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JOINT OPERATION OF TSTA UNDER THE
COLLABORATION BETWEEN JAERI AND DOE
— TSTA LOOP RUN OCTOBER 1990 —
FROM OCT. 1990 TRITIUM RUN TEST PLAN AND RESULT,
TTA-TP-100-19

March 1993

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Joint Operation of TSTA under the Collaboration between
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from Oct. 1990 Tritium Run Test Plan and Result, TTA-TP-100-19

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Under the Annex IV of the US-Japan Collaboration Program, loop test of the TSTA was performed in October, 1990. This test is focused on the Isotope Separation System with 3 distillation columns operated. The system was stably operated and DT products were continuously obtained. Composition profile along the columns were successfully measured with the Gas Chromatograph. In the Fuel Cleanup System, amount of adsorbed tritium on the cold molecular sieve beds were measured and a large impact of this phenomena on inventory was revealed.

Keywords: Nuclear Fusion, Tritium, TSTA, Fusion Fuel Cycle, Isotope Separation, Fuel Cleanup, Cryogenic Distillation, Molecular Sieves, Adsorption, Inventory

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原研-DOE協力によるTSTA共同運転
-1990年10月ループ試験-

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原研は日米協力協定AnnexIVに基づいて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模擬試験を共同で行っている。本試験は1990年10月に行ったもので、同位体分離システムにおいては3カラムによる運転、分離特性の測定、また燃料精製系ではモレキュラーシーブ塔に固定されて残留するトリチウムの挙動測定を主要な目的とした。システムは5日間に渡って安定に運転され、高濃度T₂の代わりにDTを供給燃料として取り出す簡便な配位が実証され、また深冷分離塔の塔内成分分布の定量的な測定に成功した。燃料精製系では装置停止後もモレキュラーシーブに残留するトリチウムのインベントリーに与える影響が明らかとなった。

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I. Introduction

Annex IV to the Implementing Arrangement between the Japan Atomic Energy Research Institute and the United States Department of Energy on Cooperation in Fusion Research and Development was signed in June 1987 for the joint research on the fusion fuel processing technology. Under this agreement, JAERI and DOE jointly fund and operate the Tritium Systems Test Assembly (TSTA) at the Los Alamos National Laboratory, that is a working prototype of a fuel processing loop for a fusion reactor. This system consists of a torus mockup with cryopumps, Fuel Cleanup System that purifies simulated plasma exhaust and generate pure hydrogen isotope stream, Isotope Separation System consists of four cryogenic distillation columns, waste treatment and cleanup systems, and associated storage, delivery, and transfer pumping systems. A number of "loop runs", have been performed to investigate the characteristics of the fuel loop and to develop better understanding on design and operation of the fuel systems at the TSTA since 1987. This report describes the loop test of the TSTA performed in October, 1990. This test is focused on the Isotope Separation System with 3 distillation columns operated. While four columns are interconnected in regular configuration to produce T_2 , DT, D_2 and tritium free HD stream, three columns configuration was tested for production of D_2 and DT with easier and more stable operation. In a stable state of the distillation columns, Gas chromatograph analysis is attempted to obtain composition profiles along the columns.

The other major objective of the runs is to study the nature of the molecular sieve beds that are operated at the liquid nitrogen temperature in the FCU. The difference in tritium inventory in the loop before and after the previous run was found and adsorption of tritium on the molecular sieves was suspected.

II. Test Plan TTA-TP-100-19

TEST PLAN OCTOBER 1990 TRITIUM RUN

1. Purpose

The primary purposes of this run are:

- 1) to obtain the ISS performance with a 3 column configuration without column T, where the top stream from Column D is feed back to the feed of Column H and I and to explore conditions needed to get lower tritium concentrations off the top of Column H. The feed back flow effects on concentration profile of columns for each species are to be determined.
- 2) to understand the tritium inventory held on the FCU molecular sieve beds. After extensive H₂ swamping, MSB2, CR1, and associated FCU tubing is believed to be essentially tritium free. Flowing DT through the FCU will provide an opportunity to study the transfer of tritium from the gas phase onto the stationary phase by exchange with hydrogen (already bound at the surface). The amount of tritium that was transferred will be determined by UTB inventory difference before and after the run.
- 3) to provide operating experience for TSTA staff.
- 4) to process FCU surge tank gas which contains approximately 2 grams of tritium in a total of about 100 liters of H₂. The surge tank gas will first be transferred to the UTB standard volume and inventoried. Then the hydrogen isotopes will be placed on UTB-3 (currently empty) and the dregs transferred to a 50-liter PC at LIO. At the beginning of the run, the UTB-3 gas will be loaded into ISS along with other UTB gas. These preparatory operations will be covered by a separate test plan.

2. Configuration

In this run, the TSTA flow loop will be configured in the ISS 3 COLUMN mode. Column I, H and D will be interconnected as shown in Figure 1. A portion of Column D is recycled to the feed of Column H through FCU MSB3 and the remainder of that stream goes to the feed of Column I through FCU MSB2. Both flows are pumped by TP3 MBP-A. The bottom stream of Column D flows through TP3 MBP-B and TP1 MBP-B, and the bottom stream of Column I through Column T, which is just a path and kept warm without cooling the condenser or packing, join the top stream of Column D before the TP3 Equilibrator. The waste gas off the top of Column H is discharged to TWT LPR. The system will be operated in any of the following control modes: local, computer manual.

UTB and TP1 are required to recover GC sampling and purge gases. If the pressure in the GC vacuum manifold becomes too high due to blanketing, the Standard Volume can be used to dump the residual gas from there. This gas will be circulated over a Ubed and then dumped to TWT after the run.

One refrigerator compressor will be used to supply helium refrigerant to ISS. If the capacity is not enough, both compressors should be operated.

The TSTA test cell will be configured to maintain the proper pressure differential. This requires that all door and air lock procedures be followed.

3. Subsystems Required

Process systems that will be used are: TP1, TP3, IMS, ISS, UTB, GAN, and FCU. PEV, and VAC. Other systems will be required, these are TWT, ETC, PEV, VAC, TM, VEN, EGS, UPS, SEC and MDAC. The following utility systems will be required; house vacuum system, air, helium, nitrogen (liquid and gaseous), and hydrogen-argon.

4. Personnel

All TSTA personnel will be required for this run. When not at the TSTA facility during their shift, personnel are expected to be on call as required.

During the run we specify a Loop Operator (LO). This person will generally be one of the Facility Operators. However, at times the Test Director (TD) can appoint an alternate to replace the LO.

The loop operator's responsibilities will be the coordination of loop operations and the other TSTA systems. He will work directly with the Test Director to handle the operations from the MMI. The LO will execute most of the MMI commands (especially the loop operations). The LO must be kept informed of all operations going on in the facility. The LO will also handle most of the communications from the control room.

Huddles (shift change meetings) will be held at 0730, 1530 and 2330 as usual. All TSTA members should attend huddles during the working day. The Loop Operator or Facility Operator for the oncoming shift will log in the Control Room log book all essential information exchanged during the shift change meetings and huddles. The Loop Operator or Facility Operator for the leaving shift will record, in the control room notebook, all information to be exchanged at the shift change meeting. This log in the notebook should correspond to and expand, as necessary, the information written on the support center white board.

The responsibilities for personnel during normal operations are listed in Tables I and II.

During an "emergency" (determined by the Test Director or the Loop Operator), Table III gives personnel assignments for SHIFT A and B. For SHIFT C the Loop Operator will determine the assignments.

Under emergency conditions we have the ability to isolate components and immediately dump the process gas to the uranium storage beds, thus controlling gas pressures and flows, and maintaining safe operating conditions. The emergency actions may be initiated either manually or automatically. SCRAM macros are programmed into the computer system for the transfer pumps, isotope separation, load-in/out, and PEV systems. These are predetermined sequences of operations which will shut down a particular process and restore safe conditions; they may be called up manually by the operator. Safety programs are resident in the computer system which automatically senses critical process variables and take appropriate action when necessary. For the transfer pump system, a metal bellows pump outlet pressure in excess of 1500 torr will initiate the SCRAM macro. For the isotope separation system, a combination of condenser temperature and column pressure parameters is sensed. If unsafe conditions are approached, valves to the uranium storage beds are automatically opened and the column contents are dumped without any intermediate processes.

The TSTA process loop has been designed to "fail-safe", that is, under a complete loss of electrical power all process valves will fail in the safe position, thus avoiding a loss of tritium to the secondary containment, and/or the environment. This failure mode has been tested.

5. Schedule

The time for tritium operations requiring continuous staffing of the TSTA facility will nominally be from Day 1 to at the latest Day 6. The run is expected to begin on Oct. 21, 1990 and end no later than Oct. 26, 1990.

Prior to the run the intercolumn flow controllers and other instruments will be checked, adjusted and tested according to RHS standard list.

The low level tritium leak checking of the process systems will be done as necessary on any joints which have been broken open or on any newly installed plumbing.

The ISS refrigerators will be checked and the ISS columns will be evacuated and purged with helium three times.

The transfer pumps in TP3 and ISS should be turned on for 5 minutes every time helium is filled. Helium will be maintained in the ISS system until the refrigerator starts.

All support systems will be checked and gas supplies ordered as necessary.

- (1) The TWT GC, which will separate neon from the hydrogen isotopes, will also be checked out.
- (2) TP3 will be connected, leak checked and tested.
- (3) The vacuum jackets for the UTB's will be evacuated.
- (4) The gas in the FCU surge tank, which is mainly H_2 and around 100 liter, will be circulated over UTB-3, which is empty, to collect hydrogen isotopes. The remaining gas phase components should then be transferred to a dregs PC at LIO. The hydrogen isotopes which were collected will be one source of feed gas during the run.
- (5) MSB2 has been thoroughly regenerated during the H-exchange test. MSB3 needs to be regenerated. This regeneration will be done according to the standard regeneration procedure.
- (6) At least 2 empty PC's will be attached to the LIO to provide surge volume. No pure tritium loading or removal is anticipated into or out of the loop during the run.
- (7) The new TWT Recombiner B will be on-line if operational checks are successful.

Operations with the process systems will begin on Day 1. The helium refrigeration for the ISS columns will be started on Day 1 and the temperature set at approximately 23K. The liq N_2 cooling of the ISS will be started on Day 1.

Detailed schedules for operations at FCU are covered in section 7.0.

The general schedule for operations at ISS are covered in section 7.0 (ISS Operations)

6. Possible Hazards

Large quantities (about 20 grams) of tritium will be handled during this run. If accidentally released, it could pose a health hazard to personnel. All operations that will be done during the run are covered by procedures approved through the TSTA QA system. To mitigate the effects of accidents, all safety systems are required to be operational.

