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JOINT OPERATION OF THE TSTA WITH THE J-FCU UNDER THE COLLABORATION BETWEEN JAERI AND U.S.-DOE

—A FIRST INTEGRATED TRITIUM TEST BETWEEN J-FCU AND ISS ON OCT. 1991—

March 1993

Takumi HAYASHI, Hirofumi NAKAMURA
Satoshi KONISHI, Toshihiko YAMANISHI
Masahiko INOUE[*1], Kazuhiro HIRATA[*2]
Kenji OKUNO, Yuji NARUSE, J.L.ANDERSON[*3]
J.W.BARNES[*3], R.H.SHERMAN[*3], D.TAYLOR[*3]
W.HARBIN[*3], J.E.NASISE[*3], R.S.WILLMS[*3]
and J.R.BARTLIT[*3]

日本原子力研究所
Japan Atomic Energy Research Institute

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Joint Operation of the TSTA with the J-FCU under the Collaboration between JAERI and U.S.-DOE - A First Integrated Tritium Test between J-FCU and ISS on Oct. 1991 -

Takumi HAYASHI, Hirofumi NAKAMURA, Satoshi KONISHI
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W.HARBIN[*3], J.E.NASISE[*3], R.S.WILLMS[*3]
and J.R.BARTLIT[*3]

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

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A first integrated loop test between the JAERI-Fuel Cleanup System (J-FCU) and the Isotope Separation System (ISS) was performed successfully with 80 grams of tritium on Oct., 1991. One of the major purposes of this test was to study interactions between the J-FCU and the ISS with single column. Another was to investigate dynamics of ISS column I using Laser-Raman spectroscopy.

Almost no problems occurred during interconnection between the J-FCU and the ISS systems and no major difficulties were encountered during this run, though Ceramic electrolysis cell of the J-FCU was not used for its R&D subject. The loop flow of 2 ∿ 10 SLPM was demonstrated. On-line gas analysis by Laser Raman spectroscopy was worked effectively to study

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*1 Mitsubishi Heavy Industries, Ltd.

*2 Sumitomo Heavy Industries, Ltd.

*3 Los Alamos National Laboratory

the steady-state and dynamic performances of the ISS.

This report describes this test plan and its detail results.

Keywords : Fusion, Fusion Fuel Cycle, Tritium, Fuel Cleanup, Isotope Separation, TSTA, Cryogenic Distillation, Palladium Diffuser, Ceramic Electrolysis Cell, J-FCU, Laser Raman Spectroscopy

日米協力Annex Ⅳに基づくTSTA共同試験（1991年10月）
－初のJ－FCUとISSの連結トリチウム試験の詳細結果－

日本原子力研究所那珂研究所核融合工学部
林　　巧・中村　博文・小西　哲之・山西　敏彦
井上　雅彦[*1]・平田　一弘[*2]・奥野　健二・成瀬　雄二
J.L.ANDERSON[*3]・J.W.BARNES[*3]・R.H.SHERMAN[*3]
D.TAYLOR[*3]・W.HARBIN[*3]・J.E.NASISE[*3]
R.S.WILLMS[*3]・J.R.BARTLIT[*3]

（1993年3月8日受理）

原研は日米核融合研究協力協定Annex Ⅳに基づき，米国ロスアラモス国立研究所のトリチウムシステム試験施設（TSTA）において核融合炉燃料ループの実証試験を行っている。本試験は，J－FCU（JAERI－燃料精製捕集装置：電解セル等の回収系を除く）とISS（深冷蒸留水素同位体分離装置：単塔運転）を連結した初の実験で，J－FCUの大量トリチウム運転の許可を取得した直後（1991年10月）に行われた。最も重要な試験目的は，1）J－FCUとISSの連結運転上の制御特性，問題点等の研究と，2）レーザーラマン分光法によるISSの動特性測定であった。

試験中，J－FCUとISSの連結上の問題はなく，不純物は添加できなかったがループの流量としては2～10SLPM程度の領域で安定に運転できた。レーザーラマン分光測定は短時間で非常に有効に作動し，ISSの動特性に関する知見が得られた。

本報告書は，上記試験における計計と詳細結果をまとめたものである。

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 住友重機械工業㈱

＊3 ロスアラモス国立研究所

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1. INTRODUCTION

In June 1987, the Japan Atomic Energy Research Institute (JAERI) and the United State Department of Energy (DOE) signed a collaborative agreement (originally 5 years), Annex IV to the Japan/U.S. Agreement on Fusion Energy, regarding development of technology for fusion-fuel processing. Under this agreement, JAERI and DOE have continued joint operations and experiments on fusion-fuel processing technology with the Tritium Systems Test Assembry (TSTA) at the Los Alamos National Laboratory (LANL)[1-2].

The TSTA [3] is a simulated fusion fuel loop that mainly consists of a torus mockup system with cryopumps (VAC), Fuel Cleanup System (FCU), Isotope Separation System (ISS), Transfer Pump Systems (TP1, TP3), Uranium Tritide Storage and supply System (UTB), and Tritium Load-in/load-out System (LIO). The TSTA has also safety systems such as Tritium Waste Treatment System (TWT), Secondary Containment System (SEC), Tritium Monitoring System (TM), Master Data Acquisition and Control System (MDAC), and Building Ventilation System (VEN).

Under the Annex IV collaboration, a JAERI Fuel Cleanup System (J-FCU) was designed, fabricated, and installed at the TSTA as a subsystem for the TSTA loop [4-6]. Generally, Fuel Cleanup System has mainly two functions that are purification of hydrogen isotopes and recovery tritium from impurities. For the purification, TSTA FCU uses cryogenic molecular sieve beds and J-FCU uses a palladium diffuser. For the tritium recovery, TSTA-FCU selects catalytic reactor - cold trap - hot metal bed chain and the J-FCU selects a catlytic reactor - cold trap - ceramic electrolysis cell chain.

After many deuterium test for a year [7], J-FCU tritium test started on March,'91 [8]. After a stand alone tritium test of the J-FCU, a first integrated loop test between the J-FCU and the TSTA ISS (Isotope Separation System) was performed on Oct.,'91. In the original test, it was planed to use the Ceramic Electrolysis Cell (CEC) of the J-FCU. However, a large internal leak of the CEC was found during its preparation. Therefore, the CEC was bypassed and no simulated impurities were added in this run.

This report describes the detail results of the above loop test of the J-FCU.

2. TEST PLAN (final)

2.1 PURPOSE

The purpose of this test plan is to describe the change of the configuration and procedure of the original test to accomodate the isolation of the CEC. Major objectives of this run are to :

1) study the interaction between the ISS and the JFCU,

2) to demonstrate the capability of the JFCU to produce a hydrogen isotope stream for feed to ISS,

3) to operate the ISS to investigate stability when interfaced with JFCU and measure the dynamic response of the ISS column, using Raman spectroscopy,

4) to accumulate TSTA loop operation experience with the JFCU.

2.2 CONFIGURATION

The test configuration with the loop of TP1-JFCU-ISS-TP3 is not changed, as shown in figure 2-1. JFCU is operated with the "Internal mode" that does not accept any external inert flow as shown in figure 2-2. No exhaust goes to Tritium Waste Treatment system (TWT) during the run except for the samples from JFCU GCs . No oxygen is added to the JCR1.

The ISS is operated with only column I cooled in order to provide relatively stable conditions for dynamic measurements of the interface between JFCU-ISS. Other H,D, and T columns will be warm and provide only flow paths for hydrogen isotopes. The Raman system in EXP2 will be used to measure the dynamic response of the ISS. The Raman flow will be established ISS-Raman- FCU - TP1-JFCU, as shown in the figure 2-3.

2.3 SUBSYSTEM REQUIRED

Safety subsystems PEV, TWT, SEC, TM, VEN, House VAC, MDAC, and UTIL should be operational. The MDAC System will archive data from the process and receive data from JFCU. Only FCU tubing and pump used in the connection between TP1B & FCU will be used as the raman gas return path.

2.4 PERSONNEL

Regular personnel assignments for the run will be arranged. Both JAERI and TSTA personnel will be needed for any major decision on the system operation. Operation of the JFCU will be performed by LANL operator(s) with the close collaboration of JFCU

-qualified JAERI staff and knowledgeable TSTA staff. The appropriate JAERI staff are Konishi, Hayashi, and Inoue. One of these three must be working with a LANL operator at the JFCU control console at all times. Any operations or system adjustments of any kind, must be communicated to this operator before being done. Operations of the ISS will be performed by operators under the supervision of knowledgeable TSTA and JAERI staff at the control room. Assignment plan (Table 2-1 & 2-2) is attached.

2.5 TIME

This change will take effect on October 8 after the approval is given. The run was changed for 4 days, around-the-clock operation including 3 night shifts. ISS will be operated as originally planned.

In the JFCU, CEC cooling will start immediately and the shift originally planned to take care of that will not be necessary in Thursday. The run will be shut down when sufficient data on the interaction between ISS and JFCU is obtained, or the accumulation of impurity in the JFCU makes JFCU to stop.

2.6 POSSIBLE HAZARDS and EMERGENCY CONCERNS

This change does not cause any safety concerns during the run. Due to the lack of capability to process tritiated impurity, there may be some tritium sent to TWT after the run.

Approximately 40 grams of tritium (36.0 g T, 101.8 g D, 2.3 g H are stored in U-bed No.4) will be used in the test. Off-normal situations in a run have already been considered in the SOP for ISS and JFCU. Interconnecting the JFCU and the ISS could lead to problem not previously experienced. Both the ISS and JFCU are independently interlocked for emergency situations. When one system alarms, the other can detect it by an MDAC alarm.

No discharge of gas from the JFCU to TWT is planned in this operation except for the exhausts from GCs. If the total tritium sent to TWT during the run reaches 1000 Ci, the run will be halted. The run will also be halted if during the run fluctuations become sufficient that the ISS safety program opens EVAC valves and dumps to UTB. If JFCU goes into PAUSE mode during the night hours, (hours usually associated with the C shift) the loop operation will not be restarted until the following morning when more personnel are again available.

TSTA will not be left unattended with LN_2, water, freon, or tritiated gas flowing into or through any glovebox.

No oxygen is added to the JFCU and thus hazard related to pure oxygen is not a concern.

2.7 OUTLINE

Preparations

CEC will be isolated and the cooling will be started.

Oxygen monitors will be isolated and all the JFCU process will be evacuated.

Permeated side of the PD - Scrol - BP - RT2 loop will be isorated.

PD-JCR1-CT-CEC(bypass)-RT1 loop will be back filled with He of 200 torr.

Run

Circulation will start.

If the pressure in the RT1 is far lower than the ISS pressure, add more He to the loop.

Column I will be stabilized with sufficient liquid in reboiler.

JFCU will be isolated and in a stand-by mode where only helium, as the carrier gas of Cold Trap (CT) regeneration, will be circulated in the JFCU. (At this time, CT regeneration mode is "Internal".

JFCU parameter setting will be checked and adjusted.

The above detail procedure will be followed by TTA-TP-118-15, Current Revision. Then,

1) **Check** that AV404 and AV406 are closed.

2) **Open** hand valves HV-A, HV-B, and HV-C (as shown in figure 2-1 and 2-2).

3) **Check** the situation of the outlet lines of ISS - TP3 - TP1 - JFCU HV-A,B,C line - feed line of ISS.

4) Circulation of ISS will be started using the HV-A,B,C line of JFCU (ISS-TP3-TP1-JFCU HV-A,B,C line - ISS).

