Mound Laboratories, Miamisburg, ohio (1978).
- (9) R. H. Sherman, J. R. Bartlit, K. M. Gruetzmacher, H. Yoshida, T. Yamanishi, T. naito, S. Hirata, and Y. Naruse, : 3rd Topical Meeting, Tritium Technology in Fission, Fusion and Isotopic Applications--May 1-6, 1988--Toronto, Ontario, CANADA (in press).
- (10) R. H. Sherman, J. R. Bartlit, and D. K. Veirs, : Fusion Technol., 6, 625 (1984).

- (11) M. Kinoshita, and Y. Naruse, : JAERI-M 9871 (1981).
- (12) M. Kinoshita, J. R. Bartlit, and R. H. Sherman, : Nuclear Technol./Fusion 5, 30 (1984).

Table 1 Specifications of distillation columns

	lead column	second column
Inner diameter (cm)	2.84	2.50
Packed height (cm)	412	320
Volume of condenser (cm <sup>3</sup> )	2000	2000
Volume of packed section (cm <sup>3</sup> )	2600	1600
Packing material	Heli-Pak (SUS-316) 4.4x4.4x2.3 mm	

Table 2 Experimental and calculational conditions

	Run A	Run B
Column	I	T
Pressure (Torr)	772	636
Flow rate (mol/sec)		
top	$2.2 \times 10^{-3}$	$8.5 \times 10^{-4}$
bottom	$4.0 \times 10^{-3}$	$4.9 \times 10^{-4}$
Vapor velocity (cm/sec)	4.9	2.21
Liquid holdup in reboiler (mol)	1.47	1.01
Number of total theoretical stages	84	82
Feed stage number	54	18

Table 3 The overall HETP values for Column T

Overall HETP (cm)	4.0	4.5	4.5	3.9	4.7	5.0
Vapor velocity (cm/sec)	2.2	6.4	7.3	7.5	8.1	8.3
Reflux ratio (-)	4.5	39.6	10.5	8.9	22.1	19.2

Table 4 Variation of experimental conditions

		Column I	Column T
Output of reboiler heater (W)		20→20	23→18
Flow rate (mol/sec $\times 10^{-4}$ )	top	22.0→7.1	33.0→3.7
	bottom	40.4→6.7	7.0→3.0