7. Outline

The run will begin with SHIFT B on Day 1. Shutdown will start during SHIFT A on Day 6.

7.1 Preparation Completed before Day 1

- (1) The ISS Low-level Ion Chamber will be purged with moist N_2 during the week prior to the run. The inlet is external to the main ISS glovebox and the purge gas will go directly to the TWT with no other interaction with the process loop.
- (2) Inventory UTB-4.
- (3) Detach any unnecessary gas bottles from IMS and attach deuterium and hydrogen bottles to IMS. Evacuate gas feed line.
- (4) Complete Prestart checkoff sheet.
- (5) Perform flowmeter intercomparison, ISS, TP3, and FCU (ref RHS standard list and TTA-TP-100-17).
- (6) Evacuate ISS and TPU to 50 torr; backfill with helium to 800 torr. Purge ISS GC lines.
- (7) Start TP1 and TP3 pumps and open all loop circulation valves; evacuate to 50 torr. Circulate for 5 minutes.
- (8) Repeat ISS and TP purge three times.
- (9) The ISS will be left at 800-900 torr of helium.
- (10) Evacuate ISS vacuum jacket below 10^{-3} torr.
- (11) Evacuate ISS helium transfer line vacuum spaces.
- (12) Supply LN_2 to ISS.
- (13) Attach a D_2 cylinder to the FCU regeneration gas manifold.
- (14) Evacuate, backfill with 4He , and re-evacuate the UTB standard volume.
- (15) Properly configure the three manual feed valves. Lower valve should be open for startup.

7.2 Friday before Run

- (1) Set temperature controllers on UTB-2 and UTB-4 to $200^\circ C$ (hereinafter refer TTA-SOP-108).

Note; UTB-2 and UTB-4 contain 9.031g of hydrogen, 141.056g of deuterium and 19.585g of tritium according to recent UTB inventory. These amounts are enough for the initial gas loading for the run. UTB-3, containing the residual hydrogen isotopes from the UTB standard volume, will also be loaded into the loop.

7.3 Day 1

- (1) FCU MSBF2 shall be given one last pumpout to TWT. Then LN_2 will be able to be turned on to MSBF2, MSB2 and MSB3. The MSBF2 temperature controller should be set to 190K.

- (2) 3:30 PM Shift meeting in support center.
- (3) Evacuate helium gas in the ISS columns.
- (4) Start He refrigerator. Column T should be kept warm by not cooling either the condenser or packing.
- (5) Set up the flowpath for GC sample and purge gas recovery and start up the GCs and data station of the ISS-GAN. FCU GC may also be turned on and made operational.

UNLOAD U-BEDS

- (6) Set temperature controllers on UTB-2 and UTB-4 to 450°C.
Set Safety controllers and set points of MDAC Safety Program to 500°C for UTB-2 and UTB-4.
- (7) When pressures in UTB-2 and UTB-4 are slightly greater than the ISS column pressures, open the valves to the ISS. UTB-4 should be loaded through MSB2.
- (8) Continue unloading UTB-2 and UTB-4 until empty, as determined by closing the ISS EVAC valves and monitoring the pressure increase over the top of the Ubed.
- (9) Set temperature controllers on UTB-2 and UTB-4 to 0°C.
- (10) Set temperature controllers on UTB-3 to 200°C.
- (11) After UTB-2 and UTB-4 are turned off, set the temperature controller on UTB-3 to 450°C.
- (12) Set Safety controller and set point of MDAC Safety Program to 500°C for UTB-3.
- (13) When the pressure in UTB-4 reaches the pressure in the ISS columns, begin pumping gas into ISS.
- (14) When UTB-3 is unloaded, reset the MMI and its controller to 0°C.
- (15) Preload MSB3 with D₂ supplied from the cylinder attached to the FCU regeneration gas manifold. Be sure to meter the gas in using the MKS flow controller. Record the start and stop time, and the flow rate. Stop the gas flow to MSB3 when the overpressure is 800 torr.

7.4 Day 2 to 5

- (1) 7:30am/3:30pm Shift meeting in support center.

LOOP OPERATIONS

- (2) As soon as the gas from the UTB's has been loaded into the ISS and the loop leak checked with the high levels of tritium now in the system, loop flow through ISS/TP3/TP1/FCU can be started. Operations, in general, will be as detailed in the most current revision of TTA-SOP-100-3, "Working with Tritium." Valves to be opened under 3 Column mode are as follows.

ISS: CLIC, Q-CLI, F-CLIA, SGBYP, F-CLH, CLHA, Q-CLHB, F-CLD, CLDA,
F-CLDB, F-CLTA, F-CLTB, and CLTB.

FCU: IN2, IN5, IN6, CR10, CR12, CR14 and CR15.
MSB20, MSF20, MSB22, MSB23, ISS2, ISS5 and MSB26.
MSB30, MSB32, MSB33 and MSB36.

TP3: AV1, AV2, AV3, AV4, AV5, AV6, AV9, AV10, AV12 AND AV13.

Maintenance manifold - OFF.

TP1: AV11, AV13. Maintenance manifold - OFF.

Other valves in FCU, ISS, TP3 and TP1 must be closed. TP3 pumps(MBPA and B), TP1 pump(MBPB) and ISS pumps(MBPA and B) are turned on.

FCU OPERATIONS

(3) The FCU main purification flow path is through CR1, MSBF2, and MSB2. The NBI path is through MSB3.

ISS OPERATIONS

(4) Initial ISS conditions are shown in Table IV. Establish stable flow conditions in the ISS with nominal flows determined based on an analysis of the feed stream to Column I after initial stable conditions have been obtained. For example, with a 50-50 D-T inlet ratio, just under 25% of the flow should be extracted as a top stream. H_2 will be injected by the IMS at a rate of 1% of the Column I feed flow. For the first 3 days the flows and reboiler heater powers will be determined based on the feed composition to column I such that a reflux ratio of at least 30 is maintained (about 35 Watts) and a top fraction withdrawal such that the expected HT concentration at the top of column H will be of the order of $X=1 \times 10^{-10}$ (X-mole fraction). The reflux ratio in column H should be maintained at about 200, which implies a heater power of about 20 W and a product withdrawal rate of around 50 cm^3/min .

(5) The Column H top flow will be varied as an experimental parameter between 100cc/min and 400cc/min during the run. The Column I top flow will also be changed to 800cc/min.

(6) The Column H effluent gas will be monitored continuously by the Low Range 1-liter ionization chamber in order to have an accurate measure of the HT content of this product stream. Before opening CLHA to TWT, this monitor will be read locally at ISS and reported to the loop operator. Both the monitor reading and the scale setting should be read and reported. Acceptable tritium level to be sent to TWT is below 100 uCi/min.

(7) During periods of stable operation of ISS, GC analysis will be made of feed, top, bottom, and sample tap compositions. It will be especially important to obtain the profile of Column H.

(8) H_2 gas will be continuously added; D_2 gas will be added from time-to-time as necessary to keep the ISS system stable.

(9) At all times the ISS will be operated within the safety limits defined in the most current revision of ISS safe operating procedure, TTA-OP-103-12.

Table IV.

Initial ISS Conditions

	Column I	Column H	Column D
Top Flow, cm ³ /min*	1300	50	4750
Feed Flow "	6000	5000	5000
Bottom Flow "	4700	5000	250
Column Pressure, Torr	860	800	700
Reboiler Power, W	35	20	10

* sccm

EMERGENCY SHUTDOWN OF ISS

If it should become necessary to perform an emergency shutdown of the ISS while ⁴He is being added to the loop, the uptake of hydrogen isotopes by the uranium beds in UTB will become diffusion limited, and hence sluggish, for columns I and H. This condition can be alleviated by circulation through the UBed affected and TPl, or by opening to the ISS surge tank.

If a plugging of ISS feed lines occurs prior to 3 days before the end of the run proceed as follows:

- 1) First determine the cause of the plug,
- 2) If it is determined that the plug can probably be cleared, the contents of ISS will be dumped to UTB,
- 3) Warm ISS as necessary to clear the plug,
- 4) Recool ISS, reload Q₂ from UTB and restart ISS, TPU and IMS operations.

If a plug occurs near the end of the run then ISS operation will be terminated.

SYSTEM SHUTDOWN, Day 6

(1) After the TPU pumps have been turned off and before ISS is opened to UTB, MSB2 will be isolated and briefly evacuated with TPl back into the loop. Then, at a convenient time during shutdown a path from MSB2 to the UTB standard volume should be established (through the FCU vac manifold and TPl) after making sure that the UTB standard volume had been previously evacuated. The MSB2 LN₂ will then be turned off and its heater turned on. When MSB2 reaches 400K the TPl scroll pump will be used to pump the remaining MSB2 gas into the standard volume. Thereafter, MSB2 will be isolated and may be given one last evacuation with house vac before its heater is turned off. A sample of the standard volume gas will then be collected for mass spectroscopic analysis before the standard volume gas is admitted to a UTB bed.

(2) Shutdown of the process loop will be in accordance with TTA-SOP-1Q8,R3. The valves to UTB-2 and UTB-4 will be opened, then unload the gas in the Columns to these U-beds. If the gas is not reacted rapidly due to helium blanketing, the gas should be circulated with TP3 over the U-beds. Raise the temperature of the helium refrigerant gradually to 27K. After the liquid level in the reboilers of Columns become zero, turn off the reboiler heaters and then stop LN₂ supply.

(3) Once it is established that UTB is safely accepting the gas evolved from ISS, liq N₂ to FCU-MSB3 will be turned off. After 30 minutes its heater will be turned on and the Variacs set to 50 V. The bed temperature must thereafter be monitored closely as a Variac setting of 50 V will overheat the bed if left unattended. The beds should be heated to 400 K which will require about 9 hours from the time the liq N₂ is turned off. After first obtaining a mass spectrometer analysis, the gas evolving from MSB3 will be allowed to flow into ISS Column H and thence onto UTB. To complete this operation, isolate MSB3, use TPI to evacuate MSB3 back to the loop, and then turn the MSB3 heater off.

(4) Isolate all remaining components in the FCU and evacuate them back to the loop.

(5) Circulate gas through the system (TPU-ISS) over the UBeds to remove the remaining hydrogen isotopes.

8. Data Requirements

(1) All TSTA data channels, beginning with the cool down of the ISS, will be stored on tape after the run.

(2) Relevant subsystem data will be recorded in the appropriate log books.

(3) Results of GC analysis and process data are most valuable because they will be used for cryogenic distillation analysis after the run. These numerical data are key results of the run.

(4) The amount of tritium sent to TWT will be estimated by integration of the ion chamber readings together with the associated flowmeter.

(5) A complete inventory of any PC's used in the run and all UBeds will be completed after the run and before this test plan can be closed out.

(7) Loop Operators will record notes on the operations at the MMI in the Control Room log book .