Nominal ISS operations will be in general accordance with the following table of conditions:

Item:	Column I	
Feed Flow, sccm	6000	
Top Flow, sccm	1500	
Bottom Flow, sccm	4500	
D2 Recycle, D2IN	N/A	(closed)
Reboiler Power, watts	35	
Pressure, torr	800	

When the ISS circulation is stabilized, JFCU CT regeneration will be changed to

After checking the above JFCU situation, start the gas processing at the JFCU.

(4) **Close** HV501. Then **open** AV404. **Check** again the pressure balance between the ISS and JFCU.

(5) Slowly **open** HV501.

(6) **Close** slowly HV-B, checking JFCU system parameters.

Adjust the JFCU-RT2 pressure to supply to the ISS.

(7) **Open** AV406 and **check** the value of PRA-RT2 and PRCA-ISSIN.

(The JFCU is designed to handle 6 Nl/min of D-T mixture, however, the response to process upset conditions is not known. So, in this run, JFCU processing gas flow rate will be gradually increased to ~ 6 Nl/min.)

Flow rate of the loop will be controlled by the controller in the ISS (F-CLIA, Q-CLI, and/or F-CLTB) and JFCU processing gas flow rate will be controlled by adjusting the HV-B. Some difficulty is anticipated to balance and stabilize system pressure and flow rate. Deuterium will be added from IMS when needed. Excess hydrogen isotopes can be recovered at either UTB or ZCB1.

JFCU-GCs will be used to monitor the hydrogen concentration in the bleed.

During stable operation, it is important to maintain the ISS as stable as possible, in order to check the processing capacity of the JFCU-PD as well as to obtain good column profile measurements. After a stable profile has been obtained, the conditions in column I will be changed by readjusting the top/bottom flow ratio, and measuring the top product (AN-1) every two minutes for 2 hours with the Raman system (dynamic measurement). At that time, the effect to JFCU will be watched. If possible, after returning to the initial conditions and reaching equilibrium, a second change will be made by reducing Column I reboiler heater power to 20 watts.

For equilibrium profile measurement, a GC measurement will be made with some Raman measurement, where possible. During transient condition measurements, only Raman measurements will be made as the GC takes too long, and it will be necessary to switch sample taps frequently.

The Raman system will be operated as specified according to ISS OPERATIONS and also according to the current version of test plan TTA-TP-124-04.

ISS operation will be shutdown and hydrogen isotopes stored on the UTB's by the regular procedure. At the same time, JFCU will be isolated by AV 404 and AV406 and the JFCU shut-down procedure will be started.

Tritium in the JFCU will be recovered by the ZCB 1 according to the previous DTP (TTA-TP-118-15, Current Revision).

No impurity will be processed. Minor tritiated imputiried that will be left in the loop will be damped to TWT.

The JFCU component cooling water flow will be shut off when the CEC will be cooled. ZCB1 will be regenerated and JFCU inventory will be checked. Also, UTB's inventory will be checked.

Off-normal conditions

JFCU : Exhaust to TWT is shut and the Tritium monitor will be isolated and no major tritium discharge to TWT is anticipated. Interlocked "Pause" may occur when system is in off-normal situation.

When "pause" is initiated,

(1) **Open HV-B as soon as possible.**

(2) **Turn on** TP3, if needed.

(3) **Identify and correct** the problem that caused interlock.

Then, JFCU re-start will be proceeded as follows. Following operation will be done step by step and will not need to be hurried. (Basic JFCU re-start procedure is same of TTA-TP-118-15, Current Revision.)

(1) **Turn on** the Booster pump (BP) and Scroll pump (Scroll).

(2) **Open** HCV575 and **Turn on** the Metal Belows pump (MBP).

(3) **Adjust** HCV575, checking the pressure of PRC-JFCUEX (< 1000 torr).

(4) **Open** AV414 and AV415 and **close** HCV575.

(5) **Open** AV406 and **check** the value of PRA-RT2 and PRCA-ISSIN.

After adjusting pressures (outlet pressure of TP1 > PRA-RT1),

(6) **Close** HV501. Then **open** AV404. **Check** again the above pressures.

(7) Slowly **open** HV501.

(8) **Close** slowly HV-B, checking JFCU system parameters.

(PRA-RT1, PRA-RT2, FR-PDIN(CECEX), etc.)

ISS : If it should become necessary to perform an emergency shutdown of the ISS, the uptake of hydrogen isotopes by the uranium beds may become diffusion limited due to the buildup of ^{3}He. (Basically, initial input ^{3}He from UTB,during hydrogen isotopes loading to the ISS, will be separated continuously by Palladium Diffuser (PD) of JFCU. So, it is expected that the amount of ^{3}He in the ISS will eventual be removed.) This condition can be alleviated by circulation through the U-bed affected and TP1. Refer to the TTA-SOP-108, Current Revision (System Shut-Down (Loop)).

If plugging of the ISS feed lines occurs, the JFCU has no problem; proceed as follows.

(l) First determine the location of the plug.

(2) If it is determined that the plug can not be cleared, dump the contents of the ISS to the UTB's. (JFCU-ZCB1 may also be used to dump the hydrogen isotopes of the ISS.)

(3) Warm ISS as necessary to clear the plug. (At this time, Palladium diffuser (PD) in the JFCU may be used to clean up the plugging impurities.)

(4) Cool the ISS, reload hydrogen isotope gases (Q_2, Q=H,D,T) from the UTB's (or ZCB1); and restart ISS, TPU, IMS, and JFCU operations.

2.8 DATA

Data will be recorded in the MDAC computer archive file, JFCU computer, Raman computer, and each GC. Subsystem notebooks will be used for the formal logging of the operation in addition to the MDAC logging. All the major operations will be recorded in a notebook in control room by the loop operator.

When making a GC or Raman measurement of the ISS, the column conditions will be recorded manually on the data sheets and in the ISS LOG BOOK. Record at least:

Date and Time
Column Identification
Column Pressure
Differential Pressure
Top Flow
Feed Flow
Bottom Flow
Reboiler Power
Reboiler Liquid Level

When making a GC measurement of the JFCU, the following data will be recorded manually in the JFCU LOG BOOK.

Program timer setting information
Start time of the program timer and sampling point selection
Sampling gas pressure of each sampling point (if possible)

All data will be documented and maintained in accordance with Records Control Procedure TTA-QA-10, Current Revision.

Table 2.1 Shift Assignment

	Shift A 0730-1930	Shift C 1930-0730
Test Dir.	Anderson	Bartlit
Alt TD	Coffin/Willms	
Loop Operator	Wilhelm	Jenkins
Facility Operator	Hamerdinger	King
JFCU	JAERI*	JAERI*
ISS	Harbin/Barnes((Hamerdinger)) Sherman/Barnes((Hamerdinger))	King Bartlit
FCU(for Raman)	JAERI* Willms	JAERI*
UTB	Nasise	Jenkins
TPU	Wilhelm	Jenkins
LIO	Nasise	King
TWT	Nasise	
ETC	Wilhelm	King
IMS	Nasise	
GC(ISS,JFCU)	JAERI*/Herbin	King/JAERI*
RAMAN	Sherman/Taylor/Nakamura**	
MDAC	Cole	

Notes:
1 All others will be divided between all shifts and subject to call as needed.
2.Some personnel on Shift A & C may shift hours between shifts (with the cognizance of the TD) depending on specific activities under way.
3.Personnel in ((...)) are in training.

* See Table 2.(next page)
** One person from the Raman Trio will be in TSTA during Raman operations.

Table 2.2 JAERI Team Shift Assignment

Day		Ab (7:30-17:30)	Super B (*)	bC (21:30-7:30)
10/7	Mon	Hayashi Hirata	Konishi* Yamanishi*	Inoue Nakamura
10/8	Tue	Hayashi Hirata	Konishi* Yamanishi*	Inoue Nakamura
10/9	Wed	Hayashi Hirata	Konishi* Yamanishi*	Inoue Nakamura
10/10	Thu	Hayashi Hirata	Konishi* Yamanishi*	Inoue Nakamura
10/11	Fri	Hayashi, Hirata		

* Super B : come and join the shift change meeting at 7:30, as possible as he can.
After this meeting, frex depending on the situation of the run.
But at least the period 17:30-21:30 should be covered by super B .