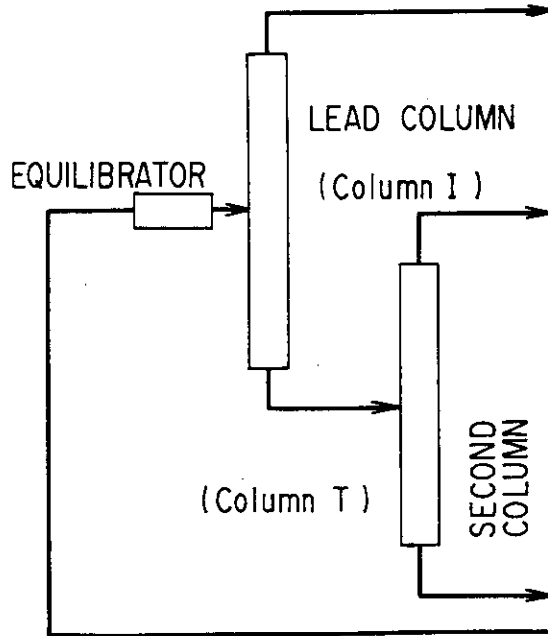


Fig.1 Configuration of two-column cascade

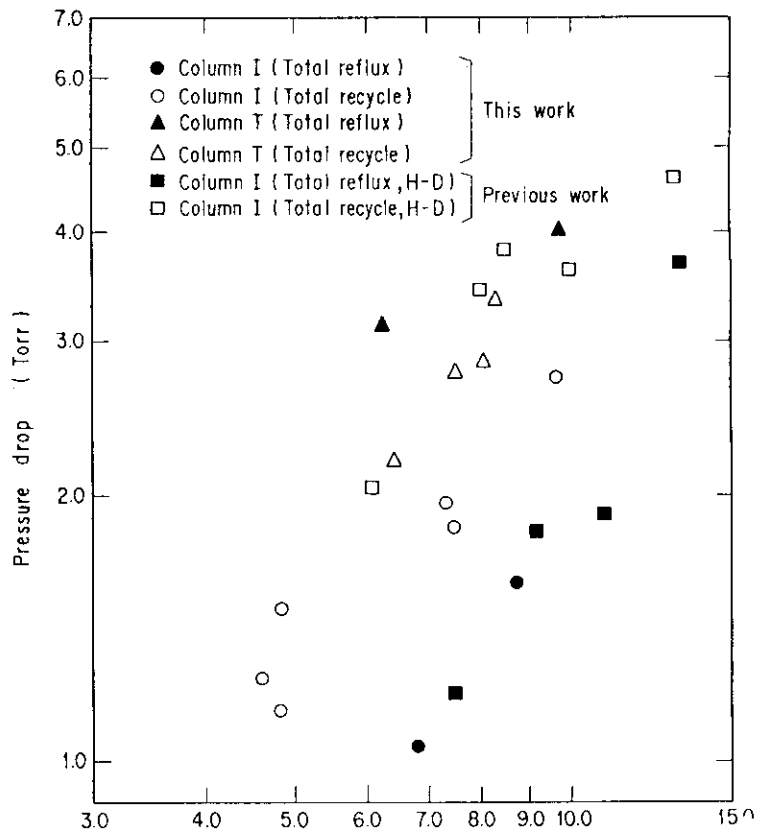


Fig.2 Variation of pressure drop across columns with vapor velocity

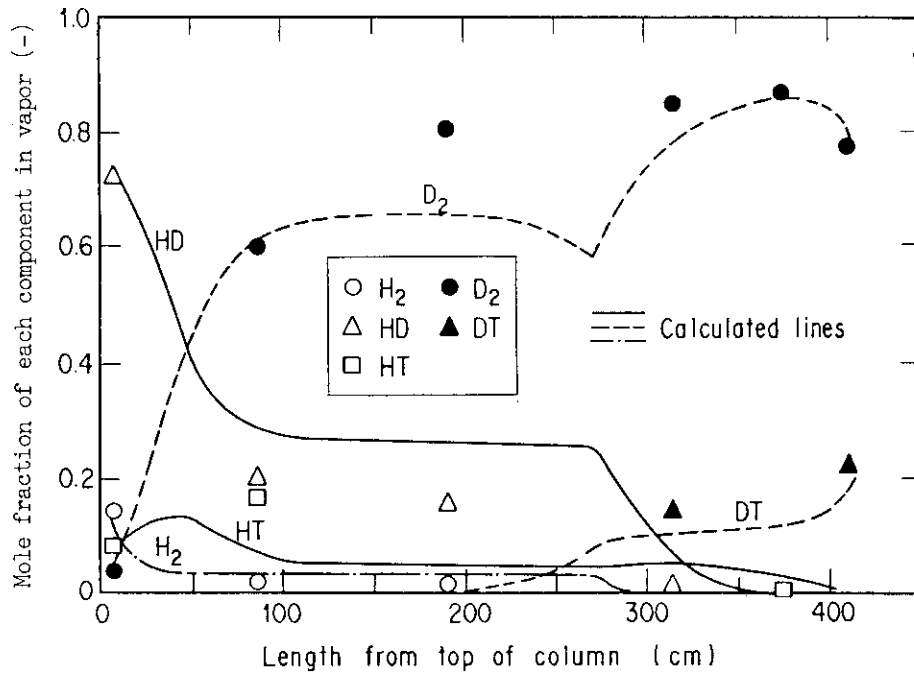


Fig.3 Comparison between experimental observation and calculated result for composition distribution within Column I for Run A

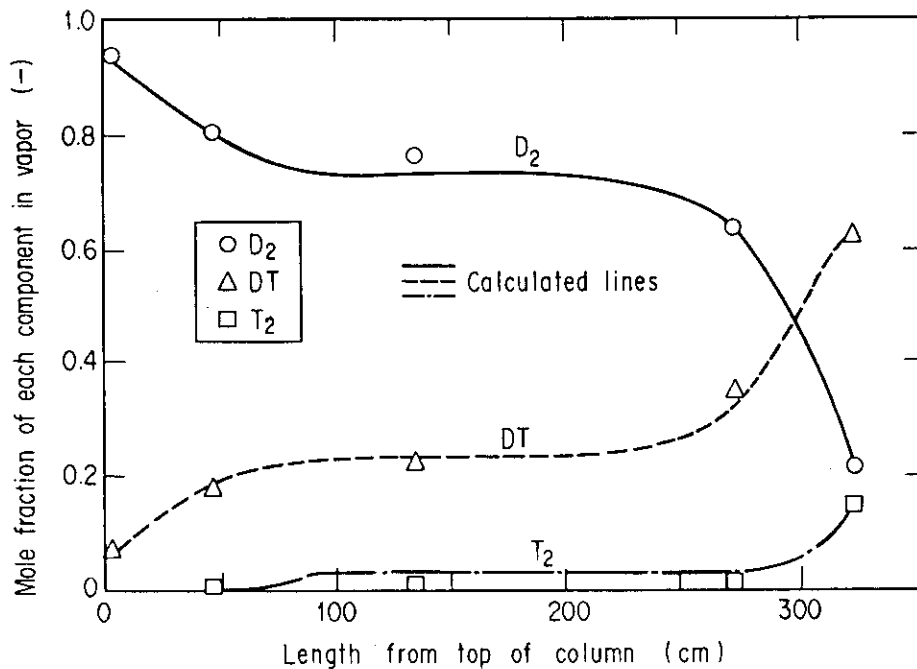


Fig.4 Comparison between experimental observation and calculated result for composition distribution within column T for Run B

