

**SEPARATION OF HELIUM AND HYDROGEN
ISOTOPES BY ISS AT THE TSTA**

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INTRODUCTION

Several experiments were performed with the Isotope Separation System (ISS) at the Tritium Systems Test Assembly (TSTA) from 1988 through 1989. These experiments included two-column experiments with D-T mixtures and four-column experiments with D-T mixtures and impurities (He and H₂). The objectives of these experiments were (1) to obtain fundamental data of cryogenic distillation columns under a wider range of operational conditions than in previous work, (2) to observe the influence of He on the operation of cryogenic distillation columns, and (3) to obtain fundamental data of cryogenic distillation columns under the influence of He.

Previous ISS experiments have been performed to obtain fundamental data by using single column or two columns of the ISS at TSTA with D-T mixtures.^{1,2} In previous experiments, the HETP value and the pressure drop in the packed section were measured in the vapor velocity range of 5 - 15 cm/s and the reflux ratio of 5 - 40. The result showed that the HETP value was not influenced by the vapor velocity and there was no evidence of a flooding point or loading point. This year, the experiments were expected to be performed in the vapor velocity range of 2 - 15 cm/s and reflux ratio range of less than 230. Also, continuous separation of He from D-T mixtures was tested for the purpose of obtaining data of ISS operation under the influence of He.

EXPERIMENTAL

The ISS at the TSTA consists of four cryogenic distillation columns and two hydrogen isotope equilibrators as shown in Fig. 1. The top stream of Column I is mixed with a Neutral Beam Return stream (D₂) and fed to Column H through the equilibrators. In Column H, the waste gas is detritiated and pumped to the Tritium Waste Treatment System (TWT). The bottom stream of Column H is fed to Column D. The top stream of Column D is recycled to the feed stream of Column H as the NBI stream. The bottom stream of Column H is fed to Column T where DT and T₂ are separated. The other streams are recycled as the feed stream of Column I (feed of ISS).

In two-column experiments, Column I and Column T were used to obtain the data on the pressure drop in the packed section and the Height Equivalent to a Theoretical Plate (HETP). The pressure drop in the packed section is the physical property that expresses whether flooding occurs or not. If flooding occurs, it becomes much higher (more than about 10 torr) than in the stable state. HETP expresses the efficiency of the separation in the distillation

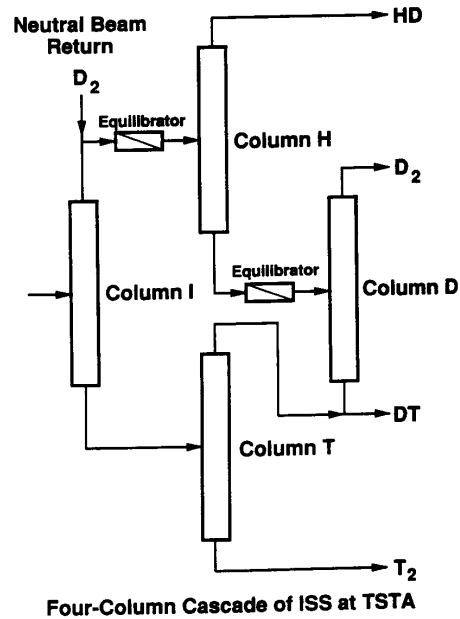


Fig. 1 Cryogenic Distillation Columns and Hydrogen Isotope Equilibrators.

column. In this study, the HETP value was obtained by making the best fit between the observed value and the calculated value of mole fractions of hydrogen isotopes at the top and the bottom of columns. The major experimental conditions of two-column experiments are listed in Table 1.

Table 1. Experimental Conditions of Two-Column Experiments.

	Column I	Column T
pressure (torr)	660 - 800	540 - 790
vapor velocity (cm/s)	2.0 - 15.0	3.0 - 15.0
top flow rate (cc/min)	2000 - 3200	450 - 1100
bottom flow rate (cc/min)	750 - 2050	220 - 1300
reflux ratio (-)	4.3 - 30	7.0 - 70

In four-column experiments, the whole ISS was used to test the separation of He and HD

from D-T mixtures. He and H₂ were injected in the feed stream of Column I and He and HD were pumped out from the top of Column H as the waste gas. At the desired point of stable and safe operation of ISS, the tritium level at the top of Column H must be as low as possible. Thus the reflux ratio (defined as the ratio of the amount of liquid going down from the condenser to the amount of gas leaving the condenser) of Column H is one of the most important parameters of the operation of ISS. The key issue of these four-column experiments was to know the minimum reflux ratio which can achieve the design value (20 Ci/y) of tritium loss from ISS. For this purpose, the flow rate of the top stream of Column H (the waste gas stream) was gradually increased so that the reflux ratio of Column H was varied from 233 to 30. The major experimental conditions of four-column experiments are listed in Table 2.