(8) Shift change and huddle minutes will be recorded in the hard-bound operations log book.

Table I Shift Assignments

	Shift A (7:30-15:30)	Shift B (15:30-23:30)	Shift (23:30-7:30)
Test Director	Anderson	Bartlit	
Alt. Test Dir	Sherman	Carlson	
Loop Operator	Binning	Jenkins	Wilhelm
Facility Operator	Harbin		King
FCU	Anderson	Carlson	
	<u>ab</u> Willms		
ISS	Sherman [JAERI]	Barnes [JAERI]	JAERI
UTB	Nasise	Nasise	
TPU	Binning	Jenkins Nasise	
LIO	Baker	Nasise	
TWT	Binning	Nasise	
ETC	Binning	Carlson	Wilhelm
IMS	Coffin (Mon./Tues.) (Nasise)(Remainder)	Nasise	
GC	Sherman JAERI	Willms JAERI	JAERI
MDAC	Cole	(Cole)	

NOTES:

1. All others will work SHIFT A (attending shift change meeting) and be subject to call as needed.
2. Some personnel on Shifts A & B may shift hours between shifts (with the cognizance of the Test Director) depending on specific activities under way.
3. Personnel in (...) are on call. Personnel in [...] are in training.
4. Details of JAERI assignments are given in Table II.

Table II JAERI Shift Assignments

Date	Ab	bC	A	B	C
Day 1				Inoue	Konishi
Day 2	Yamanishi	Nabe	O'Hira		
Day 3	Inoue	O'Hira	Konishi		
Day 4	Nabe	Inoue	Yamanishi		
Day 5	O'Hira	Yamanishi	Nabe		
Day 6	Konishi	(Nabe)	Inoue		

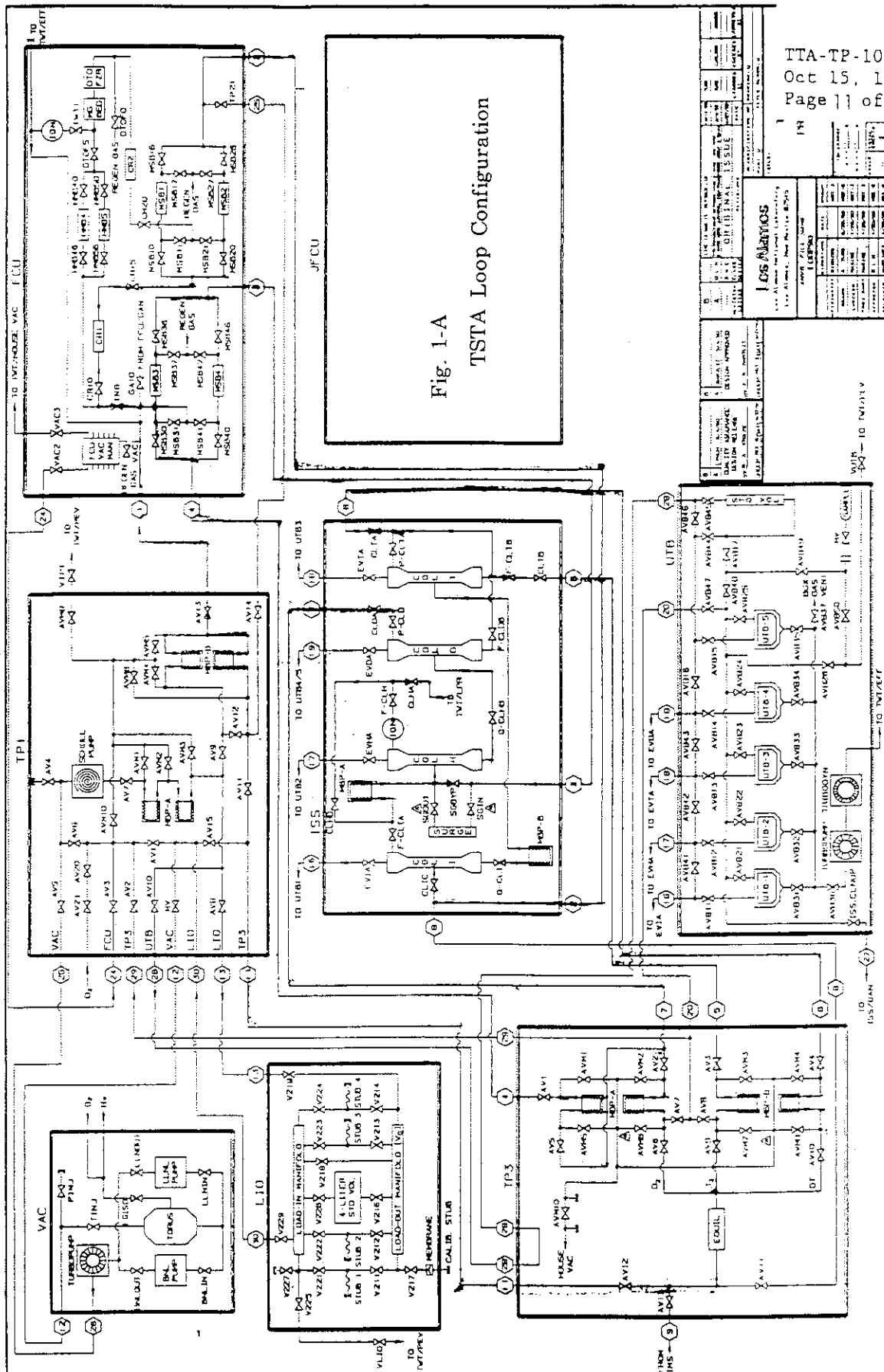
Notes:

- A - Shift A, 0730 to 1530
- B - Shift B, 1530 to 2330
- C - Shift C, 2330 to 0730
- Ab - Shift Ab, 0730 to 2000
- bC - Shift bC, 1930 to 0800

Table III Emergency Assignments

Role	Shift A	Shift B
Test Director	Anderson	Bartlit
Loop Operator	Binning	Jenkins
ISS/consulting	Sherman	Bartlit/Barnes
GAN/consulting	Sherman	Willms
TPU/consulting	Binning	Nasise
LIO/consulting	Anderson	Nasise
FCU/consulting	Willms	Willms/Carlson
UTB/consulting	Anderson	Nasise
TWT/operator	Binning	Jenkins/Nasise
MDAC/consulting	Cole	Cole
TM/consulting	Harbin/Miller	Carlson/Miller
ETC/operator	Binning	Jenkins/Carlson

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Los Alamos

ISS CONFIGURATION FOR 3-COLUMN RUN

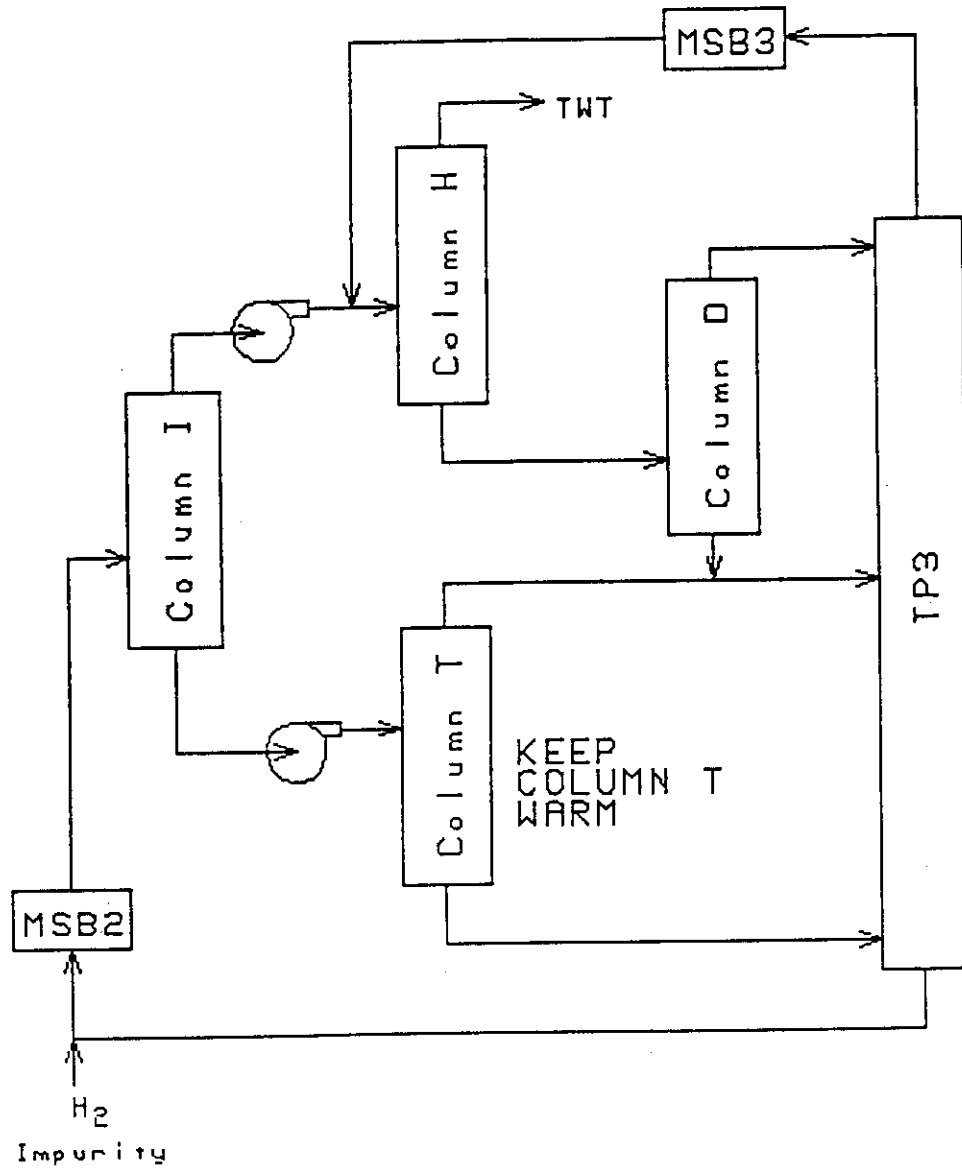


Fig. 1-B TSTA Loop Configuration

III. Test Results

TEST RESULTS OCTOBER 1990 TRITIUM RUN

1. Summary

Operation of the Isotope Separation System to produce D2 and DT products using three columns was successfully demonstrated. This operation mode requires one less column than the normal D2 and T2 production mode and significantly reduces tritium inventory. Tritium levels in the He-HD waste stream to the Tritium Waste Treatment (TWT) system averaged 0.4 ci/m³ or 40 % of acceptable limits.