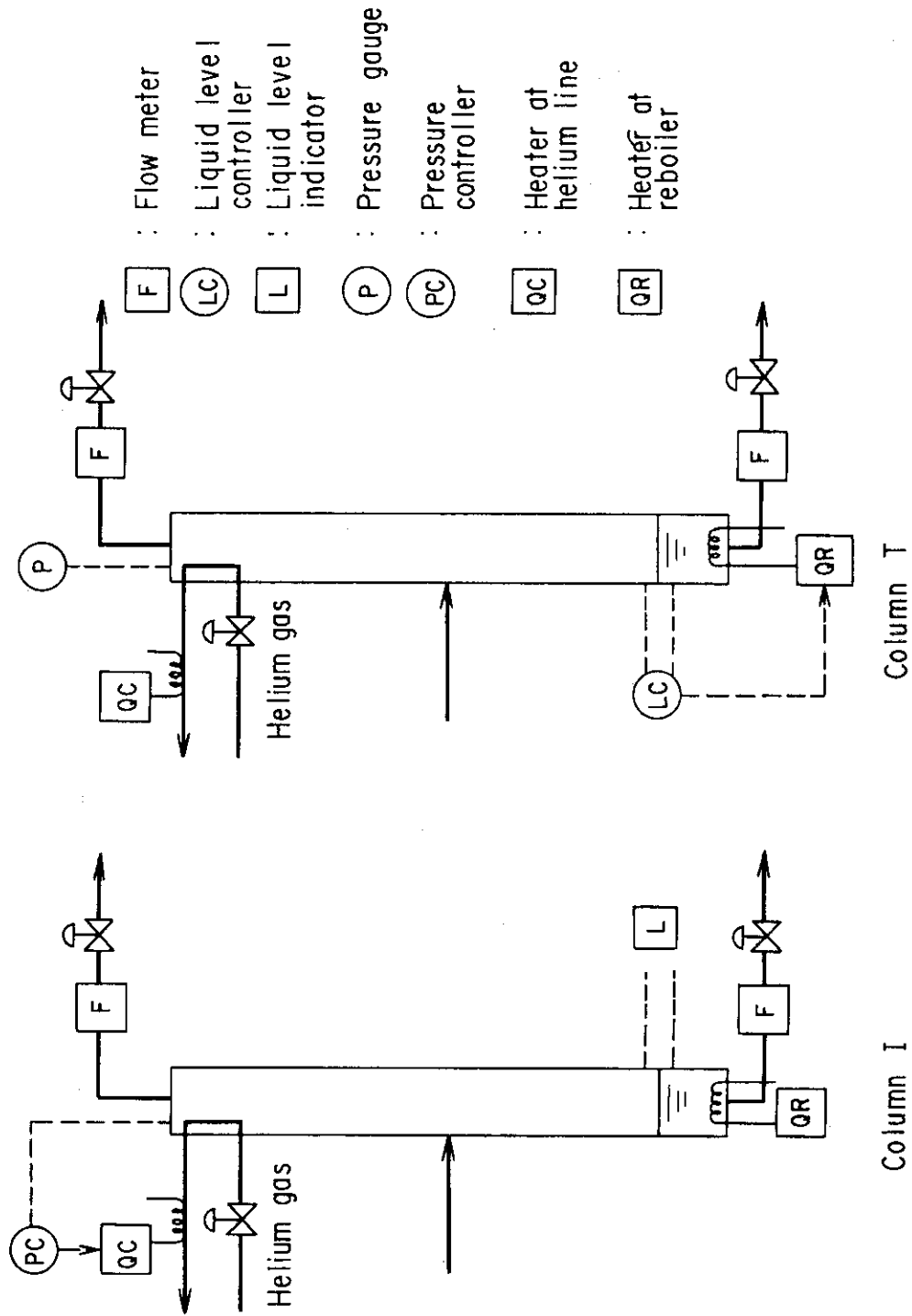


Fig. 5 Control scheme for columns

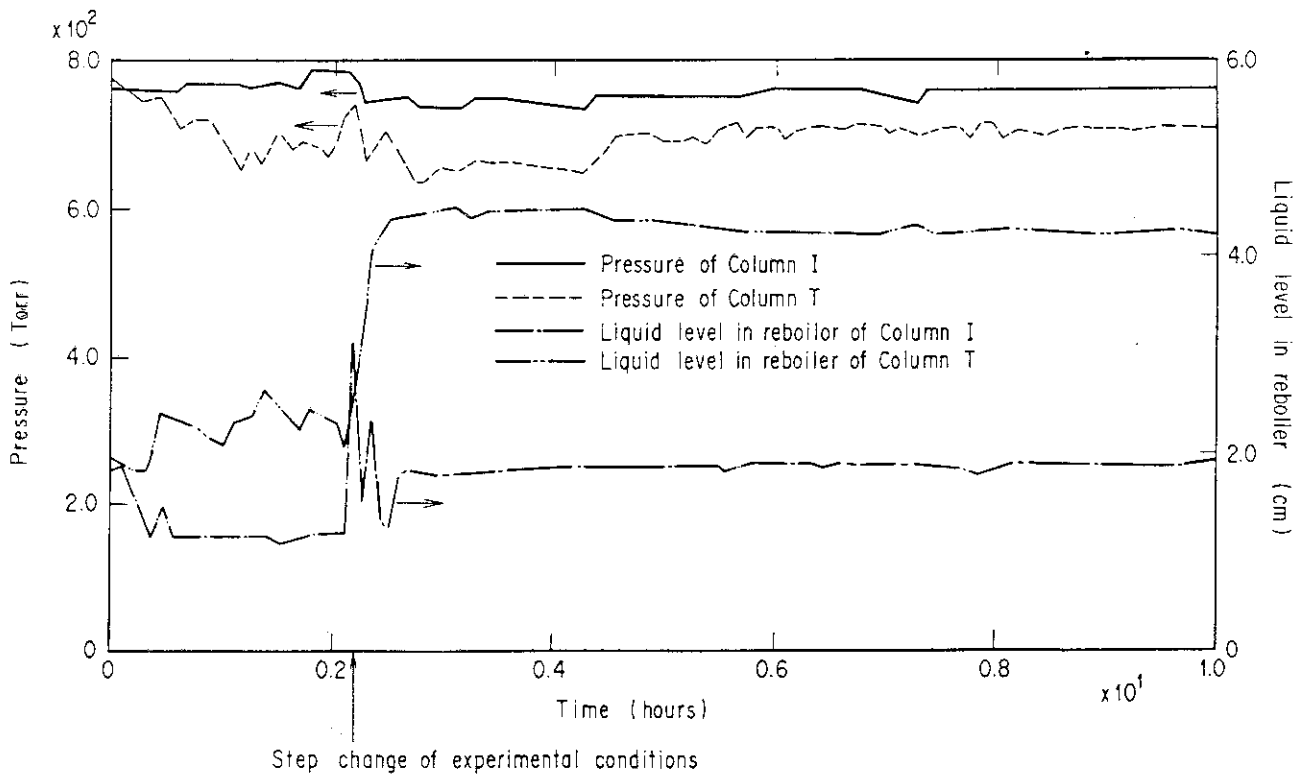


Fig.6 Dynamic variation of pressures and liquid levels in reboilers of columns for step change of reflux ratios of columns