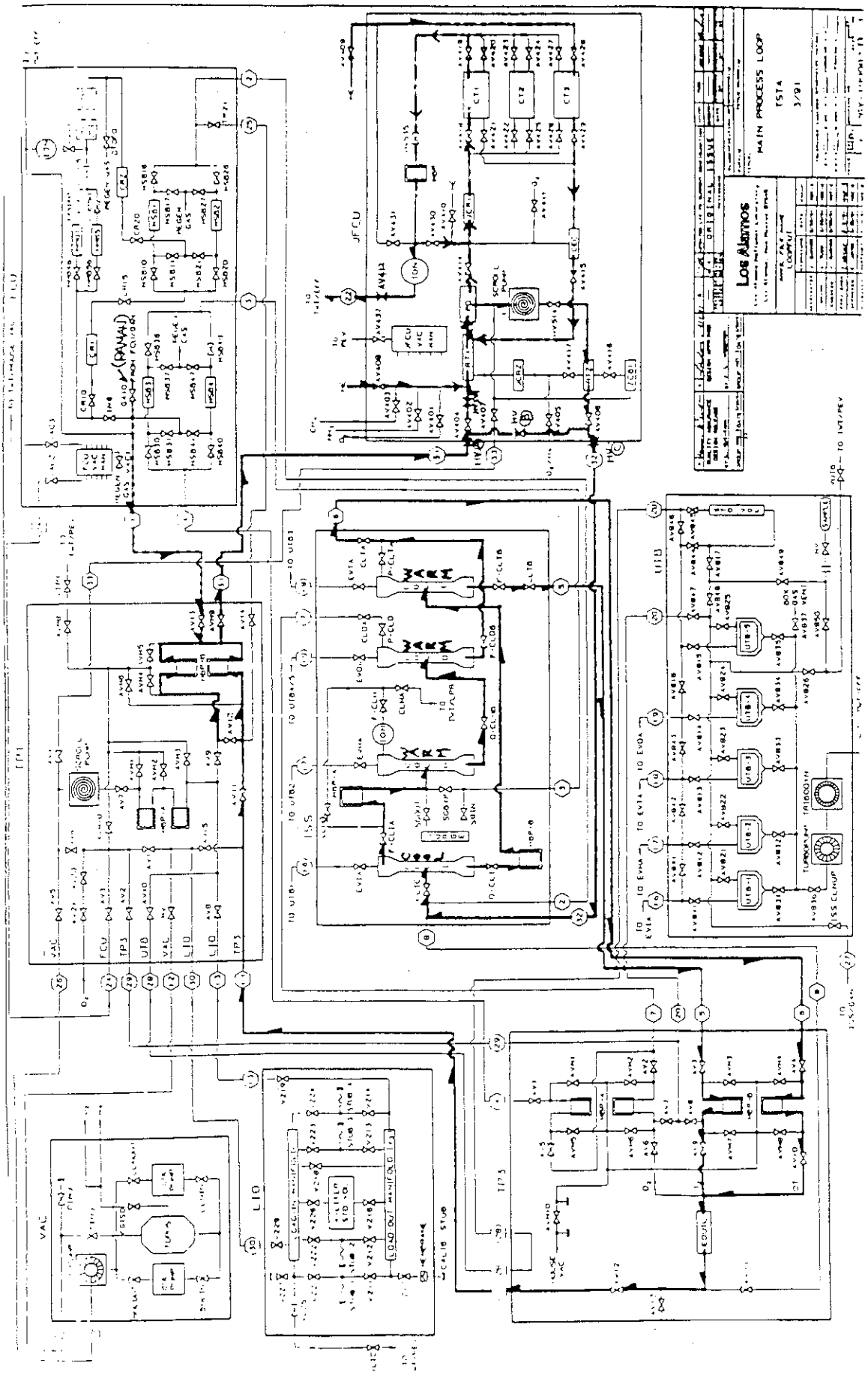


Fig. 2.1 TSTA loop flow configuration

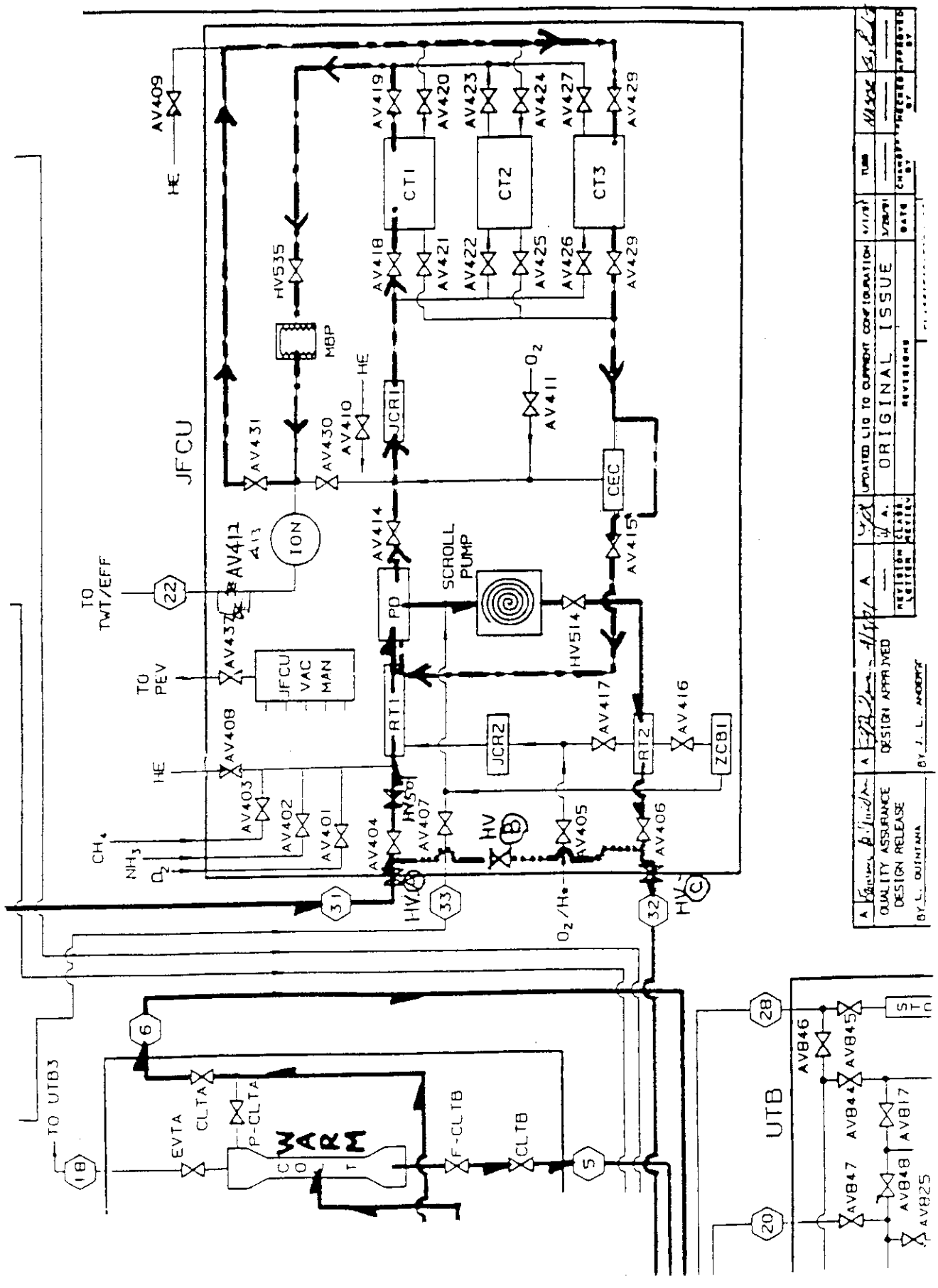


Fig. 2. 2 Detail flow configuration in the JFCU

Figure 2-3

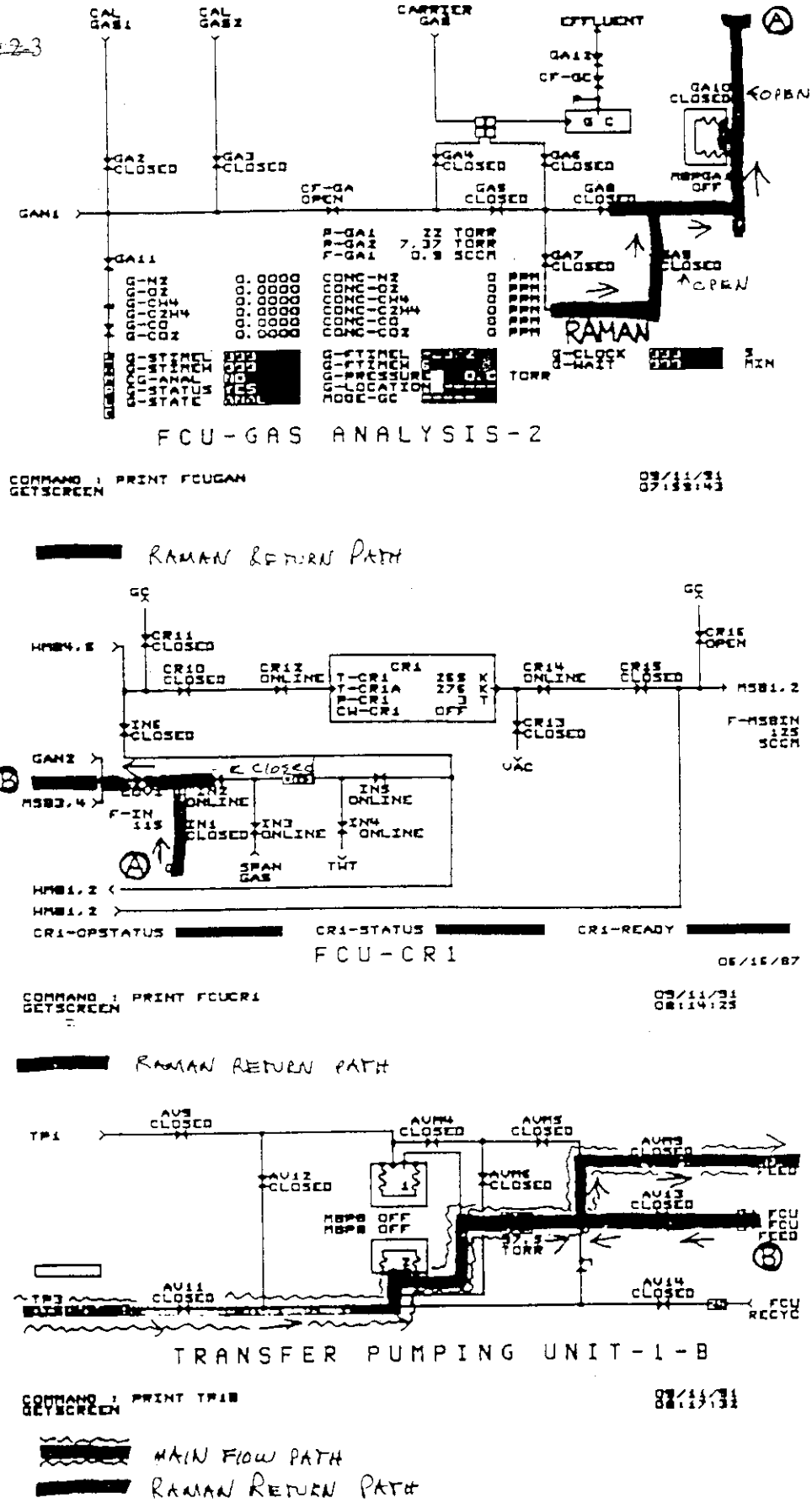


Fig. 2. 3 Detail lines for laser raman analysis

3. TEST RESULTS AND DISCUSSIONS FOR LOOP RUN 91—3 (JFCU ISS Integrated Loop Run)

3.1 SUMMARY

The JAERI Fuel Cleanup system (JFCU) and the Tritium systems Test Assembly (TSTA) Isotope Separation System (ISS) were tested as an integrated system during the week October 7-11, 1991. The test was conducted primarily

(a) to study interactions between the JFCU and the ISS with single column operation and

(b) to investigate dynamics of ISS Column I while operating in this mode using Raman spectroscopy.

3.1.1 JFCU Test Results

The first testing of the JAERI Fuel Cleanup system (JFCU) with TSTA loop was successfully performed. Although Ceramic Electrolysis Cell (CEC) was not used for its R&D subject, JFCU was operated safely without making tritiated water for two days. Impurities in feed from the ISS were stored and accumulated in the JFCU. Impurities were not oxidized in JCR1 or exhausted to the TWT during the test because of a failure in the CEC electrolysis cell. The performance of Palladium Diffuser (PD) was tested under the various loop flow conditions of 2.0 - 10.0 Nl/min.

Almost no problems occurred during interconnection of the ISS and JFCU systems. No major difficulties were encountered during this integrated run. Tritium levels of the JFCU glovebox did not increase during the run. All tritium in the JFCU was recovered in the Zirconium Cobalt Bed (ZCB1) and no major tritium loss to the TWT was detected during JFCU process evacuation at shutdown. Inventory of JFCU in this TSTA loop run was two grams of tritium (~35 % of hydrogen isotopes). That inventory under normal operation of JFCU was estimated to be 4~5 grams of tritium as the same concentration of tritium.

Operators and staff gained valuable experience during this run of JFCU in the TSTA loop Several items were developed for future improvements and testing.

3.1.2 ISS Test Results

On-line gas analysis by Laser Raman spectroscopy has been used during TSTA Run 91-3 to study the steady-state and dynamic performance of the Isotope Separation System (ISS). Raman analysis determines within seconds the molecular composition of a minuscule flow of gas from any of the several sampling ports of the four ISS cryogenic distillation columns, of which only Column I was actively cooled during Run 91-3. As Raman analysis is particularly sensitive to the nuclear spin of a homonuclear diatomic molecule, we have demonstrated that the ISS distillation columns separate not only isotopic molecules (isotopomers), but also homonuclear molecules in different nuclear spin states, called

ortho/para or even/odd modifications. Distillation column profiles have been determined by Raman analysis of every sampling port for a number of TSTA steady-state conditions during Run 91-3, such as total reflux and several flow rates to the JAERI Fuel Cleanup Unit (JFCU). Dynamic behavior has been studied by Raman analysis of a selected port during several types of transient events, such as the initial connection of JFCU to ISS, subsequent changes of JFCU flow, and changes in ISS parameters.

3.2 PURPOSE

This test was the first test of the JFCU system in the TSTA loop. The test was performed during Oct. 7 - 11, 1991. The purpose of this run was

(a) to study the flow and pressure interactions between the ISS and the JFCU,

(b) to demonstrate the capability of the JFCU to produce a hydrogen isotope stream for feed to ISS,

(c) to measure the dynamic response of ISS Column I using Raman spectroscopy,

(d) to check the effect of JFCU inventory on total loop inventory, and

(e) to accumulate TSTA loop operation experience with the JFCU.