Table 2. Experimental Conditions of Four-Column Experiments.

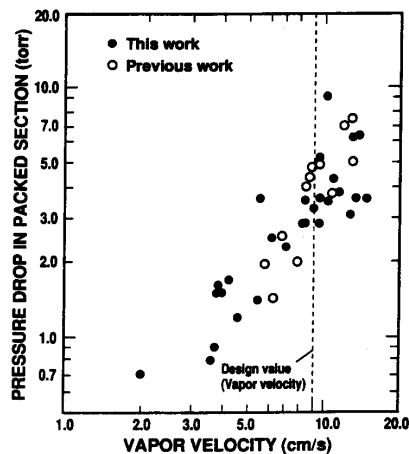
	I	H	D	T
pressure (torr)	870	840	600	600
vapor velocity (cm/s)	5.5 - 9.5	5.8 - 10.4	7.0	7.0
top flow rate (cc/min)	2500	50 - 300	---	2000
bottom flow rate (cc/min)	4000	5700 - 6950	500	2000
reflux ratio (-)	8 - 13	37 - 233	5	45

RESULTS AND DISCUSSION

Figure 2 shows the vapor velocity dependence of the pressure drop in the packed section. In this figure, the dark circle is the result obtained in this study and the white circle is the result of previous work. The data obtained in this study shows good agreement with the result of the previous work. As can be seen from this figure, there is not an unusual pressure drop increase. Thus it can be said that no flooding or loading occurred in the vapor velocity range of 2 - 15 cm/s. The design value of the vapor velocity of ISS at the TSTA is about 9 cm/s which is shown as the broken line in Fig. 2.

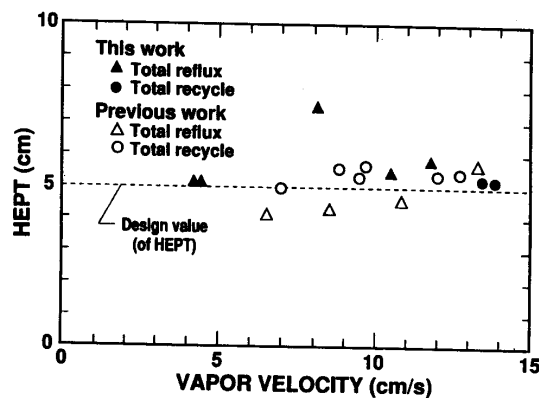
Figure 3 shows the vapor velocity dependence of the HETP value obtained by these experiments. In this figure, dark symbols represent the result of this work and white symbols represent the result of the previous work. The result of this study shows good agreement with the previous work. As can be seen from this figure, no vapor velocity dependence of HETP was observed in the vapor velocity range of 4 - 14 cm/s. Also, the HETP value observed in this work was from 4.5 to 6 cm which is close to the design value (5 cm).

Figure 4 shows the observed atom ratio of H, D, T, and He at the feed point of Column I in the four-column experiments with D-T mixtures and impurities. In this figure, the times of the initiation and termination of impurities injection are noted with broken lines. It can be seen from this figure that the D-T ratio of



Relation Between Pressure Drop in Packed Section and Vapor Velocity

Fig. 2



Effect of Vapor Velocity on HETP Value

Fig. 3

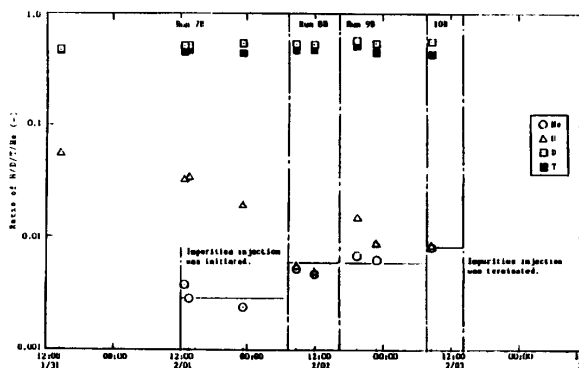


Fig. 4. Ratio of H/D/T/He at Feed of Column I.

the feed stream was almost stable (about 60 - 40%). Also, it is known that the concentration of the He was 0.22, 0.56, and 0.8% in the feed stream when the flow rate was almost 6500 cc/min.