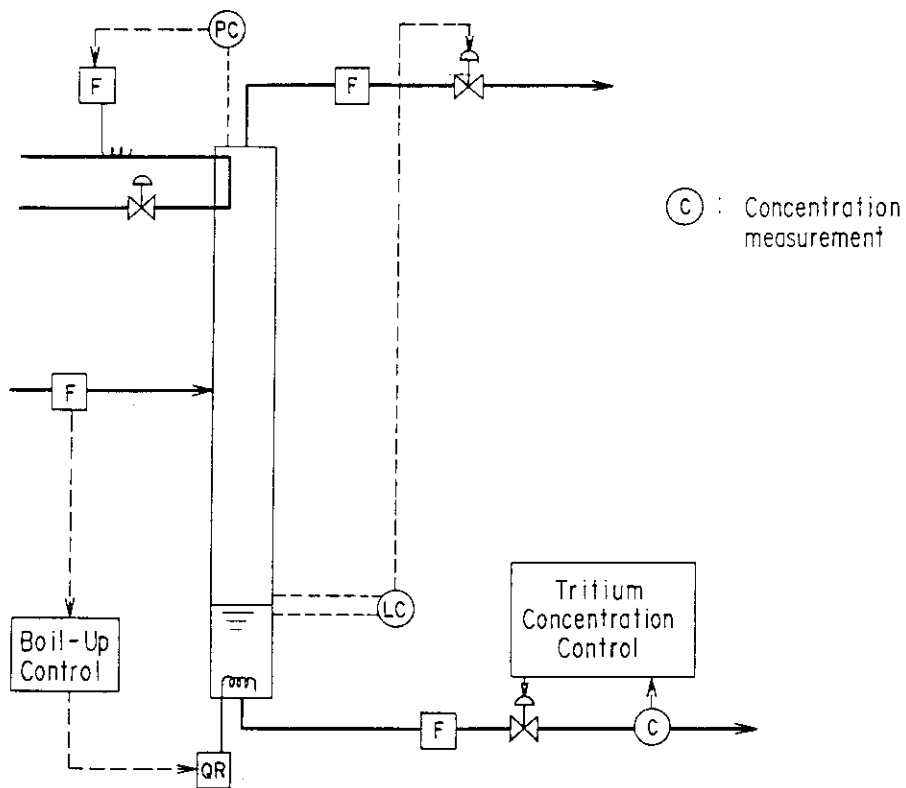


Fig.7 Proposed control method for columns



Appendix	Expeirmental results of all runs
Table A-I	Experimental conditions of all runs
Table A-II	Results of GC analyses for all runs
Figure A-1	Dynamic variation of pressures of columns during experiment
Figure A-2	Dynamic variation of pressure drops of columns during experiment
Figure A-3	Dynamic variation of liquid levels of columns during experiment
Figure A-4	Dynamic variation of output of reboiler heaters of columns during experiment
Figure A-5	Dynamic variation of flow rates of top and bottom streams of Column I during experiment
Figure A-6	Dynamic variation of flow rates of top and bottom streams of Column T during experiment

Table A-1 Experimental conditions of all runs 1/2

Run& col. No.	Pressure of column	Pressure Drop (Torr)	Liquid level in reboiler	Output of reboiler heater	Flow rate (cc/min) Top Bottom	Reflux ratio
1 col -I	715 Torr 0.941 atm	1.05	65.7mm 3.83mol	25.9W 77.97mol/h 6.8cm/s	0 0	--
1 col -T	730 Torr 0.961 atm	3.17	34.9mm 1.99mol	17.7W 53.88mol/h 6.20cm/s	0 0	--
2 col -I	763 Torr 1.004 atm	1.58	19.7mm 1.08mol	35.4W 102.7mol/h 8.72cm/s	0 0	--
2 col -T	719 Torr 0.947 atm	4.09	(33mm) (1.9mol)	28.7W (84.3mol/h) (9.82cm/s)	0 0	--
3 col -I	787 Torr 1.036 atm	1.98	27.3mm 1.52mol	31.3W 92.33mol/h 7.33cm/s	2989,3428 (8.01,9.18 mol/h)	10.53
3 col -T	730 Torr 0.961 atm	2.71	20.7mm 1.17mol	16.3W 48.14mol/h 5.56cm/s	2773,655 (7.43,1.75 mol/h)	5.48
4 col -I	767 Torr 1.009 atm	1.27	37.3mm 2.10mol	18.8W 56.36mol/h 4.59cm/s	3993,1991 (10.7,5.33 mol/h)	4.27
4 col -T	732 Torr 0.963 atm	3.17	18.5mm 1.03mol	25.2W 72.22mol/h 8.31cm/s	1336,655 (3.58,1.75 mol/h)	19.18
5 col -I	775 Torr 1.020 atm	2.78	32.7mm 1.84mol	40.6W 119.2mol/h 9.65cm/s	945,1800 (2.53,4.82 mol/h)	46.08
5 col -T	636 Torr 0.837 atm	1.84	18.2mm 1.01mol	5.2W 17.0mol/h 2.21cm/s	1145,655 (3.07,1.75 mol/h)	4.54
6 col -I	766 Torr 1.007 atm	1.87	27.4mm 1.54mol	30.8W 90.28mol/h 7.42cm/s	945,4336 (2.52,11.6 mol/h)	34.67

Table A-1 Experimental conditions of all runs 2/2

6 col -T	688 Torr 0.905 atm	2.76	12.4mm 0.65mol	21.7W 62.0mol/h 7.49cm/s	2336,2000 (6.26,5.36 mol/h)	8.91
7 col -I	772 Torr 1.016 atm	1.53	26.4mm 1.47mol	20.4W 60.31mol/h 4.9cm/s	2960,5427 (7.93,14.5 mol/h)	6.61
7 col -T	665 Torr 0.875 atm	2.89	16.6mm 0.91mol	22.6W 64.4mol/h 8.06cm/s	4482,945 (12.0,2.53 mol/h)	4.37
8 col -I	757 Torr 0.997 atm	0.09	23.0mm 1.28mol	20.2W 59.47mol/h 4.80cm/s	960,900 (2.57,2.41 mol/h)	22.13
8 col -T	704 Torr 0.927 atm	2.19	40.0mm 2.27mol	17.8W 54.3mol/h 6.43cm/s	500,400 (1.34,1.07 mol/h)	39.57

Table A-2 Results of GC analyses for all runs 1/7

## Run 1

## Composition of column-I at the transient state

Position	--	S1			
Time(min)		0	105	148	184
Component					
He	---	---	---	---	---
H2		7.91	10.81	9.52	8.43
HD		41.95	30.22	29.27	28.05
HT		26.26	25.71	26.45	26.29
D2		13.93	15.15	16.50	18.11
DT		6.94	14.23	14.40	15.17
T2		---	3.89	3.85	3.96