Isotopic exchange experiments were conducted on the Fuel Cleanup Unit (FCU) Molecular Sieve Bed (MSB) to obtain information on tritium inventory variations in MSBs. Problems with sample and data accountability limited the information gained from these experiments. The experiments will be continued during future tests.

Additional operator experience was gained during the run. One newly hired operator received on-the-job training and will be qualified for Loop operation in the future.

Approximately 26 L of mixed hydrogen isotopes collected during a previous isotope exchange experiment were processed and recovered on the uranium beds.

2. Purpose

A TSTA Loop Test Run was conducted during the week of October 21-26, 1990. The primary purposes of this run were to:

1) Test the Isotope Separation System (ISS) using a three column configuration; Columns I, H, and D, rather than the normal four columns. This configuration produces three product streams; a D2 product stream, a DT product stream, and an He/HD waste which is sent to the Tritium Waste Treatment (TWT) system for final cleanup before disposal. The T2 stream produced in the normal four column operation is not produced in this configuration. Data were collected to determine the effects of this operation on

- column concentration profiles, and
- tritium concentration in the Column H overhead waste stream.

Operating conditions planned for the test run are shown in Table 2-1.

Table 2-1 ISS Operating Conditions

	Column I	Column H	Column D
Top Flow, L/min	0.8, 1.3	0.05, 0.1, 0.4	4.75
Feed Flow, L/min	6.0	5.0	5.0
Bottom Flow, L/min	4.7	5.0	0.25
Pressure, torr	860	800	700
Reboiler Power, watts	35	20	10

2) Evaluate tritium inventory in Fuel Clean-Up Unit (FCU) Molecular Sieve Beds (MSBs). MSB2 was previously swamped with H₂ to remove deuterium and tritium. DT flowed through MSB 2 during the Test Run. Loop inventory measurements made at the end of the run were to be used to determine the extent of isotopic exchange occurring in MSB2.

3) Provide operating experience for TSTA personnel.

4) Process gas contained in the FCU surge tank to purify and recover approximately two grams of tritium gas.

4. Operations

The following is a brief description of events that occurred during the test run. All times are referenced beginning Monday, October 21, 1990.

4.1 Pre-run Preparation

Pre-run operations were conducted the week of October 14-19, 1990 to prepare for the Test Run which was conducted the following week. These operations included

- cleanup of the low-level ion chamber in the Column H overhead stream by moist nitrogen gas purging,
- inventory of gas accumulated in UTB-4 since the last Loop run, and
- normal pre-run system checkouts and preparations.

4.2 Sunday, October 20

Cool-down of the ISS columns and heat-up of the UTBs began on Sunday, October 21.

4.3 Monday, October 21 (0 - 24:00)

Load-in started at 08:00. Loop flow was initiated at 20:00. Plugging at the Column I inlet, suspected at 23:00, was verified at 24:00.

4.4 Tuesday, October 22 (24:00 - 48:00)

The ISS was unloaded to the UTBs at 24:00. Plug disappeared at 25:00 and reheating of the UTBs was initiated. Gas was loaded into the columns at 33:00. H₂ was added at 36:00. GC analysis

began with the B shift at 40:00. At 45:00 it was noticed that CLHA flow did not react to controller changes.

4.5 Wednesday, October 23 (48:00-72:00)

Full Loop operation with circulation through the columns began with the A shift at 56:00. CLHA flow problems were attributed to a stuck valve.

4.6 Thursday, October 24 (72:00-96:00)

The CLHA flow problem was traced to valve #1 on the CLHA low level radiation monitor at 80:00. This valve had not been opened preventing flow through CLHA. The valve was opened and F-CLHA meter rezeroed. Radiation levels at CLHA went off-scale but rapidly returned to 0.3 ci/m³. GC analyses indicated high concentrations of He and H₂ at the top of the column. Column flows during day 4 are shown in Table 4-1.

Table 4-1 Test Day 4 ISS Operating Conditions

	Column I	Column H	Column D
Top Flow, L/min	0.8-1.0	0.0	3.0-3.8
Feed Flow, L/min	7.5-9.5	5.5-7.5	3.0-4.0
Bottom Flow, L/min	4.0-7.0	3.0-4.0	0.5
Pressure, torr	650-800	700-850	575-675
Reboiler Power, watts	35	17-18	12-14

4.7 Friday, October 25 (96:00 - 120:00)

The ISS operated at near stable conditions during the C shift, 96:00 to 104:00. Samples were taken to establish column concentration profiles. The system was shutdown at 108:00. Column flows during day 5 are shown in Table 4-2.

Table 4-2 Test Day 5 ISS Operating Conditions

	Column I	Column H	Column D
Top Flow, L/min	1.0-2.0	0.2-0.35	3.0
Feed Flow, L/min	4.5-5.5	3.5-4.0	3.0-4.5
Bottom Flow, L/min	3.0-6.0	3.0-4.5	0.5
Pressure, torr	650-750	700-800	600-650
Reboiler Power, watts	30	17-22	10-15

5. Experimental Results

5.1 D2-DT Production

Operation of the ISS under desired experimental conditions was successfully demonstrated during the final 25 hours of the test run. Design conditions were not achieved during the initial 84 hours because of normal startup activities, pluggage in the Column I feed line which required recycling of the startup sequence, and problems encountered with measurement and control of waste (He-DT) flow.

The length of the run and amount of useful data was limited by flowmeter-controller stability problems. After the problem was diagnosed and corrected the run proceeded. However, the full envelope of flow conditions and concentration profiles was not explored because of a lack of time. Future runs will develop this additional data.

Samples were taken and analyzed by gas chromatography (GC) at multiple points along Column I and Column H to establish concentration profiles when operating in the three column D2-DT production mode. Concentration profiles are plotted in Figures 5-1 and 5-2. Design concentration profiles under the normal four column D2-T2 production mode are shown for comparison.

Concentration profiles for Column I as determined from GC analyses are compared with theoretical calculations in Figure 5-3. Differences between measured and theoretical values most likely were caused by failure to reach steady-state conditions.

GC concentration profiles for Column H are compared with theoretical calculations in Figure 5-4. Theoretical and measured values are not in good agreement for these data. This is attributed to (1) the problems encountered with control of the overhead flow

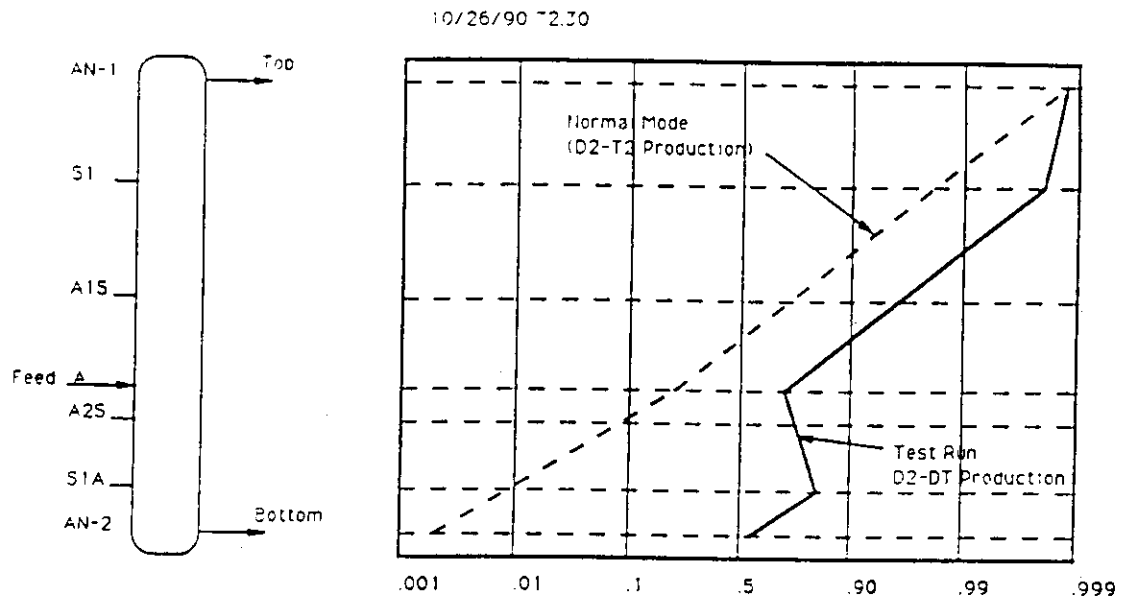


Fig. 5-1 Column I D2 and Lighter Concentration Profile

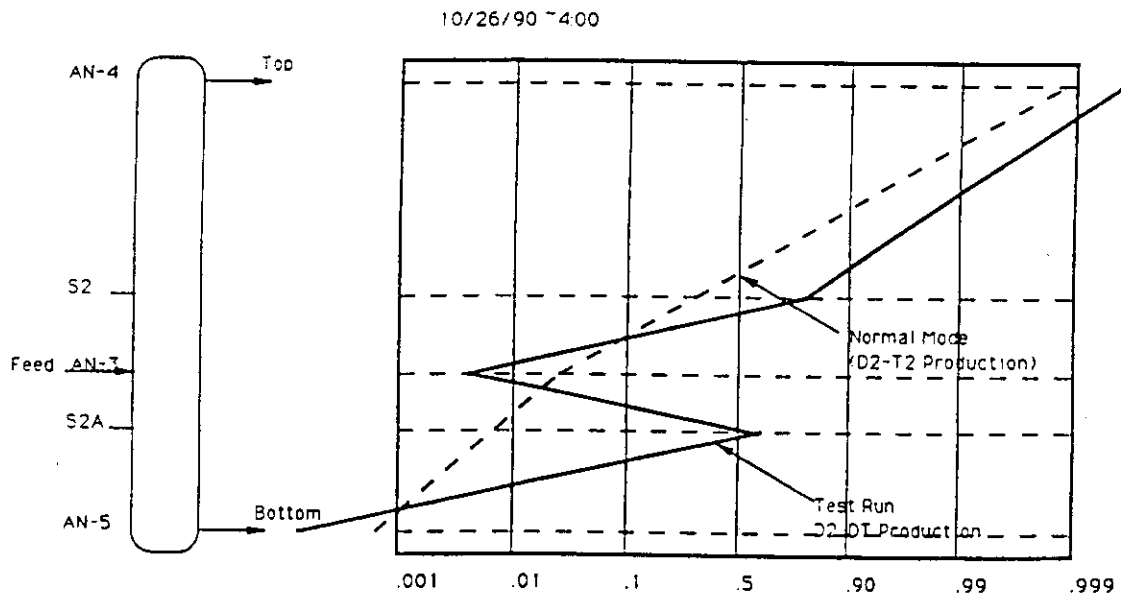


Fig. 5-2 Column H D2 and Lighter Concentration Profile

from Column H and (2) inability to maintain steady-state conditions during the test run.