Figure 5 shows the observed atom ratio of H, D, T, and He in the top stream of Column H in the four-column experiments. During this experiment, the concentration of He in the top stream of Column H was about 18, 21, 27 and 20% in Run 7B, 8B, 9B, and 10B, respectively. About 7% of the residual He which was used for purging ISS was detected before the initiation of impurities injection. The operation of Column H was successfully done in these experiments, however, the tritium level at the top of Column H became too high, at the end of Run 10B, to continue flowing the waste gas stream to TWT. There can be a value of the reflux ratio between 54 (Run 9B) and 233 (Run 7B) below which the separation deteriorates and thus the design specification of 20 Ci/y tritium release to the waste stream cannot be met. Additionally, there is a suspicion that tritium was entrained from Column I to Column H because of too high a flow rate of the top stream of Column I. It is known from dynamic simulation study of ISS that it can take more than 24 hours to get a steady mole fraction profile in Column H because of the small flow rate of the top stream (50 - 100 cc/min out of 6000 cc/min).

Figures 6 and 7 are the comparison of observed and calculated mole fractions in Column I and H in Run 7B, respectively. Calculation was done assuming an HETP of 5 cm. As can be seen from Fig. 6, good agreement is obtained between the observed value and the calculated value except for the DT and HT concentrations. A higher concentration of DT and HT is observed in the upper part of Column I than expected by the calculation. The top stream of Column I is fed to Column H with the NBI return stream. Thus tritium is thought to be transferred to Column H.

Figure 7 shows the mole fraction in Column H obtained at the same time as Fig. 6. At this time, no unusually high concentration of tritium was detected in the top stream of Column H. However, attention should be paid to the high concentration of HT, DT, and even T_2 in the packed section. In particular, HT tends to be distributed in Column H because its boiling point is close to the boiling point of D_2 . HT in the packed section of Column H does not come out of the column immediately. It seems to have come out in Run 10B.

In this study, it was demonstrated that impurities were continuously separated from D-T mixtures successfully and 27% of He was an acceptable concentration in the condenser of Column H. Further careful experiments are necessary to obtain basic data on the influence of He on the operation of cryogenic distillation columns in detail.

CONCLUSION

The result of experiments of ISS at TSTA from 1988 through 1989 is summarized as follows:

- 1) The data of pressure drop in the packed section showed the same tendency as the

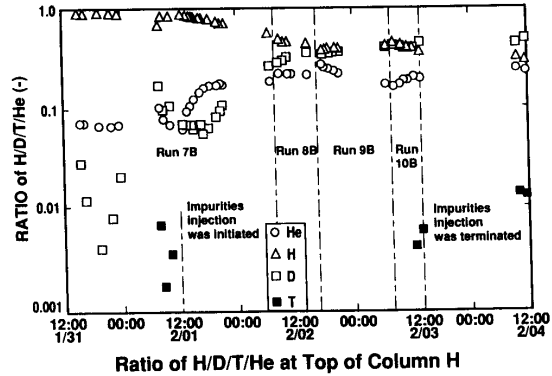


Fig. 5

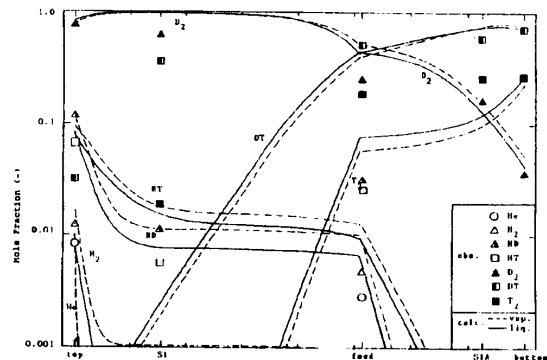


Fig. 6 Mole Fraction Profile in Column I. top flow = 4.3 mol/h, bottom flow = 10.8 mol/h, reflux ratio = 29.0.

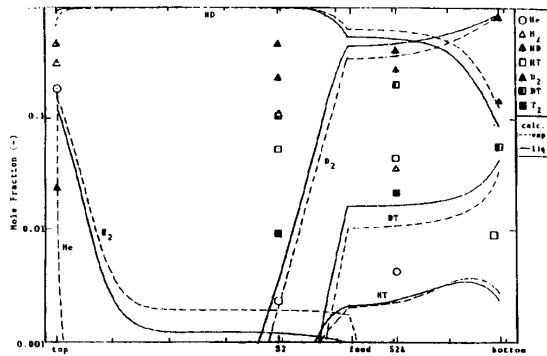


Fig. 7 Mole Fraction Profile in Column I. top flow = 0.267 mol/h, bottom flow = 10.0 mol/h, reflux ratio = 111.1. Feed composition is calculated on the basis of steady state simulation of Column I.

previous work at TSTA and did not show any evidence of the flooding point or loading point in the range of vapor velocity of 2 - 15 cm/s.

- 2) The HETP values obtained in this study were 4.5 - 6 cm and did not show dependence on the vapor velocity in the vapor velocity range of 4 - 14 cm/s.
- 3) The maximum concentration of He for normal operation of columns is greater than 27%.

ACKNOWLEDGMENT

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REFERENCES

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- [2] T. Yamanishi and R. H. Sherman, et al., JAERI-M 88-254.