3.3 CONFIGURATION

3.3.1 JFCU-ISS Configuration

Because the CEC could not used during the run, the JFCU was operated within the TSTA loop without making tritiated water. The ISS was operated with only a single column (Column I) in order to simplify operation of the integrated system and eliminate complicating perturbations that occur during a normal three or four column ISS test run. Figure 2-1 and 2-2 in the previous section illustrate the major flows of the TSTA Loop and JFCU under this test, respectively. Almost all systems were used in support of the test. Table 3-1 shows JFCU components and system operating parameters.

3.3.2 ISS-Raman Configuration

Laser Raman analysis in TSTA is conducted in a small-volume flow-through anodized aluminum cell with quartz windows that is secondarily contained by the EXP2 glovebox. The laser, optics (mirrors and lenses), and spectrometer are mounted on an optical table beneath the glovebox. The Raman detector, an intensified 1024-channel photo diode array attached to the spectrometer, displays the entire Raman spectrum on each readout, which can be programmed to occur at any time interval down to 0.01 sec. The Raman cell is connected by doubly contained 1/8" copper piping of 20-m length to the ISS Gas-Analysis manifold (ISS-GAN) and to the Fuel-Cleanup Unit (FCU). These connections permit gas analysis of small flows from any ISS sampling port or from the FCU, with exhaust of this flow into the FCU, as was demonstrated during TSTA Run 91-2, for analysis of hydrogen isotopomer composition or molecular impurity species. During Run 91-3 the small Raman flow was pumped by the FCU to rejoin the main flow within TP1 glovebox for return via JFCU to the ISS.

The Raman flow rate varies slightly among the ISS ports (due to different tubing sizes within ISS) and with TSTA operating conditions (such as ISS column pressure), but is typically <.02 Nl/min., or <.04% of typical ISS reflux rates of 50 Nl/min.; thus the Raman flow constitutes a negligible perturbation of ISS operation. The small Raman flow rate could limit the temporal resolution of TSTA transients by Raman analysis. We measured this limitation by recording Raman spectra once per second after changing the ISS sampling port by manually closing the hand-valve from the initial port and opening another simultaneously. These sequences of Raman spectra displayed no change for a delay time that scales inversely with the final steady-state flow rate, from 22 sec for .018-Nl/min. to 28 sec for .012-Nl/min., in agreement with similar tests conducted during TSTA Run 91-2. This flow delay limits the rapidity with which ISS sample ports can be changed while measuring profiles of the distillation columns. During Run 91-3 Raman analyses of column profiles were conducted by averaging 100 times of 1-sec exposures to achieve suitable precision; adding the time required for manual valve changes and the flow delay, we obtained each point of a column profile in 3 minutes and conducted a complete profile of ISS Column I within 20 minutes, more than ten times faster than achievable by gas chromatography. For laminar flow through the Raman sampling lines, this flow delay limits the temporal resolution of TSTA transients to about half the flow delay, or typically 15 seconds. As this is much faster than most TSTA transients, we typically studied TSTA dynamics by averaging 100 times or more 1-second exposures.

Raman analysis of relative mole-fractions of hydrogen isotopomers can be performed based upon ab-initio theoretical calculations, without needing specially prepared calibration samples. Small corrections for variations in optical efficiency and pixel response in the Raman detector were made based upon an equilibrium sample of bottled deuterium; residual uncertainties in these corrections lead to an estimate of accuracy among isotopomer densities of ~1.5%. For typical operation of the laser at 3 Watts single-pass (to avoid detector saturation for 1-sec exposures) and averaging 100 times of 1-sec exposures, the reproducibility of Raman

density measurement of a majority component in a static sample was ~0.5%, exactly as predicted based upon shot-noise (the ultimate performance set by quantum statistics), and was improved relative to a single exposure as the square-root of the number of exposures averaged. For these typical operating conditions, the Raman detectability, defined as the fractional density that yields a Raman signal with Signal-to-Noise Ratio (SNR) of unity, ranged from about 0.02% for equilibrium H_2 with little T_2 present to about 0.07% for DT.

3.4 JFCU OPERATIONS

The main operation log for the JFCU is listed in Appendix 5-1. These operations are summarized below.

3.4.1 JFCU Run Preparations

(a) Connect line between ISS and JFCU. Tritium leak check this connection.

(b) Tritium leak check of JFCU.

(c) JFCU-JCR1 inventory check by hydrogen circulation and analysis method

(d) Loop evacuation and purging TSTA loop including the lines between JFCU - ISS, TP1-JFCU, and JFCU HV-A,B,C line , as shown in fig 2-1 and 2-2, and JFCU.

(e) JFCU-GC calibration and JFCU-GC training of operators.

(f) Shielding of fixed piping heaters to protect rubber gloves.

3.4.2 Connection of ISS−JFCU

Column I of ISS operation was established in the total reflux mode. After stable operation was attained in Column I, the JFCU was isolated and operation was established in a stand-by mode where only helium, as the bleed flow of the PD and the carrier gas of Cold Trap (CT) regeneration, were circulated. CT regeneration mode was "Internal" throughout the run.

Circulation of ISS (TSTA loop circulation) was started using the HV-A,B,C line of JFCU (ISS-TP3-TP1-JFCU HV-A,B,C line - ISS). When the ISS circulation was stabilized, loop flow was introduced to JFCU gradually as shown in the Test plan. When AV405 and HV501 were opened, more than 10.0 Nl/min flowed to RT1. Loop flow was about 2.0 Nl/min at this time. It is believed that the excess flow occurred because of backflow from the ISS feed line. Input to the JFCU was purified in the Palladium Diffuser and recovered in RT2.

JFCU shutdown occurred when the pressure reached 850 torr in RT2 because AV406 opening was late. The "ISSIN Press" safety interlock initiated the shutdown. TP1 then shutdown because of a high pressure that occured when AV404 was closed during the shutdown.

The system was restarted after these problems were corrected. Flow rate of the loop was then controlled by ISS controllers F-CLIA, Q-CLI, and F-CLTB .

3.4.3 JFCU-Loop Operation

During the loop run, the performance of PD was tested by changing the loop flow rate. The loop flow rate was changed from about 2.0 Nl/min. to about 10.0 Nl/min. No problems were encountered during these changes. GC sampling analysis program was conducted during the loop run. Sampling points #2, #3, #5 were continuously and automatically analyzed. Sampling operation was stopped at 13:00 on Oct. 10.

3.4.4 Tritium Recovery and Inventory Check

After isolation between JFCU and ISS, hydrogen isotopes in RT2 were recovered by Zirconium cobalt bed (ZCB1), operating pumps continuously and recovering residual hydrogen isotopes in JFCU by PD for about two hours.
Residual gases (almost pure Helium) was evacuated to the TWT. No major amount of tritium was detected in TWT.

ZCB1 was regenerated by using JFCU-Scrol+BP pumps. Released hydrogen isotopes were transferred to LIO-PC through TP1. This isotopic component was measured by mass spectrometer.

3.4.5 ISS Operations

Test conditions for the ISS are detailed in the run Test Plan, TTA-TP-100-23, R0.

3.5 J-FCU RESULTS AND DISCUSSIONS

3.5.1 Palladium Diffuser

During this test, Palladium Diffuser (PD) was controlled 573 K and worked well. Total amount of purified hydrogen isotopes* was about 510 mol. Purification ratios** were more than 96 %, even if the loop flow about 9~10 Nl/min. Details are shown in Appendix 5-2. Loop flow rate was 1.6 ~ 9 Nl/min. (jfcu-f-vactpu) and He circulation flow rate was about 1.6 ~ 2.2 Nl/min. Therefore, identical hydrogen isotopes concentration in the feed of PD would be 50 ~ 80 %.

* : calculated by the integration of "jfcu-f-vactpu".

** : calculated from hydrogen isotope feed of JFCU ("jfcu-f-vactpu"= loop flow rate) and bleed hydrogen isotope conc. of PD measured by GC.

3.5.2 Gas Chromatographs

Sampling program and analysis program worked automatically. Several samples at the sampling points #2, #3, #5 were taken periodically. Sometimes hydrogen isotopes peak was

missed to analyze, however, this peak was recalculated manually after the run. All results of GCs are shown in Appendix 5-2.

During this test, hydrogen isotope composition changed depending on the ISS situation. So, further calculation is needed to obtain good result because the calibration of GC was done by using H_2. However, all GC data were consistent with ideal value within its measuring error and the above calibration error.

3.5.3 Zirconium Cobalt Bed

During the shutdown operation and inventory check operation, the Zirconium Cobalt Bed (ZCB1) worked well as same as the 1 gram run. All amount of hydrogen isotopes recovered safely and quickly. Almost all amount of stored hydrogen isotopes was released by regeneration of ZCB1.

3.5.4 Performance without Impurity Oxidation

The JFCU was operated safely without making tritiated water and electrolyzing water in the CEC during this run. The interaction between the ISS and the JFCU under the condition of flow and pressure fluctuations generated by the ISS was studied during this run. Because ISS operation was quite stable and TSTA loop flow was controlled, no major problem was observed with the interaction of ISS-JFCU. Impurities were not added in the TSTA loop; however, small amounts of residual TSTA loop impurities, such as tritiated methane and decay He-3, were effectively removed by the PD and accumulated in the JFCU.

The capability of the JFCU to produce a hydrogen isotope stream for feed to ISS was demonstrated up to ~9 Nl/min. (jfcu-f-vactpu) safely without making tritiated water. TSTA loop operation experience with the JFCU was accumulated.

Subjects that remain for future testing and development are

(a) CEC cell design, development and replacement .

(b) Mass flow meter and controller signals fluctuation.

(c) JMSB3 ~ 5 regeneration and increase performance.*

(d) Check O_2 concentration control by MDAC.*

(e) Check "JFCU-SAFTY" program by MDAC.*

* : These items were picked up during the 1 gram run and could not be evaluated in this run.

3.6 ISS RESULTS AND DISCUSSIONS

3.6.1 Ortho-Para Balances

While bottled deuterium presented a Raman spectrum with alternating intensities that reflect the equilibrium distribution of D_2 with 2/3 of the molecules in even-numbered rotational states, the samples flowing from the ISS distillation columns presented Raman spectra that indicated varying distributions among the D_2 rotational states. All the even-numbered rotational states were always in equilibrium with each other, while all the odd-numbered rotational states were in equilibrium with each other, both at the temperature of the Raman cell. However, the fraction of D_2 molecules in even rotational states varied. Based upon the systematic variation of the even fraction across the ISS sample ports and the observation of fractions below 2/3 as well as above 2/3, which would be expected for equilibrium at cryogenic temperatures, we determined that the ISS distillation columns are separating not only isotopomers but also ortho/para modifications of the homonuclear molecules. We have observed ortho/para separations of H_2, D_2, and T_2 to varying extents in all the active distillation columns in both Runs 91-3 and 91-2.