Additional comparative analysis of system behavior using a dynamic simulation code is being performed by M. Inoue. Results of this study will be documented in a separate report.

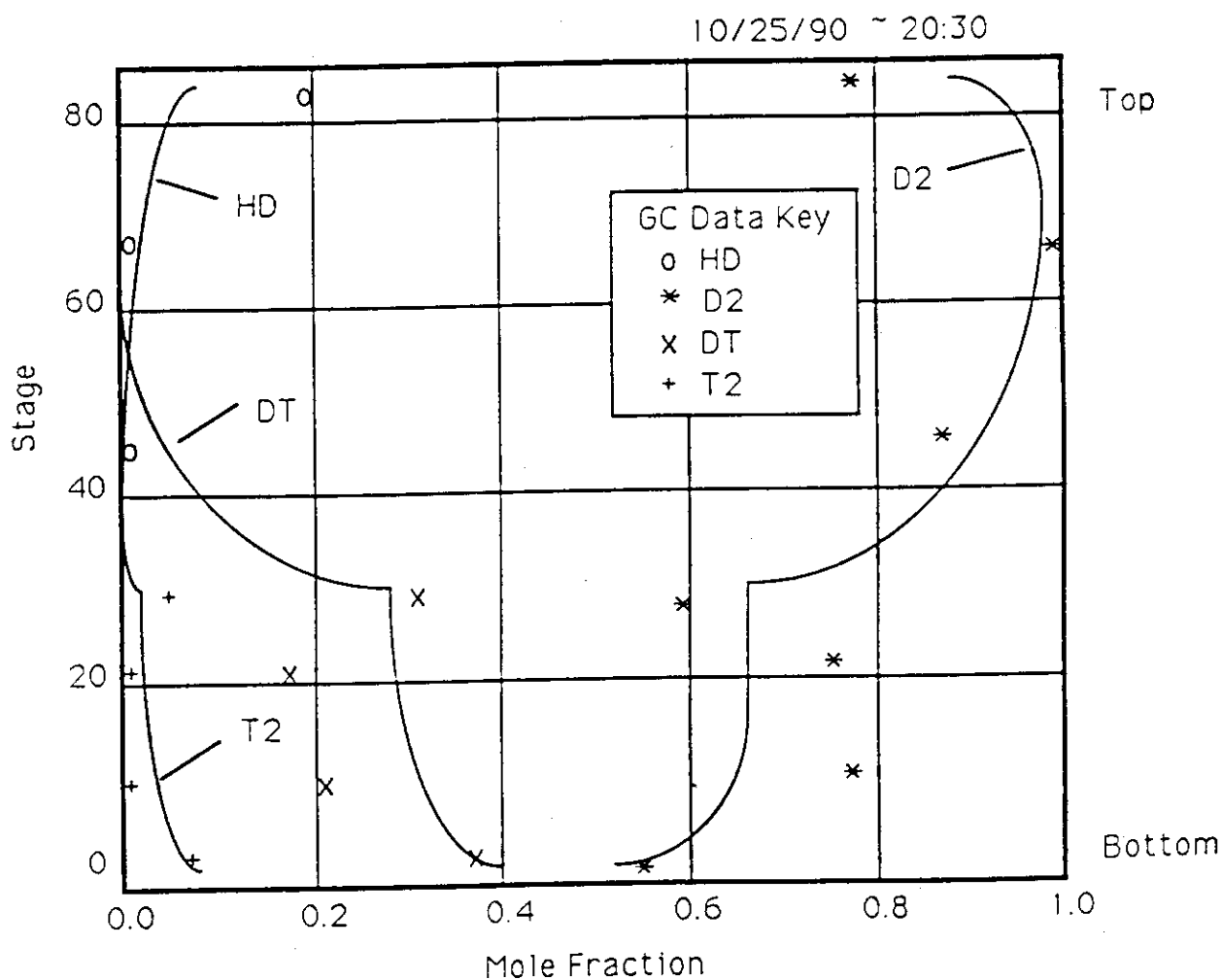


Fig. 5-3 Column I Comparison of GC Measurements and Calculated Concentration Profiles

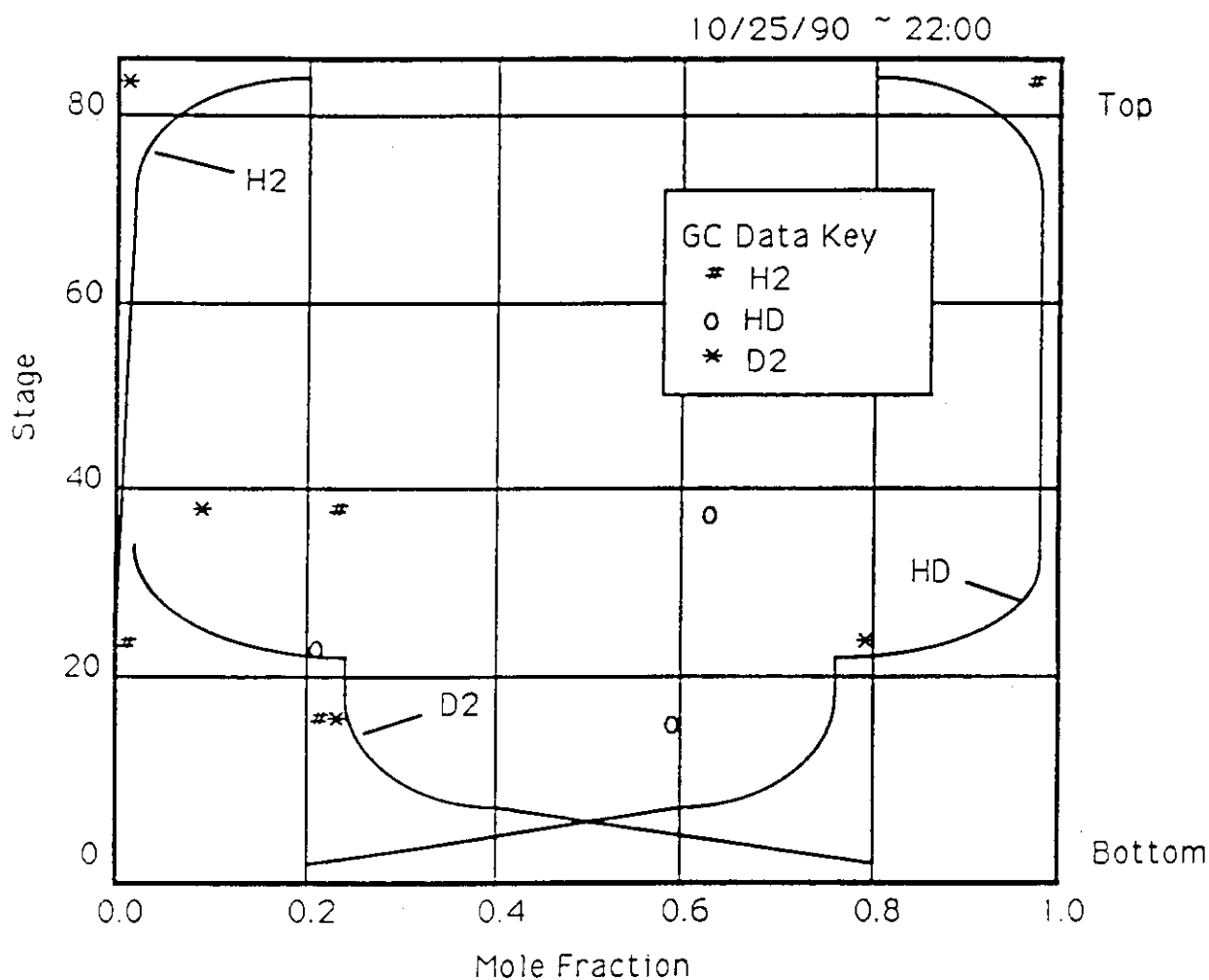


Fig. 5-4 Column H Comparison of GC Measurements and Calculated Concentration Profiles

Tritium concentrations in the He-HD waste stream during the final 20 hours of the run when design conditions were attained are shown in Figure 5-5. Tritium discharges to the TWT averaged 40 % of allowable discharge levels during this time period.