## Composition profile of column I at the steady state

Component	Position (height from bottom : cm)						
	AN-1 (412) Top	S1 (327.7)	A1S (226.1)	A (144.8) Feed	A2S (99.1)	S1A (38.1)	AN-2 (0) Bottom
He	--	--	--	--	--	--	--
H2	--	8.43	--	--	--	--	--
HD	--	28.05	5.30	--	12.91	--	--
HT	--	26.29	1.11	--	2.54	--	--
D2	--	18.11	90.84	--	74.32	92.59	70.79
DT	--	15.17	2.75	--	10.24	7.41	28.00
T2	--	3.96	--	--	--	--	1.21

## Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 (320) Top	S3 (261.6)	S3A (231.1) (Feed)	S3B (190.5) (83.8)	AN8 (0) Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	2.14	--	--	--	--
HT	1.08	--	--	--	--
D2	88.53	100.0	0.31	39.05	31.14
DT	8.25	--	96.87	59.00	55.87
T2	--	--	2.82	1.95	12.99

Table A- 2

2/7

Run 2

Composition profile of column-I at the steady state

Component	AN-1	S1	A1S	Position			
				A	A2S	S1A	AN-2
He	--	--	--	--	--	--	--
H2	--	0.15	--	--	--	--	--
HD	15.24	4.29	--	--	--	--	--
HT	--	18.95	--	--	--	--	--
D2	84.76	73.63	--	--	--	87.96	57.96
DT	--	2.70	--	--	--	12.04	39.46
T2	--	0.28	--	--	--	--	2.58

Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 Top	S3 (Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	2.31	--	--	--	--
HT	1.47	--	--	--	--
D2	93.27	100.0	97.52	36.00	--
DT	2.96	--	2.48	59.43	--
T2	--	--	--	4.57	--

Run 3

Composition of column-I at the transient state

Position	--	AN-2		
Time(min)		12	47	83
Component				175
He	---	---	---	---
H2	---	---	---	---
HD	---	---	---	---
HT	---	---	---	---
D2		80.41	81.73	83.67
DT		18.33	17.14	15.29
T2		1.28	1.13	1.04
				90.24
				9.14
				0.62

Table A-2

3/7

Run 3

## Composition of column-T at the transient state

Position	AN-8			
Time(min)	0	34	70	187
Component				
He	---	---	---	---
H2	---	---	---	---
HD	---	---	---	---
HT	---	---	---	---
D2	11.27	10.59	12.08	14.00
DT	72.13	65.88	65.16	65.30
T2	16.60	23.53	22.76	20.70

## Composition profile of column-I at the steady state

Component	Position						
	AN-1	S1	A1S	A	A2S	S1A	AN-2
He	--	--	--	--	--	--	--
H2	21.20	1.82	1.44	7.87	--	--	--
HD	69.27	16.12	11.81	33.89	0.48	--	--
HT	6.45	14.29	2.84	3.75	0.18	--	--
D2	2.58	66.18	83.48	42.57	92.24	91.72	90.24
DT	0.51	1.59	0.43	11.31	7.10	8.28	9.14
T2	--	--	--	0.62	--	--	0.62

## Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 Top	S3 (Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	--	--	--	--	--
HT	--	--	--	--	--
D2	98.45	22.18	87.90	33.26	14.00
DT	1.55	76.77	11.96	61.47	65.30
T2	--	1.05	0.14	5.27	20.70

Table A-2

4/7

Run 4

Composition profile of column-I at the steady state

Component	Position						
	AN-1	S1	A1S	A	A2S	S1A	AN-2
He	--	0.12	--	0.80	--	--	--
H2	28.81	4.42	1.65	18.12	0.01	--	--
HD	55.99	18.78	14.62	39.09	1.25	--	--
HT	10.64	8.22	1.69	6.32	0.09	0.05	--
D2	4.32	67.33	80.63	24.13	85.41	89.20	64.15
DT	0.24	1.14	1.41	10.72	13.02	10.60	33.64
T2	--	--	--	0.82	0.22	0.15	2.21

Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 Top	S3 (Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	--	--	--	--	--
HT	--	--	--	--	--
D2	100.00	97.61	89.39	52.38	18.02
DT	--	2.39	10.50	45.78	60.89
T2	--	--	0.11	1.84	21.09

Run 5

Composition of column-T at the transient state

Position Time(min) Component	AN-11							
	0	12	30	48	64	84	98	118
He	--	--	--	--	--	--	--	--
H2	--	--	--	--	--	--	--	--
HD	--	--	--	--	--	--	--	--
HT	--	--	--	--	--	--	--	--
D2	98.67	97.25	98.57	97.30	94.85	95.15	94.22	95.33
DT	1.33	2.75	1.43	2.70	5.15	4.86	5.78	4.67
T2	--	--	--	--	--	--	--	--

Table A-2

5/7

Run 5

Composition profile of column-I at the steady state

Component	AN-1	S1	A1S	Position			AN-2
				A	A2S	S1A	
He	1.16	--	--	--	--	--	--
H2	12.66	2.92	0.13	--	--	--	--
HD	80.77	21.93	9.40	14.92	--	--	--
HT	1.24	30.91	1.11	1.05	0.49	0.07	--
D2	4.03	33.69	88.86	69.02	92.73	91.90	75.31
DT	0.14	8.72	0.50	13.23	6.78	8.03	23.54
T2	--	1.84	--	1.78	--	--	1.15

Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)					
	AN-11 Top	S3	(Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--	--
H2	--	--	--	--	--	--
HD	--	--	--	--	--	--
HT	--	--	--	--	--	--
D2	93.07	80.71		76.62	63.81	18.47
DT	6.93	18.51		22.20	34.68	62.39
T2	--	0.78		1.18	1.51	19.14

Run 6

Composition profile of column-I at the steady state

Component	AN-1	S1	A1S	Position			AN-2
				A	A2S	S1A	
He	1.60	--	--	--	--	--	--
H2	14.99	7.33	--	0.73	--	--	--
HD	61.42	16.69	--	11.89	--	--	--
HT	10.62	39.03	--	2.23	--	0.15	--
D2	7.76	16.56	--	56.70	--	86.96	66.04
DT	3.17	14.25	--	26.06	--	12.68	31.12
T2	0.45	6.14	--	2.38	--	0.21	2.83