Ortho/para modifications of homonuclear diatomic molecules (H_2, D_2, and T_2, but not HD, HT, or DT) are caused by nuclear spin statistics. For protons and tritons, which have nuclear spin I=1/2, the formation of a homonuclear molecule can produce a total nuclear spin T of 0 (spins antiparallel, degeneracy 1) or 1 (spins parallel, degeneracy 3). For deuterons, which have I=1, the formation of D_2 can result in T=0, 1, or 2, with degeneracies of 1, 3, and 5, respectively. Because of quantum statistics, homonuclear hydrogen molecules with even-T can exist only in even-numbered rotational states, while those with odd-T can exist only in odd-numbered rotational states. For practical purposes, interconversion can occur only by processes that break and reform the molecules, such as catalysis. Liquefaction also does not offer rapid interconversion, only about 1%/hr for H_2 and much slower for D_2. Thus in many situations the even and odd modifications must be treated as separate species, with different heat capacities (first experimental proof), boiling points, and Raman spectra. The designation "ortho" refers to the modification that is most plentiful in Room-Temperature Equilibrium (RTE), odd for H_2 and T_2, even for D_2; however, as this designation does not describe the physical/chemical behaviors of these modifications, we prefer to designate them as "even" and "odd", for example, even-H_2 (e-H_2). In equilibrium at liquid-hydrogen temperatures, all the homonuclear molecules are in the even modifications with no rotation (J=0); however, total equilibration is not generally achieved in the relatively short residence times in the ISS distillation columns.

The ISS distillation columns separate even and odd modifications of H_2, D_2, and T_2 because of their slightly different boiling points: 20.27 K for e-H_2, 20.45 K for o-H_2, 23.52 K for e-D_2, and 23.66 K for o-D_2. The more volatile even modification tends to be more concentrated at the upper end of the isotopomer's range in the distillation column. Gas flowing

from a column sample port to the Raman cell has scant opportunity for even/odd interconversion, while warming up to room temperature. Rotational Raman analysis, with its well-resolved rotational lines, is well-suited for the measurement of even/odd fractions from the ISS distillation columns, for which other diagnostic techniques are incapable (mass spectrometry) or poorly suited (gas chromatography, which requires catalytic surfaces for analysis of H-D-T systems). The ability of the ISS distillation columns to separate even/odd modifications does complicate Raman analyses for molecular fractions, as 9 species must be treated instead of 6 in an H-D-T system.

The separation of even/odd modifications by the ISS distillation columns is most dramatically seen in D_2, which is relatively abundant at most of the sample ports of all the active columns. The e-D_2 fraction generally followed a monotonic profile across each active column, ranging from as high as 0.88 near the top, to RTE (0.67) at the feed (all the active columns in Runs 91-3 and 91-2 have room-temperature equilibrators in their feed lines), to as low as 0.40 at the bottom. The e-D_2 fraction profiles varied as TSTA operating conditions changed. For example, increasing the flow out of the top or bottom of an active column drove the e-D_2 fraction at that location closer to the RTE value. Decreasing the reboiler power drove the e-D_2 fraction at the bottom closer to RTE while leaving the e-D_2 fraction at the top unaffected. Some observed changes in e-D_2 fraction were not so easily related to TSTA operating conditions, such as occasional drifts over an hour or more in the e-D_2 fraction at I-Top while no other obvious changes were occurring in TSTA. The deviations in e-D_2 fraction from its RTE value are expected to be largest when the ISS distillation columns are in total reflux; this prediction was verified for Column I in Run 91-2, yet in Run 91-3 the e-D_2 fraction remained at RTE at I-Top (while significantly higher at the next lower sample port) during total reflux and increased above RTE only as flow to JFCU commenced.

Similar monotonic profiles were observed for e-H_2 and e-T_2 profiles in the active distillation columns. As expected, these fractions measured in the feed lines were 0.25 (RTE). The highest e-H_2 fraction observed at I-Top was 0.36, which was surprisingly measured following an increase in I-Top flow rate during Run 91-3. The highest e-H_2 fraction observed at H-Top during Run 91-2 was 0.53, which was followed by a slow drift over the next hour to 0.36 with no known cause. The e-T_2 fraction profiles in Column I show their highest values (up to 0.34) at the sample port just below the feed and progressively lower values at lower sample ports. No e-H_2 or e-T_2 fractions below 0.25 were ever measured.

3.6.2 Column I Profiles

Composition profiles of ISS Column-I were made by Raman analysis at numerous periods of TSTA steady-state operation; these profiles are tabulated in Appendix 5-3. The first two profiles were measured while Column-I was in total reflux. For all later profiles, TSTA operation was known to be in steady-state, because the profiles were measured following the conclusion of a transient that was monitored by Raman analysis of a selected port, as will be

discussed in the next section. A typical composition profile is shown graphically in Figure 3-1 for R913026 of 10/8/91 at 20:33, following the transients of connecting the JFCU. As Column-I is designed to be the ISS input column, the more volatile isotopomers (H_2, HD, and HT) are concentrated toward the top, the less volatile (DT and T_2) toward the bottom, and D_2 is most abundant near the center of the column. These general trends are obvious in Figure 3-1. The sample port at Plate 29 is actually a tee in the column's feed line (rather than a port in the column opposite the feed port); therefore its composition may be expected to differ from an interpolated composition within the column, at least for moderate-to-high reflux ratios. Thus, every increase in JFCU flow rate (R913028 -> R913031, R913037 -> R913039), which reduces the reflux ratio, reduced the deviation of the feed composition from the interpolated composition within the column. This sample port location also makes the composition measurement at Plate 29 ambiguous when Column-I is in total reflux, as the Raman flow path is completed outside the distillation column, while being open to the column, so that the Raman measurement depends upon previous history of the flow path and fluctuates upon mixing with column contents, with time constants of about 1 minute, as seen following the valve-switching transient in R913023. This ambiguity explains the differences among Plate-29 measurements in the first two profiles in total reflux and explains their deviations from the interpolated composition.

Increasing the helium-refrigerator temperature while Column-I is in total reflux increases temperatures across the entire distillation column, thereby reducing separation factors, and should thus flatten out the isotopomer profiles. After the temperature setting was increased from 18.3 K to 19.3 K, the pressure at the top of Column I increased from 609 Torr to 753 Torr, and the isotopomer composition in the column changed as shown by comparison of the first (R913015) and second (R913016) profiles in Appendix 5-3. The reduced H_2 fraction and the increased D_2 fraction at the top are the strongest evidence of the expected profile flattening, as is the reduction of DT at the bottom. However, the T_2 profile actually became sharper at the bottom, while the D_2 profile below the top did not become flatter. The even/odd modifications drew closer to RTE values, since the gas, although not flowing, was in contact with the equilibrator in the feed line.

Changes in the steady-state composition profiles of the distillation column as the flow to JFCU commenced and was increased in a series of steps are reflected in Appendix 5-3, although interspersed among the JFCU flow changes are several other changes in TSTA conditions, which somewhat complicate the interpretation of flow dependence. The most dramatic changes in column profile were observed in the composition at I-Top, which is displayed in Figure 3-2 with supplemental data from end-points of Raman dynamics monitoring. Increases in JFCU flow affect the column profile in several ways: reduction in reflux ratio (flattening the profile), increase in temperature at the top of the column (flattening the profile), and effects due to any other parameters adjusted by the operator. For example, during the flow increase to 6.3 Nl/min., the operator reduced the helium-refrigerator

temperature setting to maintain constant pressure at the top of the column (sharpening the profile). Except for the first step from no flow (total reflux), the net effect of the JFCU flow increases in Run 91-3 is an overall flattening of the profile, as evidenced particularly by the increasing D_2 and decreasing HD fractions at the top (Figure 3-2). The initial JFCU connection was accompanied by severe flow and temperature transients and adjustments, resulting in lower temperatures throughout the column but a net increase in temperature gradient to cause the profile sharpening at the top of the column that is seen in the first step of Figure 3-2. However, during this first step the composition at the bottom of the column appeared flatter, with lower T_2 and higher D_2. With increasing JFCU flow the bottom composition showed little change.

Decreasing the reboiler power reduces the reflux ratio (flattening the profile), while helium-refrigerator adjustments made to maintain constant pressure at the top of the column increases column temperatures (flattening the profile). The major composition change observed upon decreasing the reboiler from 34 to 20 W (R913026 -> R913028 in Table II) was a profile flattening near the bottom of the column, with lower T_2 and DT and higher D_2. Very little change was observed near the top of the column. The increase in reboiler from 20 back to 34 W (R913031 -> R913033) sharpened the profile near the bottom with little effect near the top.

Increasing the ratio of top flow to bottom flow from Column I while maintaining constant feed flow tips the composition profile, so that the top deviates further from its total reflux composition while the bottom grows closer to its total reflux composition. Most of these predictions were fulfilled when we increased T:B to 1:1 from 1:3 (R913033 -> R913035): at the top HD decreased while D_2 increased; at the bottom DT increased and D_2 decreased, but T_2 decreased slightly. The original profile was restored when we decreased T:B back to 1:3 (R913035 -> R913037).

During true steady-state operation, each active distillation column must satisfy conservation of each isotopomer: the total inflow must equal the total outflow of the i-th isotopomer, or

$$f_F\, x_F(i) = f_T\, x_T(i) + f_B\, x_B(i), \qquad (1)$$

where f is the flow velocity and x(i) is the mole-fraction at Feed, Top, or Bottom. An effective outlet composition can be defined as the right-hand side of Eq. 1 divided by $f_F = f_T + f_B$; in steady-state this effective outlet composition must equal the inlet composition. Based upon the flow velocities recorded by MDAC during each Raman profile, which are recorded in Appendix 5-3 as mean and standard deviation, the effective outlet composition is listed in Appendix 5-3 for each Raman profile (except in total reflux), as is its ratio to the inlet composition. This ratio is generally equal to the ideal steady-state value of 1.0 within the experimental uncertainties (flow velocity standard deviations plus Raman reproducibilities).

The cases that differ from 1.0 by more than 15% are all minority species in at least two of the sample ports involved. That the compositions based upon Raman analysis satisfy isotopomer conservation proves that no significant exchange occurs during the flow to the Raman cell, which would compromise the usefulness of Raman analysis. Also displayed in Appendix 5-3 are the atomic fractions (H, D, and T) in the inlet composition and effective outlet composition. The identity of these compositions is also a criterion for steady-state, which is essentially verified, but is a less stringent criterion than isotopomer conservation for the validity of Raman analysis. However, the HDT compositions in Appendix 5-3 show significant variations among different TSTA operating conditions; this variation implies a complementary variation in the composition of the liquid within the distillation column.

3.6.3 Column I Dynamics

TSTA dynamic behavior in approaching each of the steady-state conditions was monitored by Raman analysis of composition of a selected port of Column I, the only active distillation column during Run 91-3. The temporal resolution for observing TSTA dynamics was chosen for each sequence, based upon anticipated transient speeds and desired observation duration, ranging from 1 to 3 minutes. Each Raman spectrum in a sequence is a corresponding average of 60 to 180 1-second exposures.

The most dramatic dynamic behavior seen during Run 91-3 occurred during the initial connection of JFCU, which was monitored by Raman analysis at ISS Column-I feed port, as shown in Figure 3-3, covering 18:12 - 20:13 on 10/8/91 with 1-minute resolution. Prior to the period of Figure 3-3, an ISS flow of about 2 Nl/min. had been started at 16:39, which was conducted through a JFCU internal bypass. This flow established a composition profile within Column I that was qualitatively similar to that shown in Figure 3-1, including the notable feature that the feed composition was quite different from the interpolated column composition at the feed location. At the time indicated in Figure 3-3 by "ON", the JFCU pump was started, which resulted in a reversal of flow at the ISS feed port, recorded by MDAC as -10.24 Nl/min. and lasting for 4.1 minutes before the JFCU pump tripped "OFF". The composition of this reversed flow was the interpolated column composition, which was substantially richer in D_2 and leaner in every other isotopomer compared to the original feed composition. The original feed composition was restored for 3.7 minutes after the JFCU pump tripped. Subsequent attempts to establish JFCU connection resulted in less flow-reversal, until a successful start 18.2 minutes after the initial start resulted again in a strong flow-reversal and an accompanying transient spike of high D_2 in Figure 3-3 lasting 4.1 minutes. Following this transient spike the composition of the ISS feed port gradually evolved with a time-constant (defined as the time to achieve 63% of the change) of 18.8 minutes to a steady-state composition that was essentially identical to the initial feed composition, as expected. Only HD showed an overshoot at the conclusion of the transient spike.