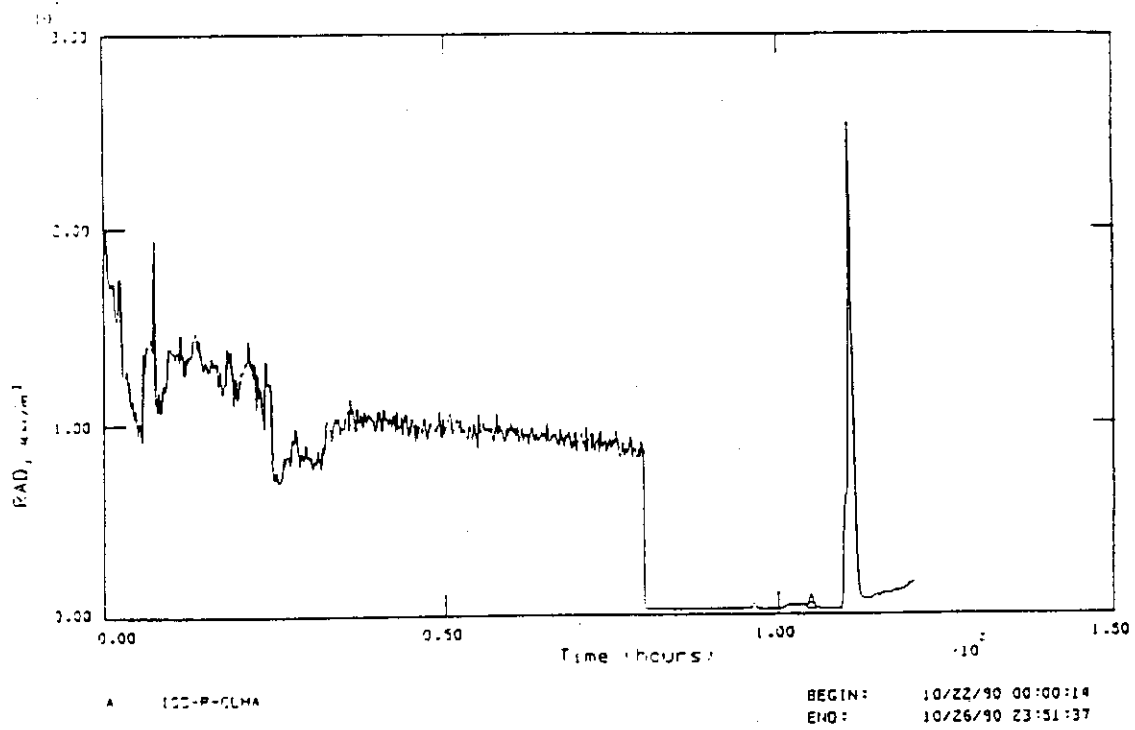


Fig. 5-5 F-CLHA Tritium Concentration

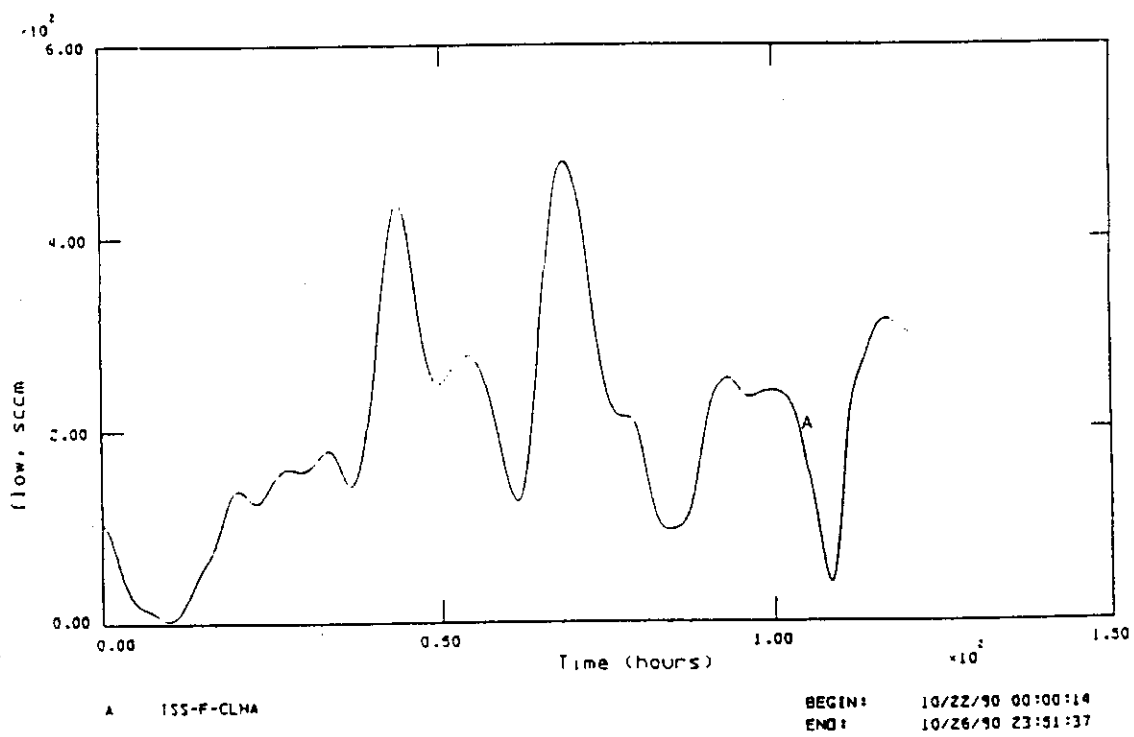


Fig. 5-6 FC-CLHA Indicated Flow

Problems with flow measurement-controller zero stability led to erroneous readings on the He-HD stream (F-CLHA) during the 45 - 80 hour time period. Figure 5-6 shows readings for F-CLHA during the test run. No flow occurred through F-CLHA during the first 80 hours of the test run. The problem appears to be generic to thermal mass flowmeter-controllers installed in the TSTA. Flow measurement-controller systems are being modified to eliminate the observed problems. These modifications will be tested after the modifications have been completed.

5.2 FCU Mol Sieve Bed Inventory

Problems with sample and GC analytical data traceability and accountability limited the information gained from this test run. These experiments will be repeated during a future run.

At the end of the run GC samples were collected at the feed to ISS Column I. This was indicative of the gas concentrations flowing over MSB2. The results are as follows:

<u>Sample</u>	<u>H(%)</u>	<u>D(%)</u>	<u>T(%)</u>
1	4.71	77.47	17.83
2	5.92	73.64	20.44

Future H₂ exchange experiments will be used to determine the extent to which this isotopic mixture exchanged with the Q₂O adsorbed on MSB2.

IV. Conclusion

The Isotope Separation System was stably and successfully operated and DT products were continuously obtained. In some modes and configurations in the future fusion reactor fuel systems, pure tritium will not be necessarily used as a fuel and DT mixture will mainly be fed to reactor. This test demonstrated the fuel production for such application. Major advantage of this configuration of the distillation columns are easier and more stable, and much less tritium inventory than that for extracting pure tritium.

Composition profile along the columns were successfully measured with the Gas Chromatograph. However it took several hours for the entire measurement with GC, while operating distillation columns stably for that period is extremely difficult. It is strongly desired to install much faster analysis system for the ISS. Laser Raman Spectroscopy is a planned improvement for near future.

In the Fuel Cleanup System, amount of adsorbed tritium on the cold molecular sieve beds were measured by isotopic exchange on the beds. Incomplete regeneration as well as the existence of the "permanent" water in molecular sieves are suspected to be the potential tritium inventory that exchanges with gas phase hydrogen isotopes. It should be noted that this phenomena will give a large impact on inventory in the loop.

APPENDIX A
ISOTOPE SEPARATION SYSTEM TEST DATA

Table A-1 ISS EVENT LOG October 1990 Test Run

Time, hrs	Explanation
1	1:00 am Monday, October 22, 1990
9	- Started UTB to ISS gas transfer
20	- Started Loop flow - Started Column H heater
22	- U Beds opened to EVs I, T, and D
23	- Plug suspected at Column I inlet
24	Tuesday, October 23, 1990 - Column I inlet plug confirmed - Unloaded ISS to UTBs - Started ISS heatup
25	- Plug has disappeared - Started UTB heatup
32	- Reloading Column H
33	- Loading Column I
36	- Started Loop flow - Adding H ₂ at 5.0 L/min for 40 minutes
37	- Stopped H ₂ addition after 200 L input - Added additional 60 L of H ₂
39	- Column I feed switched from bottom to middle
40	- GC sampling started
42	- Helium temperature dropped to 16.7 K
43	- Column H heater dropped to 17 watts
44	- Column H heater raised to 17.5 watts
45	- F-CLHA setpoint changed from 400 to 200 sccm - No flow change observed

Table A-1 (cont.) ISS EVENT LOG October 1990 Test Run

- 46 - GC sampling continues
- 47 - Column H heater dropped to 17 watts
- 48 Wednesday October 24, 1990
- 56 - F-CLHA problem determined to be a "stuck valve"
- 62 - Sample taking continues all day
- 63 - Set CLIA flow controller to 1.0 L/min
- 72 Thursday October 25, 1990
- 80 - ISS low level chamber valve #1 discovered closed indicating no
 flow has come from the top of Column H
 - Opened valve #1; radiation level went off-scale, then dropped
 from 8.7×10^6 to 3.6×10^5 . F-CLHS is now controlling; F-CLHA
 indicates 100 sccm, MMI indicated 143 sccm initially then
 dropped to 104 sccm
 - GC indicates H₂ and He at the top of Column H
- 81 - Set F-CLHA to 200 sccm. Controller is not controlling; replaced
 with controller earlier; appears to be working properly
 - Re-zeroed F-CLHA after shift; controller set to 100 sccm
 - Burped EVHA to UTB to remove tritium from dead-ended line
- 83 - Low level ion chamber reading dropping from 304 to 50
- 85 - Burped top of Column H to UTB and circulated gas via CLHD to
 remove stagnant gases around tritium monitor
- 87 - Column I flow controller was found turned off
- 88 - Increased helium temperature from 16.7 to 17.0 K
 - Increased helium temperature to 17.5 K
 - Increased helium temperature to 18.0 K
- 89 - Pumped out UTB manifold to receive ISS GAN gas
- 90 - Set CLHA flow to 200 sccm
 - Set Column H liquid level controller to 18 mm
 - Set Column I liquid level controller to 18 mm
 - Set Column I liquid level controller to 15 mm

Table A-1 (cont.) ISS EVENT LOG October 1990 Test Run

- 91 - Set Column H liquid level controller to 14 mm

- 94 - Loading D2 into Loop at 2.0 L/min
 - Increased CLIA flow to 1.5 L/min

- 96 Friday October 25, 1990

- 104 - Raised Column H and Column I pressures to get flow to Column D
 - Flushed out bottom of Column D to Column I

- 108 - Taking last sample from CLIC
 - Low level ion chamber bypassed
 - Started unloading of columns to UTB

Table A-2 Column I GC Analyses

CLIA-AN1				AN-1	
No:	30	33	46	59	73
Date:	24-Oct	24-Oct	25-Oct	25-Oct	26-Oct
Time:	3:58	21:08	11:50	19:44	2:01
He					
H2	1.209	1.399	0.918	0.625	0.038
HD	29.749	29.522	26.427	18.966	0.694
HT	6.321	6.109	5.099	3.474	0.147
D2	61.660	61.660	66.624	76.360	98.974
DT	1.061	1.310	0.928	0.575	0.094
T2			0.004		0.053
Total	100.000	100.000	100.000	100.000	100.000
CLI-S1				S1	S1
No:				54	60
Date:				25-Oct	25-Oct
Time:				16:35	20:03
He					
H2					
HD				0.540	0.665
HT					0.337
D2				99.460	98.572
DT					0.426
T2					
Total				100.000	100.000
CLIC-A1S				A1S	A1S
No:				55	61
Date:				25-Oct	25-Oct
Time:				17:02	20:24
He					
H2				0.260	
HD				6.657	1.033
HT				0.549	7.540
D2				82.942	90.499
DT				9.592	0.928
T2					
Total				100.000	100.000

Table A-2 (cont.) Column I GC Analyses

CLIC-A	A					FD-1	FD-1	FD-2
No:	31	32	45	56	62	75	90	91
Date:	24-Oct	24-Oct	25-Oct	25-Oct	25-Oct	26-Oct	26-Oct	26-Oct
Time:		20:16	11:10	17:21	20:38	2:36	10:53	11:13
He								
H2	0.357	0.063	0.193	0.118	0.026	0.213	0.265	0.417
HD	7.603	4.029	4.002	3.192	2.573	0.045	7.324	8.804
HT	1.752	0.812	0.777	0.441	0.514	0.013	1.564	2.203
D2	56.505	55.723	59.744	78.054	58.871	63.106	59.957	55.037
DT	29.745	35.456	31.381	16.386	32.292	32.406	27.694	28.394
T2	4.038	3.917	3.903	1.811	5.724	4.217	3.196	5.145
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000
CLIS1A	S1A					S1A		
No:				57	63	76		
Date:				25-Oct	25-Oct	25-Oct		
Time:				17:45	21:02	2:49		
He						0.011		
H2								
HD				0.173				
HT						0.022		
D2				99.827	77.645	78.793		
DT					20.812	21.174		
T2					1.543			
Total				100.000	100.000	100.000		
CLIB-AN2						AN2		
No:	29	34	47	58	64	77		
Date:	24-Oct	24-Oct	25-Oct	25-Oct	25-Oct	26-Oct		
Time:	13:16	21:58	12:35	17:59	21:16	3:24		
He								
H2								
HD				0.024				
HT				0.023	0.016			
D2	57.587	67.991	57.212	96.531	54.355	50.724		
DT	36.752	29.433	37.340	3.422	38.616	43.320		
T2	5.661	2.576	5.448		7.013	5.956		
Total	100.000	100.000	100.000	100.000	100.000	100.000		