Table A- 2

6/7

Run 6

## Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 Top	S3 (Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	--	--	--	--	--
HT	0.06	--	--	--	--
D2	98.94	87.61	80.54	75.37	33.59
DT	1.00	12.24	18.67	24.60	60.89
T2	--	0.15	0.79	0.03	5.52

Run 7

## Composition profile of column-I at the steady state

Component	Position						
	AN-1	S1	A1S	A	A2S	S1A	AN-2
He	1.18	0.07	0.07	0.40	--	--	--
H2	14.41	1.82	1.22	3.99	--	--	--
HD	72.30	19.44	15.59	26.37	1.17	0.11	--
HT	8.35	16.91	2.22	2.94	0.30	1.07	0.28
D2	3.11	59.56	79.84	51.58	84.42	86.45	76.99
DT	0.66	2.06	1.04	14.26	13.83	12.19	22.02
T2	--	0.14	0.01	0.47	0.28	0.19	0.71

## Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 Top	S3 (Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	0.06	0.14	--	--	--
HT	0.37	--	--	--	--
D2	97.10	88.09	82.74	52.30	14.72
DT	2.47	11.53	16.66	45.56	68.09
T2	--	0.25	0.60	2.14	17.19

Table A- 2 7/7

Run 8

## Composition profile of column-I at the steady state

Component	Position						
	AN-1	S1	A1S	A	A2S	S1A	AN-2
He	4.25	0.05	--	--	--	--	--
H2	75.02	6.99	--	--	--	--	--
HD	6.70	14.91	--	--	--	--	--
HT	--	46.44	--	--	--	--	--
D2	10.66	12.13	--	--	--	95.30	67.80
DT	3.37	12.36	--	--	--	4.59	30.57
T2	--	7.13	--	--	--	0.11	1.63

## Composition profile of column-T at the steady state

Component	Position (height from bottom : cm)				
	AN-11 Top	S3 (Feed)	S3A	S3B	AN8 Bottom
He	--	--	--	--	--
H2	--	--	--	--	--
HD	--	--	--	--	--
HT	--	--	--	--	--
D2	100.00	99.50	97.64	95.68	38.10
DT	--	0.50	2.36	4.24	54.66
T2	--	--	--	0.09	7.25