The dynamics of subsequent increases in JFCU flow were monitored by Raman analysis of the composition of I-Top with 2-minute resolution. In each case a smooth transition was observed to the new steady-state profile, with no delay. The observed time constants for these transitions generally decrease for higher flow rates, although the smaller composition changes at the higher flow rates reduce the accuracies of these time constants. Slight overshoots in several species, including D_2, HD, and HT, were observed in several of these transients; these overshoots were not as strong as the HD overshoot seen in Figure 3-3, but required about two time-constants for correction.

The dynamic behavior following the reduction in reboiler power was monitored by Raman analysis of I-Bottom with 2-minute resolution. This composition remained essentially constant for the first 6 minutes after the change, then underwent a smooth transition with no overshoot to the new steady state with a time constant of 8.2 minutes. The dynamic behavior following the restoration of the original reboiler power was monitored by Raman analysis of I-Top, which unfortunately displayed no significant change in composition.

The dynamic behavior following the increase in Top:Bottom flow ratio to 1:1 was monitored by Raman analysis of I-Top with 2-minute resolution, as shown in Figure 3-4. The majority constituents shifted dramatically during the first 12 minutes toward higher D_2 and lower HD, then shifted by a much smaller
amount over the next 40 minutes, with D_2 continuing to increase but HD rebounding slightly. The minority species HT and DT displayed an additional fast response in the first few minutes (HT rising, DT declining), then somewhat followed the dynamics of the majority species to lower levels of each. The dynamic behavior following the restoration of the original Top:Bottom flow ratio of 1:3 was also monitored by Raman analysis of I-Top (Figure 3-4), and showed virtually a mirror image of the above dynamics.

Raman analysis of TSTA dynamic behavior gives additional insight into the overall system stability when operating with nominally constant control parameters. In most of the nominally steady-state periods during Run 91-3, the composition as measured by Raman analysis with 1- or 2-minute resolution was just as steady as static samples, limited by quantum statistics. By coincidence, Raman monitoring of the dynamic behavior during the JFCU flow increase to 8 Nl/min. overlapped the accidental TSTA liquid-nitrogen pressure loss about 9:00 on 10/10/91, which produced ripples of about 3% in the mole-fractions of majority components at the top of Column I. Of course, the source of this instability in TSTA operation is understood. In contrast, while monitoring the composition of I-Top overnight during Run 91-2 (8/22-23) after an intentional change, two episodes of appearance of HD and HT (previously undetectable) at about 2% levels with time constants of about 30 minutes occurred with no apparent causes. Similarly drifts in even/odd modification ratio over hours (such as the drift in e-D_2 from RTE to 78% and back to RTE during the Run 91-2 overnight vigil) occurred on several occasions during both runs with no apparent causes.

3.7 TRITIUM INVENTORY

The effect of JFCU inventory on total TSTA loop inventory was checked. The tritium inventory of JFCU in this loop run was about two grams by mass analysis. (T concentration in the Hydrogen isotopes was about 34 %). The JFCU did not make water during this run. The design value for water processing water is about 0.78 mol/hour at the Cold Traps. If water inventory is 1.0 mol and if T concentration is 34 %, this tritium amount is about two grams. Tritium inventory of other JFCU components, such as adsorbed in JCR1, will be one order of magnitude less than the above value (~ 0.2 ~ 0.4 gram order). Therefore, tritium inventory of JFCU under normal operation will be evaluated to about four to five grams when T isotope concentration of the processed fluid is about 34 %.

Pre- and post-run tritium inventrories in the Loop were measured at 79.02 and 76.18 grams respectively. The observed difference is within the normal measurement error.

Table 3.1 JFCU Operating Parameters

Palladium Diffuser	573 K
Catalytic Reactor No. 1	773 K
Cold Traps	160 K (trapping) 343 K (regenerating)
Ceramic Electrolysis Cell	Did not use
Zirconium Cobalt Bed No. 1	723 K (at regenerating)
CT Regeneration Mode	"Internal"
JCR1 Dilution Mode	"Internal"
PD Bleed Control Mode	"Pressure"

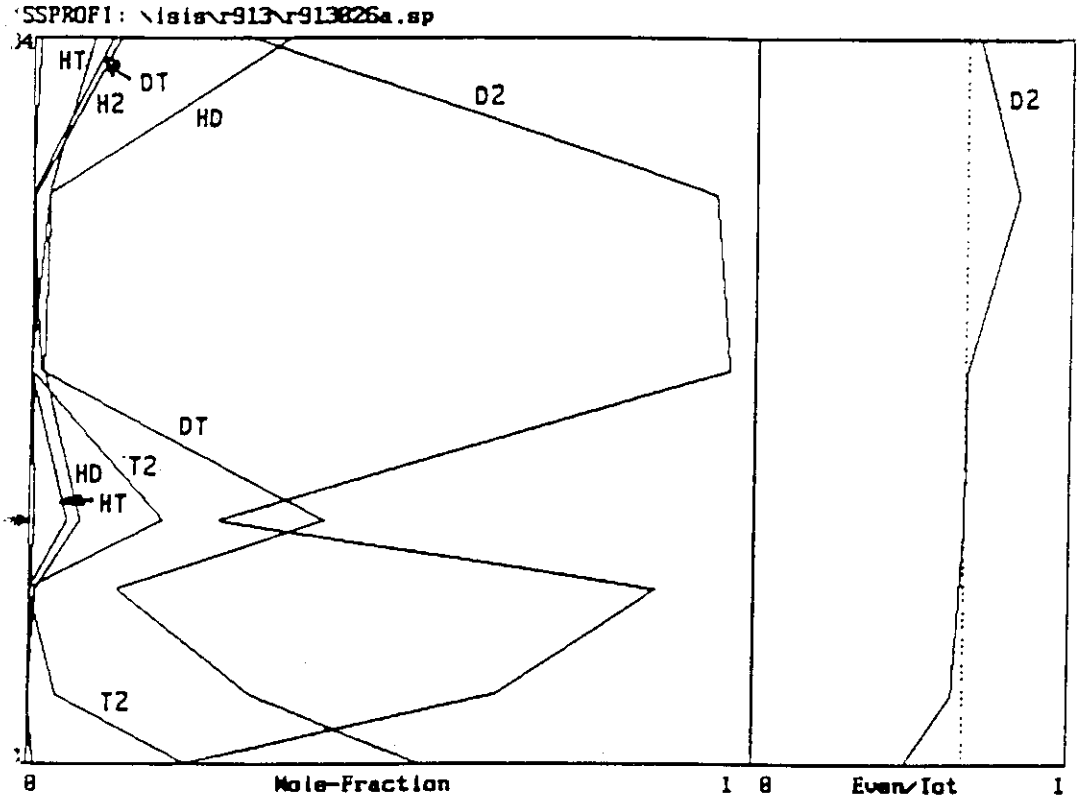


Fig. 3. 1 Composition and even/total profiles of ISS Column-I following JFCU connection.

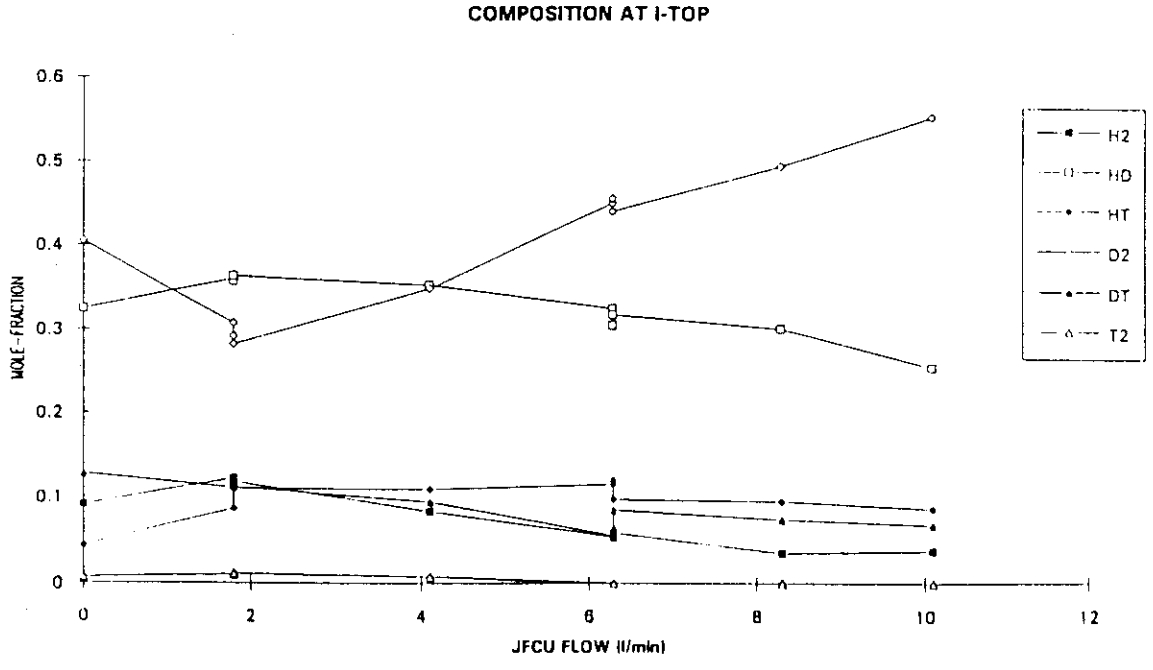


Fig. 3. 2 Steady-state compositions at top of ISS Column-I at different JFCU flow rates. (Lines are drawn between data points in chronological sequence to show additional ISS changes at 1.8 and 6.3 NL/min.)

