LIGO-E940021-05-B

* * * FACSIMILE MESSAGE * * *

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PAGE: 1 **OF:** 10

DATE: August 10, 1994

TO: Larry Jones
Caltech

FAX NO.: 818/304-9834

FROM: Warren A. Carpenter
Process Engineering Department
CBI Technical Services Co.

RE: COUPON TEST PROCEDURE
COUP-02
LIGO QUALIFICATION TEST FACILITY
930212 File # 2.2.5

Attached is revision 5 of the coupon outgassing test procedure COUP-02.

Regards,

Warren A. Carpenter
Senior Engineer

FAXED 1030

CBI PROPRIETARY		IDENTIFICATION COUP-02			
TITLE	COUPON OUTGASSING TEST PROCEDURE	REFERENCE NO. 930212		SHT 1 OF 9	
		OFFICE RCE		REVISION 5	
PRODUCT	LIGO BEAM TUBE MODULES QUALIFICATION TEST CALIFORNIA INSTITUTE OF TECHNOLOGY	MADE BY WAC	CHKD BY PM	MADE BY WAC	CHKD BY PM
		DATE 7/8/94	DATE 7/12/94	DATE 8/10/94	DATE 8/10/94

COUPON OUTGASSING TEST PROCEDURE

Single Chamber Configuration, Ambient Temperature Outgassing Test

RECORD KEEPING

All operating data taken, the time, the date, the coupon identification, all physical actions (such as opening or closing a valve) and all mental impressions, visual evidence or unusual occurrences (such as how fast the system is pumping down or that this batch of coupons seems to be more oxidized than usual) shall be recorded in the lab notebook and the appropriate computer. Operating data shall include pressures, piping, vessel and coupon temperatures, bake-out durations etc. All RGA data will be automatically recorded on the RGA computer.

PREPARATION

The coupon test system is assumed to have been previously conditioned to provide a low background outgassing rate. The system should be rebaked if a portion of the clean pumping system (except the chamber as a normal occurrence) has been opened to atmosphere. The system should also be leak checked if it has been in any way disassembled. The material samples have been cut into coupons which are 1" wide by 18" long.

The facility shall be inspected to ensure that the utilities are available. Breakers are turned on, water and air are available, nitrogen and helium bottles are available in sufficient quantities for the test.

CBI PROPRIETARY	IDENTIFICATION COUP-02			
TITLE: COUPON OUTGASSING TEST PROCEDURE	REFERENCE NO. 930212		SHT 2 OF 9	
	OFFICE RCE		REVISION 5	
PRODUCT: LIGO BEAM TUBE MODULES QUALIFICATION TEST CALIFORNIA INSTITUTE OF TECHNOLOGY	MADE BY WAC	CHKD BY PM	MADE BY WAC	CHKD BY PM
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Apparel

Test personnel shall wear, as a minimum, the following protective clothing:

1. Lab coat
2. Clean room gloves
3. Clean room hat
4. Clean room shoe covers

Cleaning

The first step of the preparation is to clean the 110 coupons required for each test. This shall be accomplished by following the cleaning procedure which has been developed for the coupon outgassing tests. The project cleaning procedure shall be strictly followed.

Warnings:

1. ***Failure to follow* the cleaning procedure may result in significant changes in the outgassing rate which will invalidate the results of the test.**
2. **After cleaning; the coupons shall not be touched directly by the hands; shall not be placed on a non-clean surface; and shall not be wiped with anything but a *clean, lint free, clean room quality* cloth (Rimple Cloth or Kim Wipes).**

The coupons shall then be loaded into the coupon chamber. The operator shall ensure that the permanent coupon (with the thermocouple) is located in the center of the chamber in order to represent a "worst temperature" location.

Chamber Sealing

Seal the coupon test chamber. Always use a new conflat gasket and remember to torque the bolts in a clockwise or counter clockwise sequence, not in an across pattern as wheel lug nuts would be installed. The bolts should be torqued approximately 1/4 turn per time and each bolt should be torqued at least four times.

CBI PROPRIETARY	IDENTIFICATION COUP-02			
TITLE: COUPON OUTGASSING TEST PROCEDURE	REFERENCE NO. 930212		SHT 3 OF 9	
	OFFICE RCE		REVISION 5	
PRODUCT: LIGO BEAM TUBE MODULES QUALIFICATION TEST CALIFORNIA INSTITUTE OF TECHNOLOGY	MADE BY WAC	CHKD BY PM	MADE BY WAC	CHKD BY PM
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SYSTEM PUMP-DOWN

Valve Alignment

The system valving shall be aligned as follows:

	Valve No	Position
• Chamber A isolation valve to the dirty pump system	V-1	closed
• Chamber A isolation valve to the clean pump system	V-2	closed
• Chamber re pressurization valve	V-3	closed
• Calibrated leak system isolation valve	V-4	closed
• Dirty system turbomolecular pump isolation valve	V-5	closed
• Dirty system roughing pump isolation valve	V-6	closed
• Hydrogen calibrated leak valve	V-7	closed
• RGA isolation valve	V-8	closed
• Cold trap vent valve	V-9	closed
• Clean system turbomolecular pump isolation valve	V-10	closed
• Clean system roughing pump isolation valve	V-11	closed

Roughing

Start the roughing pump and allow the pump to warm-up for five minutes for both the dirty and clean pumping systems (RP-3 & RP-4). The automatic viscous flow inbleed valves will open during this period when the pressure in the roughing lines reach 0.3 torr. After the warm-up period, open both of the pump systems roughing isolation valves (V-6 & V-11). Open the dirty system turbomolecular pump isolation valve (V-5). Open both of the chamber isolation valves (V-1 & V-2). Evacuate the system until the pirani gage pressure drops below 1 torr..