Table A-3 Column H GC Analyses

CLHA-AN4	AN-4	AN-4	AN-4	AN-4
No:	65	78	86	88
Date:	25-Oct	26-Oct	26-Oct	26-Oct
Time:	21:45	3:30	5:41	9:46
He	0.352	0.076	0.006	0.035
H2		88.578	85.159	87.320
HD	99.603	11.298	14.046	11.849
HT	0.009	0.049	0.118	0.203
D2			0.625	0.594
DT	0.035		0.047	
T2				
Total	99.999	100.001	100.001	100.001
CLH-S2	S2	S2	S2	
No:	72	79	87	
Date:	25-Oct	26-Oct	26-Oct	
Time:	22:55	3:57	6:06	
He				
H2	23.501	25.641		
HD	65.149	47.239	0.738	
HT	0.024	0.104	0.037	
D2	11.276	27.004	98.832	
DT	0.050	0.012	0.394	
T2				
Total	100.000	100.000	100.001	
CLHC-AN3	AN-3	AN-3	AN-3	
No:	70	80	84	
Date:	25-Oct	26-Oct	26-Oct	
Time:	22:15	4:18	5:10	
He				
H2	1.283	0.008		
HD	17.884	0.417	0.099	
HT	3.790	0.006	0.004	
D2	76.242	98.522	99.273	
DT	0.801	1.047	0.624	
T2				
Total	100.000	100.000	100.000	

Table A-3 (cont.) Column H GC Analyses

CLHC-S2A	S2A	S2A	S2A
No:	71	81	85
Date:	25-Oct	26-Oct	26-Oct
Time:	22:27	4:27	5:35
He	0.003		
H2	21.302	13.080	0.221
HD	55.927	49.067	8.099
HT	0.121	0.140	0.075
D2	22.535	37.549	90.515
DT	0.111	0.164	0.230
T2	0.001		0.860
Total	100.000	100.000	100.000

CLHB-AN5	AN-5	AN-5
No:	66	82
Date:	25-Oct	26-Oct
Time:	21:52	4:38
He		
H2	0.033	
HD	15.536	
HT	0.162	
D2	83.292	97.566
DT	0.977	2.434
T2		
Total	100.000	100.000

Table A-4 Column D GC Analyses

CLDA-AN9	AN-9	AN-9
No:	83	89
Date:	26-Oct	26-Oct
Time:	5:15	9:59
He	0.008	
H2		
HD		8.443
HT	0.010	0.107
D2	98.544	91.450
DT	1.438	
T2		
Total	100.000	100.000

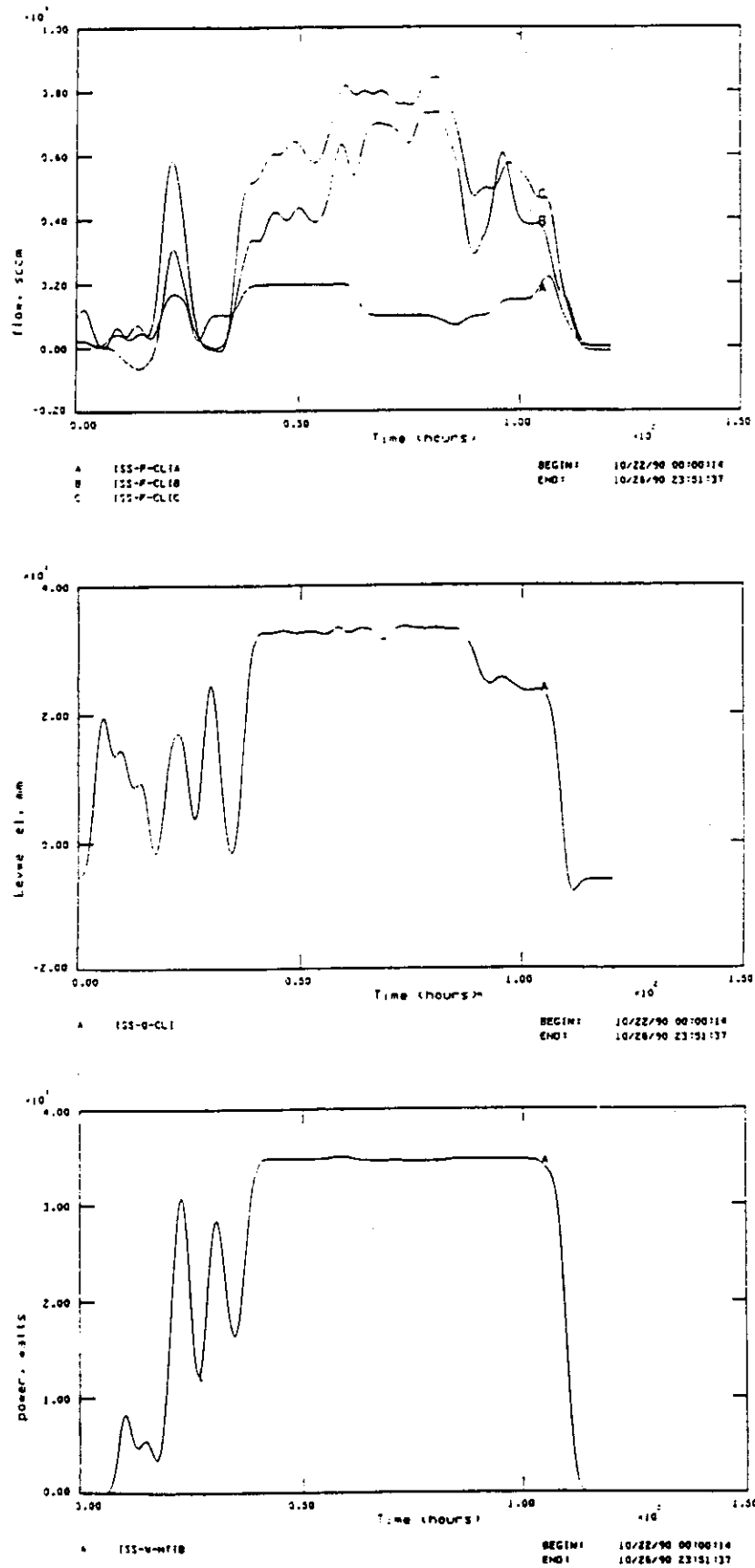


Fig. A-1 Column I Flows, Level and Heat Input

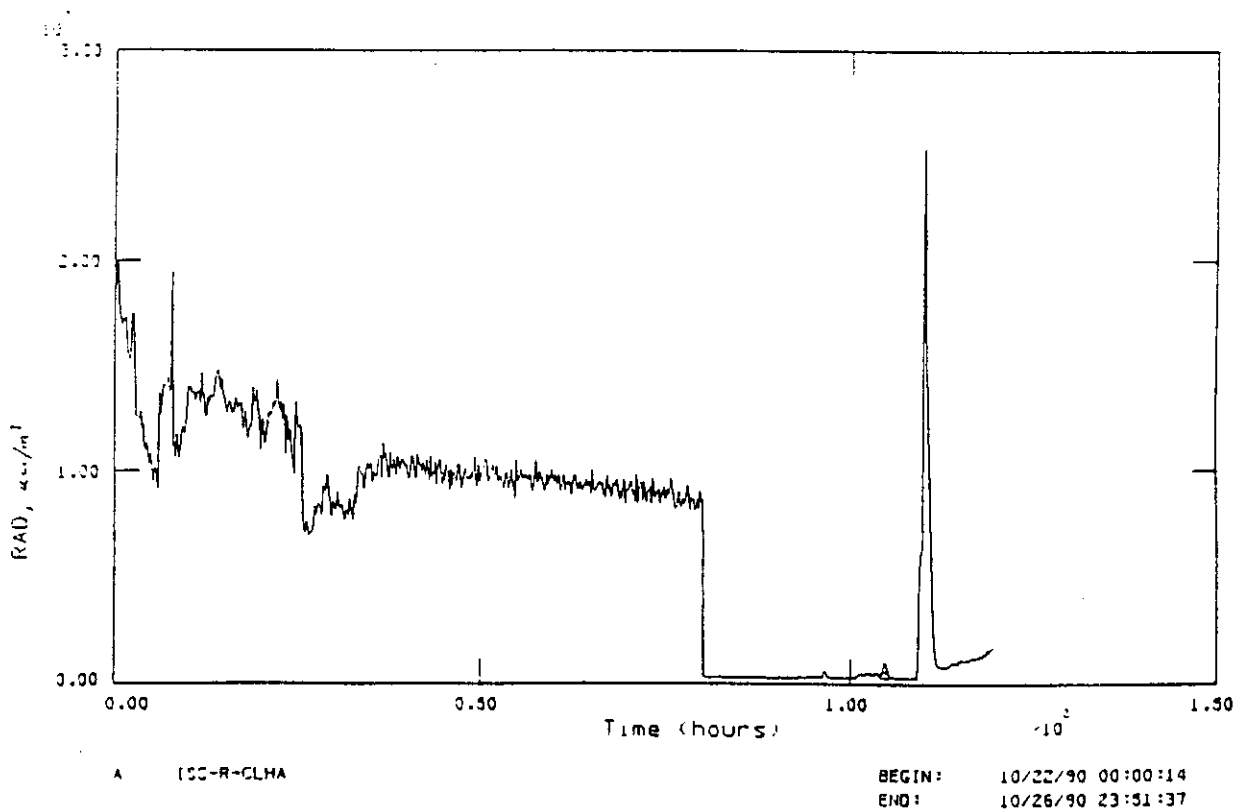
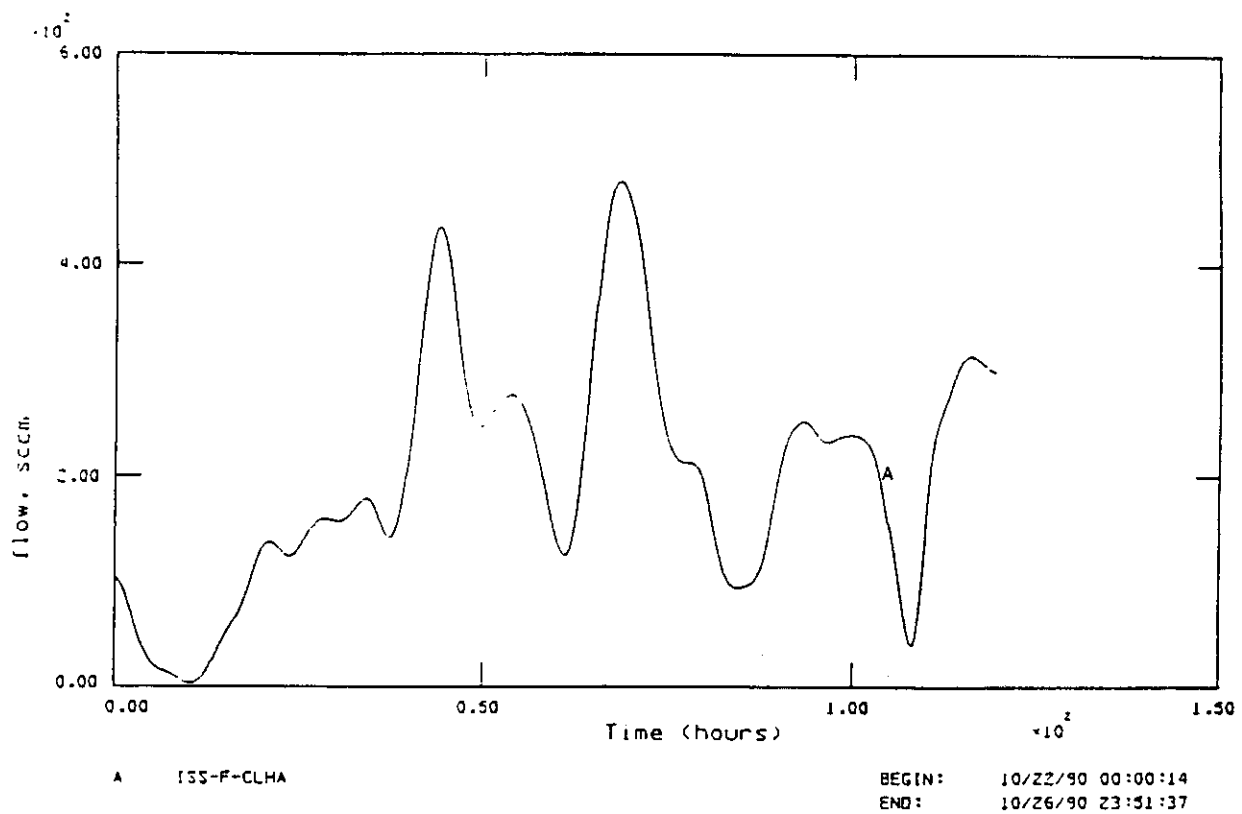