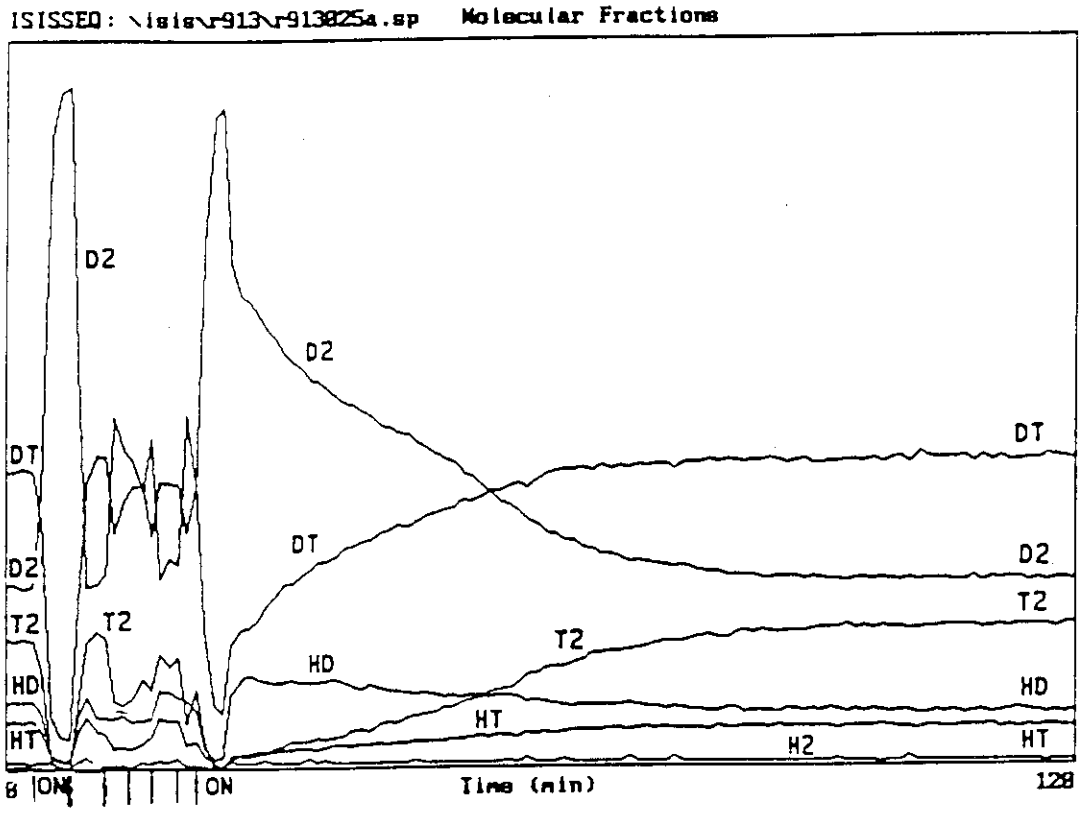
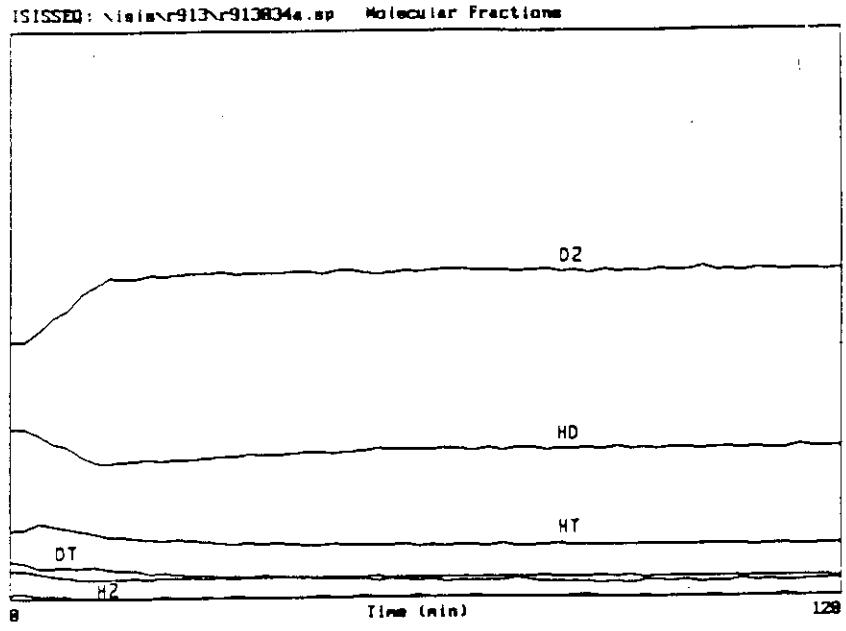


Fig. 3. 3 Dynamic behavior of composition at ISS feed during connection of JFCU.



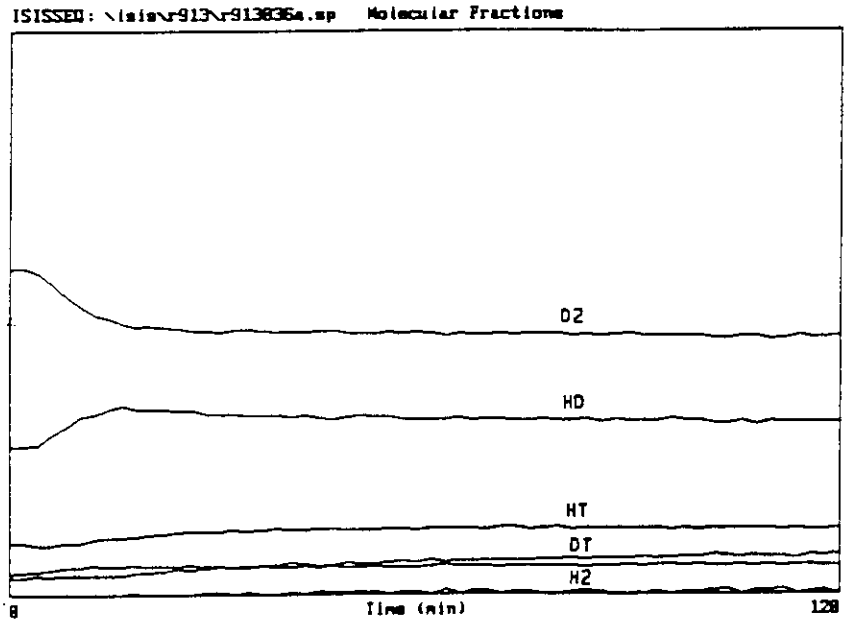


Fig. 3.4 Dynamic behavior of composition at top of ISS Column－I following: (Top) Increase in Top: Bottom flow ratio to 1 : 1; (Bottom) Restoration of Top: Bottom flow ratio to 1 : 3.

4. CONCLUSION

While the viability of Raman analyses in TSTA was demonstrated in Run 91-2 and further validated in Run 91-3, the application of Raman analysis to ISS composition measurements during Run 91-3 has provided valuable data regarding dynamic performance and steady-state operations that shed new understanding on ISS performance and interactions with other TSTA subsystems, such as JFCU. In many instances (such as separation of even/odd modifications and JFCU connection transient compositions), these data could not have been obtained by any other instrumentation, while in other instances (such as column profiles following changes in control parameters) the time required would have been prohibitive. While we have attempted above to explain most of the observed phenomena, several mysteries and alternative explanations offer prospects for further fruitful research using the Raman analysis capability at TSTA. Obvious extensions of Raman analysis at TSTA include validation of diagnostics in other TSTA subsystems (as we have demonstrated for JFCU CEC gas analysis during Run 92-1), analysis of impurities in FCU and JFCU streams, and integration with MDAC as part of a real-time feedback control system for ISS. Desirable system improvements include computer control of the ISS sample-port valves and improved Raman sensitivity. Future data analysis includes comparisons with steady-state and dynamic models of distillation columns.

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APPENDIXES

1. JFCU Operation log

Day / Time		Explanation
Oct. 7,	10:00	- Checked heating pattern of CEC and water flow of CEC.
	11:00	- Components heating started.
	13:00	- REF "on" and CT cycle operation start.
	17:00	- Flow meter/controllers zero cal. and Pressure sensors zero cal.
		- O2 monitors cal.
	21:00	- JCR1 temp. reading correction.
	23:00	- Valve position checked.
Oct. 8,	02:00	- CT2 inlet heater power adjustment
	07:00	- Leak across CEC tubes found.
		- Re-discussion of run configuration and schedule
		- Working instruction making
		- CEC cool down started
	13:00	- JFCU process purging by He
	15:00	- RT2 evacuated. Made JFCU stand-by situation.
	16:00	- Started the connection between ISS and JFCU
		- Loop flow : ~ 1.6 NNl/min. (jfcu-f-vactpu).
		- He circulation of JFCU : ~ 1.2~1.6 NNl/min.
	23:00	- GC sampling started
Oct. 9,	09:00	- Changed Loop flow : ~ 3.6 NNl/min. (jfcu-f-vactpu).
	10:00	- Changed He circulation of JFCU : ~ 2 NNl/min.
	11:00	- Changed Loop flow : ~ 5.6 ~ 6 NNl/min. (jfcu-f-vactpu).
Oct. 10,	08:00	- Changed Loop flow : ~ 7.5 NNl/min. (jfcu-f-vactpu).
	09:00	- Loss of facility N2 pressure.
		- Caused sudden decrease of loop flow
	11:00	- Changed Loop flow : ~ 9 NNl/min. (jfcu-f-vactpu).
	13:00	- Recovered hydrogen isotopes in RT2 to ZCB1.
		- Stopped CT cycle operation.
	15:00	- Evacuated residual process gas to TWT
		- All pumps "off" and closed all boundary valves
		- Started cooling down of components (PD, JCR1)
		- CEC cooling water flow stopped
Oct. 11,	10:00	- Differential pressure of CEC tubes checked again.
		- Found again leak of CEC tubes.

2. GC Analysis Results

Time	10/9,09:41	10:59	12.25	13.43	15.01
Sampling # and Species					
#2 Q2	65.3 (69)	65.5 (64)	69.1 (74)	71.1 (74)	70.4 (74)
#3 Q2	3.6	5.1	6.6	7.4	7.4
#5 Ion ch.	---	---	---	---	---
Loop flow	3.6	3.6	5.6	5.6	5.6
Bleed flow	1.6	2	2	2	2
Purified %	98.4	97.2	97.6	97.3	97.3

Time	10/9,16:41	18:00	19:18	21:08	22:48
Sampling # and Species					
#2 Q2	71.4 (74)	70.7 (74)	72.5 (74)	71.7 (74)	71.9 (75)
#3 Q2	7.4	7.4	7.2	6.9	7.2
#5 Ion ch.	---	---	---	---	---
Loop flow	5.6	5.6	5.6	5.6	6
Bleed flow	2	2	2	2	2
Purified %	97.3	97.3	97.4	97.5	97.6

Time		10/10,00:48	02:36	05.33	07:12	08:40
Sampling # and Species						
#2	Q2	70.2 (74)	72.7 (74)	67.8 (74)	67.4 (74)	69.5 (78)
#3	Q2	7.4	7.3	7.6	7.3	8.5
#5	Ion ch.	---	---	---	---	---
Loop flow		5.8	5.8	5.8	5.8	7.6
Bleed flow		1.6	2	2	2	2.2
Purified %		97.4	97.5	97.4	97.5	97.5

Time		10/10,10:58	12:00
Sampling # and Species			
#2	Q2	73.4 (76)	74.3 (79)
#3	Q2	9.7	12.2
#5	Ion ch.	---	---
Loop flow		7.4	9
Bleed flow		2.4	2.6
Purified %		96.9	96.5