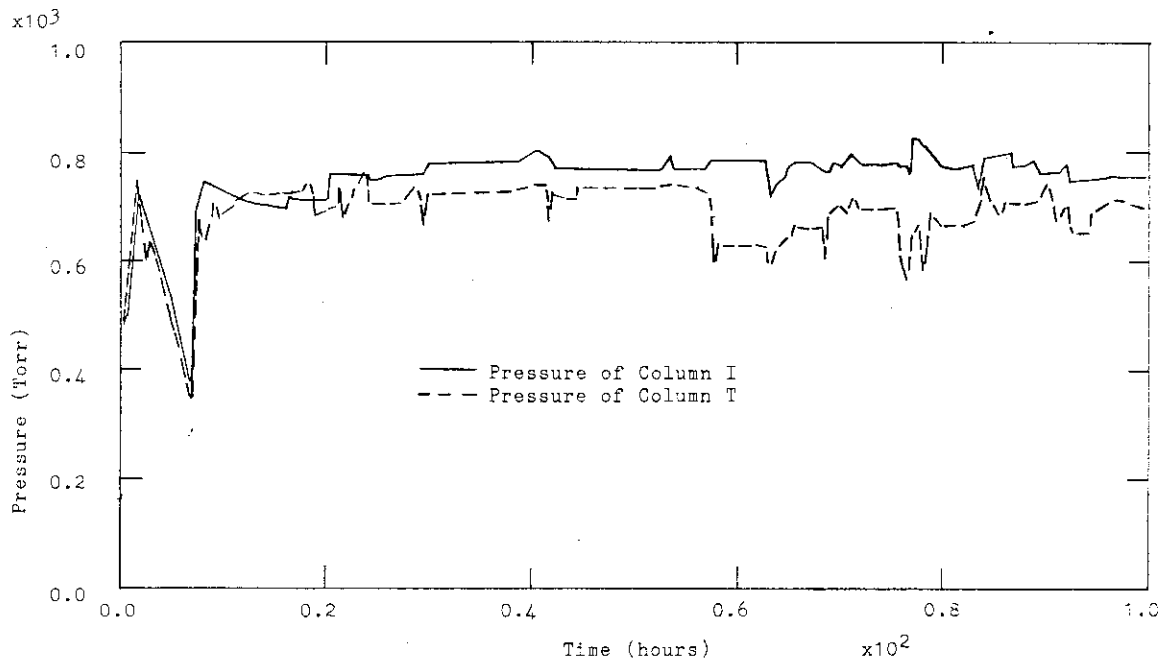


Fig. A-1 Dynamic variation of pressures of Columns during experiment

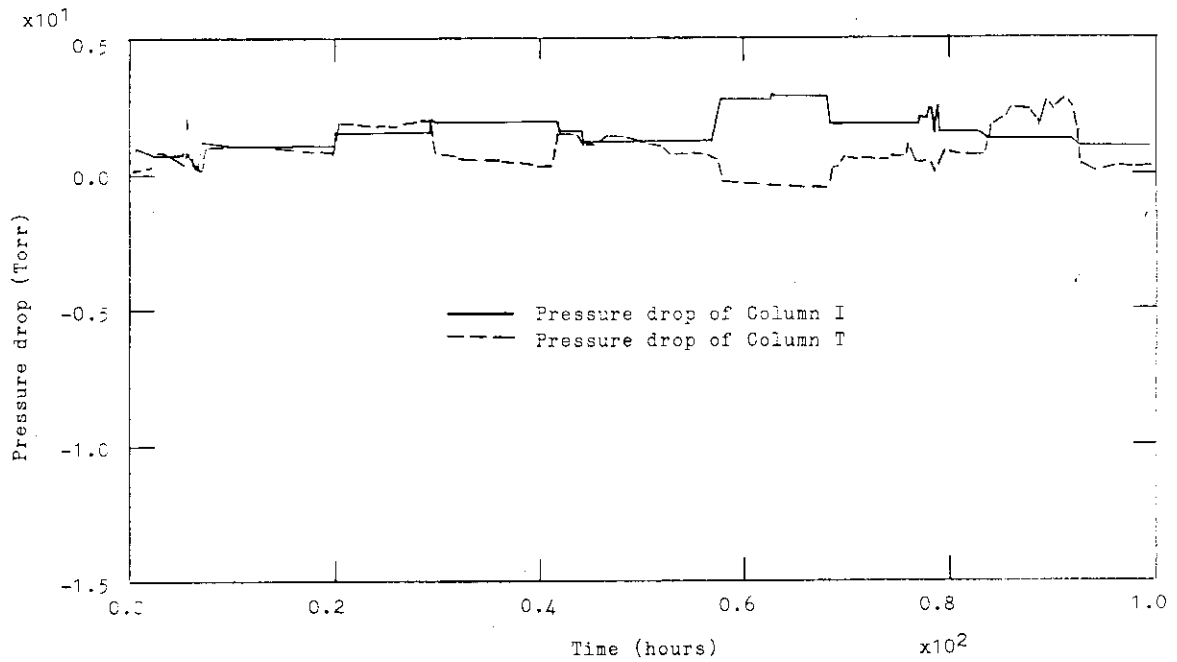


Fig. A-2 Dynamic variation of pressure drops of columns during experiment

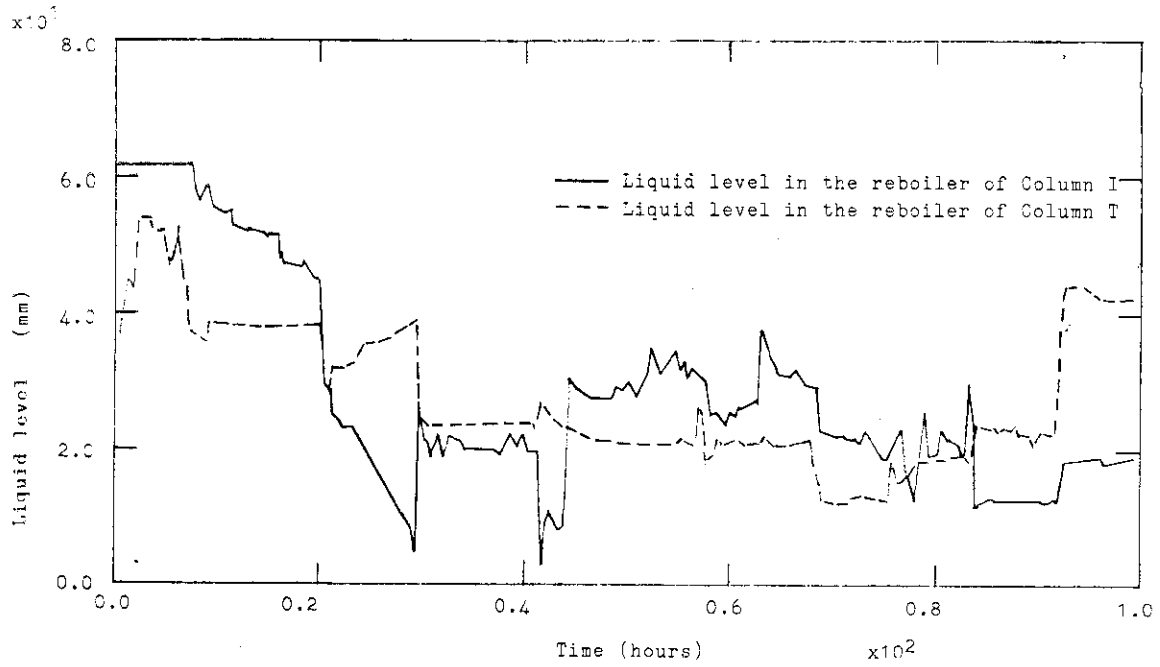


Fig. A-3 Dynamic variation of liquid levels of columns during experiment

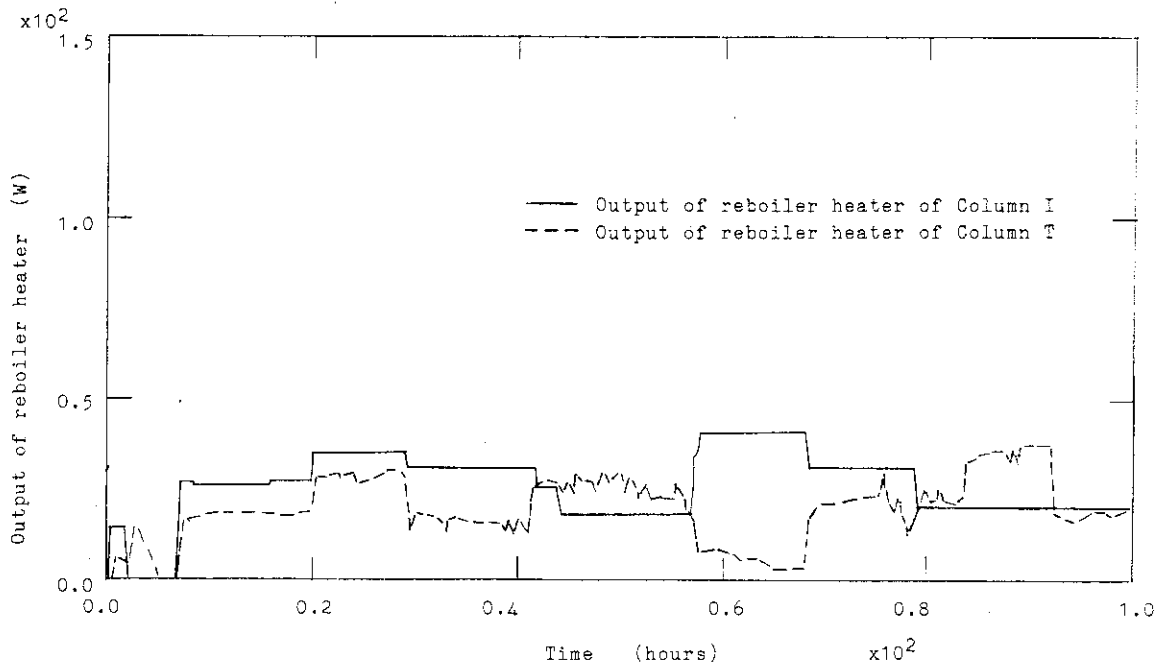


Fig. A-4 Dynamic variation of output of reboiler heaters of columns during experiment