Fig. A-2 Column H Overhead Flow and Tritium Concentration

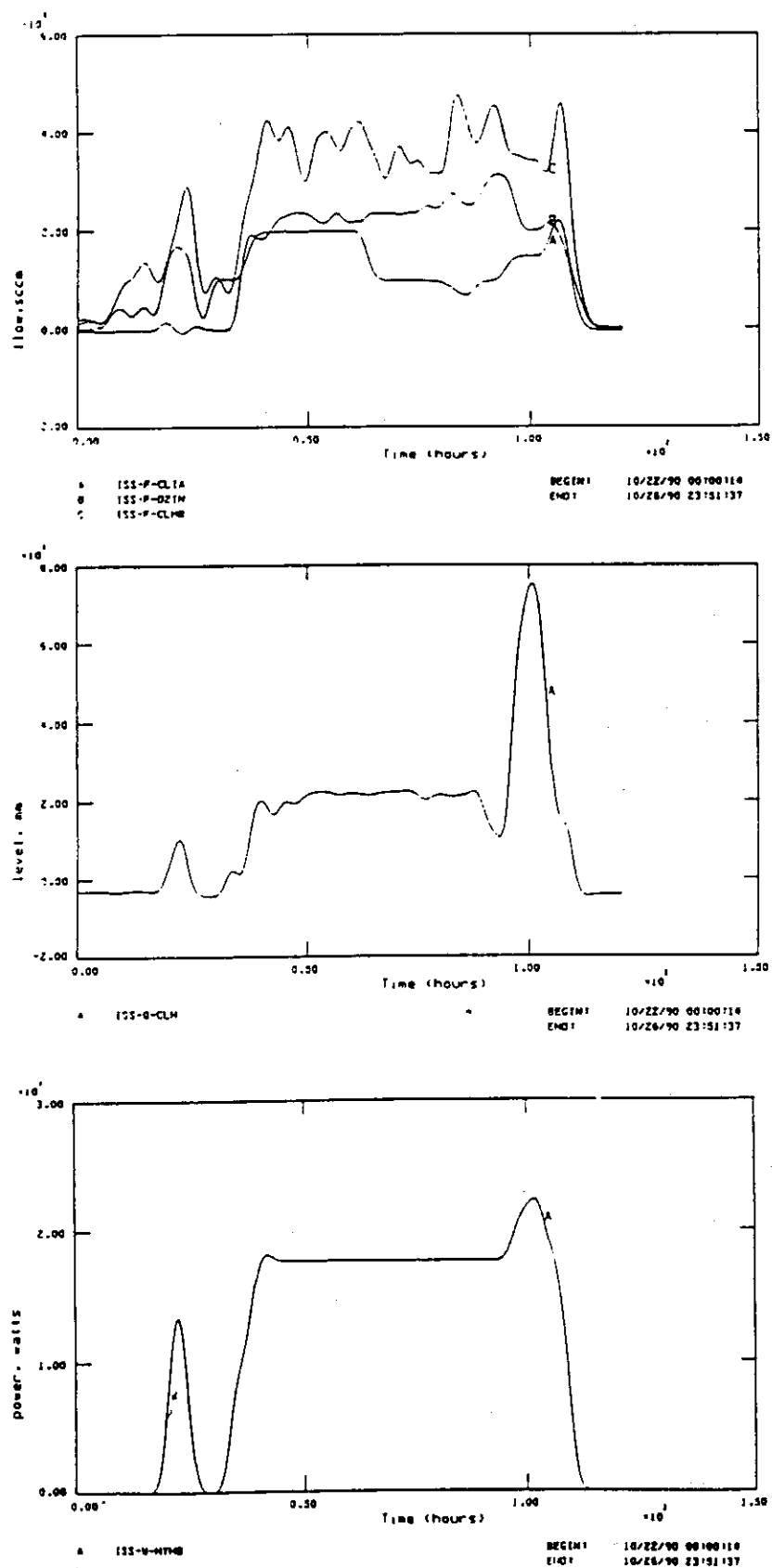


Fig. A-3 Column H Flows, Level and Heat Input

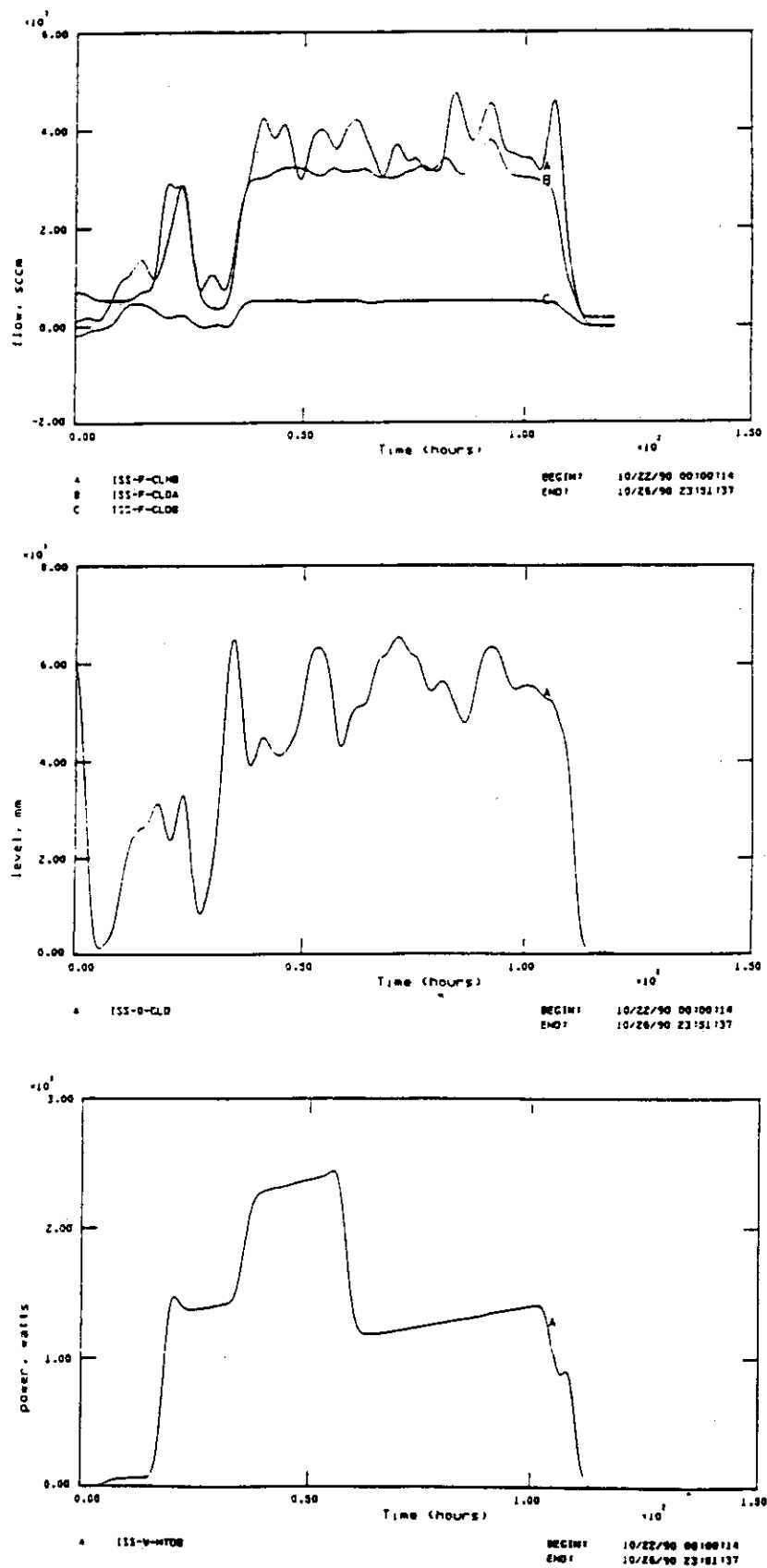


Fig. A-4 Column D Flows, Level and Heat Input