3. Laser－raman analysis data

Column I Composition Profiles - Run 91-3

File	Date	Time	Plate	H2	HD	HT	D2	DT	T2	H	D	T
R913015	10/8/91	9:33	84	.1278	.3285	.0602	.3225	.1412	.0199			
Total reflux			66	.0021	.0105	.0120	.9621	.0133	.0000			
			46	.0020	.0076	.0000	.9782	.0122	.0000			
			29	.0015	.0223	.0058	.6147	.3155	.0402			
			21	.0016	.0075	.0000	.9250	.0609	.0051			
			9	.0020	.0053	.0000	.6129	.3342	.0457			
			1	.0032	.0043	.0000	.1559	.5844	.2522			
R913016		11:52	84	.0914	.3250	.0436	.4048	.1282	.0071			
Refrigerator increased			66	.0034	.0164	.0130	.9673	.0000	.0000			
			46	.0021	.0076	.0000	.9903	.0000	.0000			
			29	.0807	.3216	.0461	.4124	.0133	.0067			
			21	.0055	.0118	.0000	.9143	.0685	.0000			
			9	.0018	.0064	.0000	.5560	.3728	.0630			
			1	.0000	.0085	.0000	.1674	.5285	.2956			
R913026		20:33	84	.1230	.3611	.0866	.3079	.1112	.0103			
JFCU connected			66	.0054	.0258	.0237	.9450	.0000	.0000			
			46	.0019	.0182	.0000	.9659	.0141	.0000			
		Inlet	29	.0071	.0726	.0531	.2659	.4137	.1876	.0699	.5091	.4210
			21	.0025	.0089	.0000	.8629	.1258	.0000			
Top Flow	350.9	23.4	9	.0025	.0000	.0000	.6467	.3114	.0395			
Bottom Flow	1383.8	31.9	1	.0000	.0081	.0000	.2201	.5467	.2251			
			Outlet	.0249	.0795	.0175	.2379	.4586	.1817	.0734	.5069	.4197
			Out/In	3.5239	1.0955	.3295	.8944	1.1085	.9684			
R913028	10/9/91	8:17	84	.1169	.3582	.1095	.2921	.1117	.0116			
Reboiler decreased			66	.0024	.0179	.0225	.9428	.0144	.0000			
			46	.0037	.0233	.0000	.9532	.0199	.0000			
		Inlet	29	.0062	.0785	.0482	.2948	.4217	.1506	.0696	.5448	.3856
			21	.0014	.0082	.0000	.8425	.1479	.0000			
Top Flow	343.4	22	9	.0019	.0000	.0000	.7321	.2398	.0261			
Bottom Flow	1390.3	56.4	1	.0040	.0000	.0000	.2757	.5177	.2026			
			Outlet	.0263	.0709	.0217	.2790	.4373	.1648	.0726	.5331	.3943
			Out/In	4.2183	.9041	.4503	.9464	1.0370	1.0937			
R913031		14:00	84	.0538	.3257	.1158	.4498	.0549	.0000			
JFCU to 6.3			66	.0090	.0395	.0312	.9077	.0127	.0000			
			46	.0019	.0445	.0000	.9139	.0397	.0000			
		Inlet	29	.0055	.0771	.0441	.3278	.4067	.1388	.0661	.5697	.3642
			21	.0021	.0123	.0000	.6453	.2990	.0413			
Top Flow	1493.1	12.9	9	.0000	.0000	.0000	.5669	.3701	.0631			
Bottom Flow	4721	32.9	1	.0000	.0056	.0000	.2703	.5403	.1838			
			Outlet	.0129	.0825	.0278	.3135	.4236	.1396	.0681	.5665	.3654
			Out/In	2.3316	1.0700	.6312	.9563	1.0417	1.0061			
R913033		16:37	84	.0524	.3055	.1219	.4559	.0643	.0000			
Reboiler increased			66	.0038	.0384	.0308	.9270	.0000	.0000			
			46	.0015	.0410	.0000	.9148	.0427	.0000			
		Inlet	29	.0112	.0680	.0465	.2820	.4254	.1668	.0685	.5287	.4028
			21	.0000	.0089	.0000	.6281	.3210	.0421			
Top Flow	1489.8	13.6	9	.0025	.0000	.0000	.5391	.3895	.0690			
Bottom Flow	4772	23.6	1	.0000	.0000	.0000	.2250	.5469	.2281			
			Outlet	.0125	.0727	.0290	.2799	.4321	.1738	.0633	.5323	.4044
			Out/In	1.1096	1.0689	.6234	.9925	1.0157	1.0423			
R913035		22:09	84	.0407	.2617	.0903	.5805	.0268	.0000			
T:B=1:1			66	.0098	.0385	.0290	.9228	.0000	.0000			
			46	.0025	.0516	.0000	.8933	.0527	.0000			
		Inlet	29	.0160	.1195	.0532	.3506	.3665	.0942	.1024	.5936	.3041
			21	.0023	.0000	.0000	.6225	.3302	.0450			
Top Flow	3010.5	8.1	9	.0023	.0070	.0000	.4095	.4858	.0954			
Bottom Flow	3304	35.2	1	.0000	.0076	.0000	.1380	.6596	.1947			
			Outlet	.0194	.1287	.0431	.3490	.3579	.1019	.1053	.5923	.3024
			Out/In	1.2106	1.0771	.8096	.9955	.9766	1.0812			
R913037	10/10/91	7:48	84	.0585	.3182	.0979	.4400	.0854	.0000			
T:B=1:3			66	.0032	.0340	.0331	.9166	.0130	.0000			
			46	.0028	.0399	.0084	.9054	.0436	.0000			
		Inlet	29	.0108	.0730	.0463	.2842	.4209	.1649	.0704	.5311	.3985
			21	.0062	.0116	.0000	.6316	.3101	.0405			
Top Flow	1489	10.3	9	.0000	.0000	.0000	.5448	.3883	.0669			
Bottom Flow	4851	28.6	1	.0000	.0048	.0037	.2250	.5466	.2199			
			Outlet	.0137	.0784	.0258	.2755	.4383	.1683	.0659	.5338	.4003
			Out/In	1.2783	1.0747	.5576	.9694	1.0414	1.0201			
R913039		10:30	84	.0480	.2973	.0941	.4878	.0729	.0000			
JFCU to 8			66	.0076	.0402	.0303	.9095	.0123	.0000			
			46	.0015	.0413	.0000	.9063	.0509	.0000			
		Inlet	29	.0048	.0648	.0429	.3003	.4189	.1683	.0586	.5422	.3992
			21	.0042	.0107	.0000	.6035	.3304	.0512			
Top Flow	2000	7.1	9	.0000	.0000	.0000	.5096	.4077	.0827			
Bottom Flow	6104	38.8	1	.0000	.0000	.0000	.2302	.5432	.2266			
			Outlet	.0118	.0734	.0232	.2938	.4272	.1706	.0601	.5440	.3958
			Out/In	2.4809	1.1322	.5406	.9783	1.0196	1.0142			

4. JFCU Inventory Check Result

Mass Spectrometer Analysis - EXTENDED AMMONIA
Revision of May 21, 1990
Sensitivities Revised 10/30/1991
Oct 30, 1991 13:04

10/29/91 PC1050/JFCU 1530

70 V Electrons — P,T,V= 383.5 t= 20.4 C — 49.2 Liters

		V Obs, %	RANGE	Corr f/ He-3	Sens torr/V	Corr (Calc)		mol %	HDT fr of tot	HDT gas %	
H2	2.01565	6.104	10	.610	.9285	.399	H2	2.904	15.203	15.755	H
He-3	3.01603	.644	10	.053	2.2735	.070	He-3	.506	48.025	49.768	D
HD	3.02193	34.658	10	3.466	.9310	1.856	HD	13.502	33.270	34.478	T
He-4	4.00260	.749	10	.075	1.7430	.065	He-4	.475			
HT	4.02387	31.271	10	3.127	.9787	1.526	HT	11.097	.895	Q2O	
D2	4.02820	70.944	10	7.094	1.0453	3.695	D2	26.876	.162	CQ4	
DT	5.03015	100.000	10	10.000	.8879	3.959	DT	28.796	.697	N2	
T2	6.03210	44.989	10	4.499	1.0000	1.832	T2	13.324			
CH4	16.0313	.051	10	.005	1.8248	.002	CH4	.017	Moles =	1.030	
NH3	17.0265		10		1.8248		NH3		as H, D, T gas		
CH3D	17.0376	.066	10	.007	1.8248	.003	CH3D	.021	H	.157	
H2O	18.0106	1.512	10	.151	1.8248	.065	H2O	.473	D	.495	
NH2D	18.0328	.223	10	.022	1.8248	.010	NH2D	.070	T	.343	
CH3T	18.0395	.073	10	.007	1.8248	.003	CH3T	.023	Grams H	.316	
CH2D2	18.0439		10		1.8248		CH2D2		Grams D	1.994	
HDO	19.0168	.552	10	.055	1.8248	.023	HDO	.168	Grams T	2.068	
NH2T	19.0348	0	10		1.8248		NH2T		Curies =	19891.3	
NHD2	19.0391	0	10		1.8248		NHD2				
CHD3	19.0501	.122	10	.012	1.8248	.005	CHD3	.037	as water		
Ne-20	19.9924	.287	10	.029	1.9941	.013	Ne-20	.093	H	.006	
HTO	20.0188		10		1.8248		HTO		D	.003	
D2O	20.0231	.623	10	.062	1.8248	.025	D2O	.185	T	.001	
NHDT	20.0411		10		1.8248		NHDT		Grams H	.012	
ND3	20.0454	0	10		1.8248		ND3		Grams D	.023	
CH2T2	20.0477	.146	10	.015	1.8248	.006	CH2T2	.043	Grams T	.003	
CD4	20.0564	0	10		1.8248		CD4		Curies =	33.6	
DTO	21.0251	.089	10	.009	1.8248	.004	DTO	.026			
NHT2	21.0430		10		1.8248		NHT2		as methane		
ND2T	21.0473		10		1.8248		ND2T		H	.002	
CD3T	21.0584		10		1.8248		CD3T		D	.001	
Ne-22	21.9914		10		1.8329		Ne-22		T	.001	
T2O	22.0270	.353	10	.015	1.8248	.006	T2O	.043	Grams H	.003	
NDT2	22.0493	.072	10	.007	1.8248	.003	NDT2	.020	Grams D	.005	
CHT3	22.0560	0	10		1.8248		CHT3		Grams T	.005	
CD2T2	22.0603	0	10		1.8248		CD2T2		Curies =	51.0	
NT3	23.0512	0	10		1.8248		NT3				
CDT3	23.0622	.074	10	.007	1.8248	.003	CDT3	.020	as Ammonia		
CT4	24.0642		10		1.8248		CT4		H	.001	
CO	27.9949		10		1.3082		CO		D		
N2	28.0061	3.874	10	.387	1.3082	.096	N2	.697	T		
O2	31.9898	.050	10	.005	1.5139	.001	O2	.010	Grams H	.001	
Ar	39.9624	4.642	10	.464	1.0332	.076	Ar	.552	Grams D	.002	
CO2	43.9898	.281	10	.028	.7877	.003	CO2	.024	Grams T	.001	
						13.748		100.000	Curies =	12.2	
									96.498	100.000	

5. TEST Plan Change Document

TSTA PROCEDURE/TEST PLAN CHANGE

Date: Oct 8, 1991

TSTA Procedure /Test Plan number: TTA-TP-100-23 R0

Change Number (assigned by QA): 01 01-5

Change to be performed:

During the loop run described in the test plan TTA-TP-100-23, R0, the CEC was found to leak and decided to be isolated. The test plan is therefore to be changed to run the JFCU without any processing of impurities. Impurities from TP1 will be removed by the Palladium Diffuser and pure DT will be sent to ISS. Impurities will be circulated in the He carrier flow of the JFCU and be accumulated. Final recovery of DT will be done by the ZCB1 in the JFCU after the run. Minor tritiated imputiries will be discharged to the TWT. CEC cooling will start immediately and the shift originally planned for CEC cooling will not be necessary on Thursday. The run will be shut down when sufficient data on the interaction between ISS and JFCU is obtained, or the accumulation of impurities in the JFCU requires termination of the run. This change will take effect on October 8 after the approval is given.

Safety Assessment:

This change does not cause any safety concerns during the run. Due to the lack of capability to process tritiated impurity, there may be some tritium sent to TWT after the run.

Future Actions:

The recovery of major tritium in the JFCU will be done as described in the test plan. The CEC will be isolated and a separate plan developed for testing,diagnosis and replacement of the CEC.

Approvals (before change implemented):

Technical Review: _____ Date: 10/8/91

TSTA Safety: _____ Date: 10/8/91

TSTA Management: _____ Date: 10/8/91

TSTA QA: _____ Date: 10/09/91

This form is to be attached to all controlled copies of the procedure/test plan by QA.