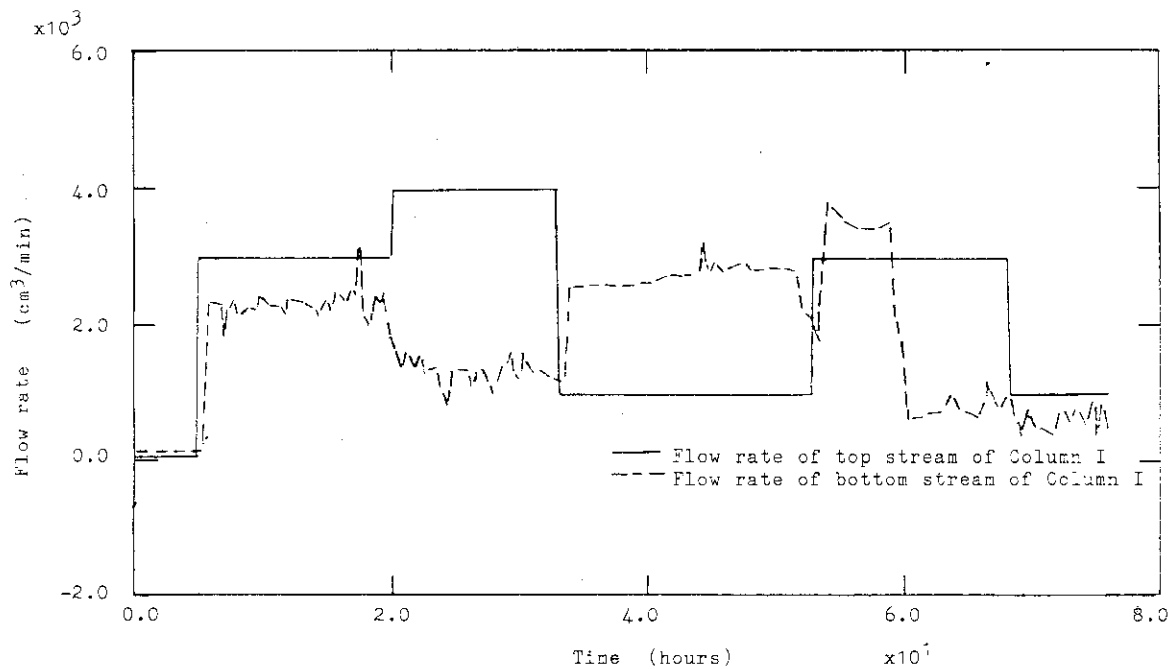


Fig. A-5 Dynamic variation of flow rates of top and bottom streams of Column I during experiment

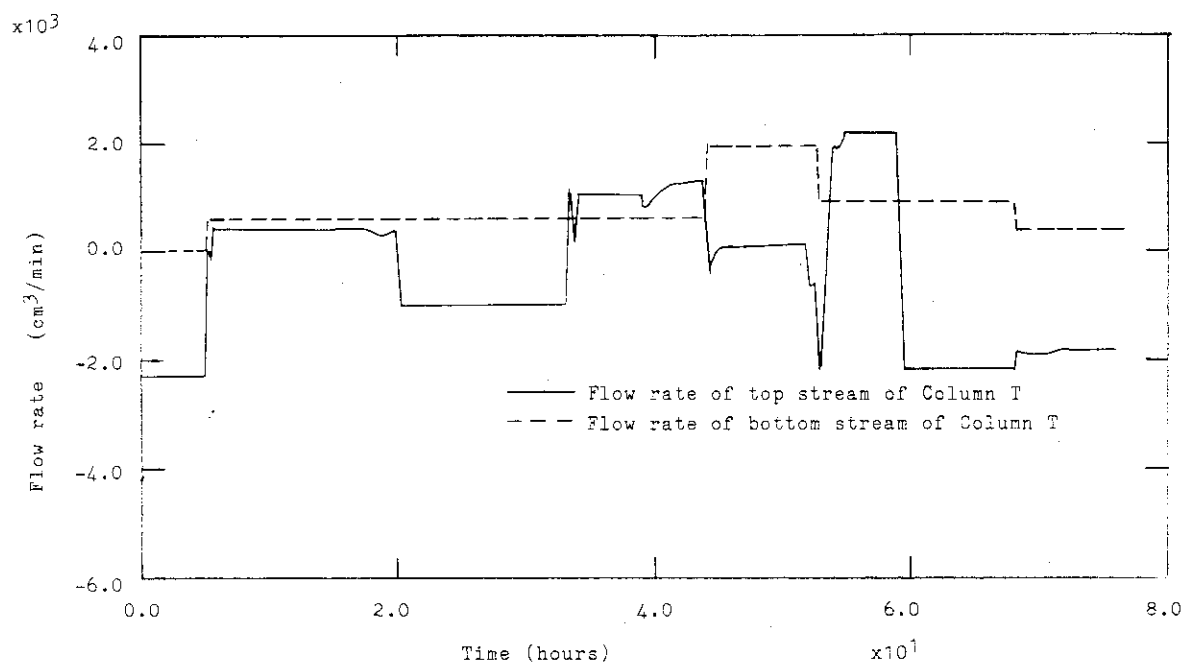


Fig. A-6 Dynamic variation of flow rates of top and bottom streams of Column T during experiment