High Vacuum Pumping

Start both of the pump systems turbomolecular pumps (TMP-4 & 5). Allow the dirty pump system to evacuate the chamber and system for a minimum of 10 minutes after the turbomolecular pump indicates it is at full speed and then activate the Cold Cathode Gages. Cool down the LN2 Cold Trap to operating temperature. Slowly open the RGA Isolation valve (V-8). Allow the pump to evacuate the system to below 1 X 10^{-5} torr and activate the RGA in the faraday cup mode. Check the system for an air leak using the RGA and spraying with helium. Fix any leakage that is detected with the RGA and retest the leak location. Close the clean pumping system chamber isolation valve (V-2). Open the clean system turbomolecular pump isolation valve (V-10).

CBI PROPRIETARY

TITLE	COUPON OUTGASSING TEST PROCEDURE	IDENTIFICATION COUP-02			
		REFERENCE NO. 930212		SHT 4 OF 9	
		OFFICE RCE		REVISION 5	
PRODUCT	LIGO BEAM TUBE MODULES QUALIFICATION TEST CALIFORNIA INSTITUTE OF TECHNOLOGY	MADE BY WAC	CHKD BY PM	MADE BY WAC	CHKD BY PM
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BAKE-OUT

Activate the bake-out system for the chamber and the piping to the chamber isolation valves. Set the temperature controllers to 250° C. Initially, the pressure gage must be monitored to ensure that the system pressure does not rise above the operating range of the cold cathode gage and the turbomolecular pump (1×10^{-2} torr). If the pressure nears 1×10^{-2} torr, lower the setpoint of the chamber temperature controller to a point slightly below the current chamber temperature. Allow the chamber pressure to drop and then slowly increase the setpoint of the controller until it is set at 250° C. Continue the bake-out for 24 hours after the thermocouple inside the chamber reaches 250° C.

Chamber Cool Down

Turn the bake-out heater temperature controllers to off. Activate the water cooling system on the chamber by opening the water supply valve. Monitor the chamber and system temperatures until the temperature of the chamber shell is at 25° C. Throttle the cooling water inlet valve to maintain approximately 25° C while the coupons cool down at a slower rate. The cooling water valve may be turned off after the chamber shell reaches 25° C if it is unrealistic to throttle the water temperature due to availability of personnel, etc. Shut the chamber isolation valve to the dirty pump loop (V-1). Open the chamber isolation valve to the clean pump loop (V-2) and the calibrated leak manifold isolation valve (V-4). ~~Turn the bake-out heater temperature controllers to off.~~

OUTGASSING TEST

Outgassing Measurement

Close the chamber isolation valve to the clean pumping system (V-2) and start an accurate timer. Allow the chamber to accumulate the outgassing for one hour . Approximately five minutes prior to the end of the accumulation time, record the system pressure as indicated by the cold cathode gage and shut off the cold cathode gage. Start recording the RGA measurements just prior to the end of the accumulation time, shut the turbomolecular pump isolation valve to the clean pumping system (V-10). Exactly at the end of the chamber accumulation time, open the chamber isolation valve (V-2). Continue to monitor and record the RGA measurements for approximately 30 seconds.

The RGA will be set in the table mode recording mass number 2 at the minimum integration time period available. Immediately open the turbomolecular pump isolation valve (V-10) after the RGA measurement has been completed.

Record P_{st} which is the hydrogen partial pressure just after the pressure step.

RGA Calibration

Open the hydrogen calibrated leak valve (V-7) and start the cold cathode vacuum gage. Allow the system pressure to stabilize. Record the system pressure and shut off the cold cathode gage. Determine the calibration accumulation time from the rate of flow of the calibrated leak, the partial pressure measured for the system during the outgassing rate test and the measured volume of the system. The goal of the pressure calculation is to have the same partial pressure measured for the calibration test as for the coupon outgassing test. Close the hydrogen calibrated leak valve (V-7) and start the accumulation timer for the calculated time. Prior the end of the accumulation time, start recording the RGA measurements and shut the turbomolecular pump isolation valve (V-10). Exactly at the end of the chamber accumulation time, open the hydrogen calibrated leak valve (V-7). Continue to monitor and record the RGA measurements until the pressure in the system has jumped (measure for about 30 seconds). Immediately reopen V-10 the clean pump system isolation valve after the RGA measurement has been completed.

Record P_c which is the hydrogen partial pressure immediately after the pressure step.

Background Outgassing Measurement

If the system has not been operated sufficiently to be sure of the total system background outgassing rate, the entire test procedure shall be repeated with an empty chamber.

Record P_{sb} which is the accumulated pressure and P_c which is the calibration partial pressure

CBI PROPRIETARY

TITLE	COUPON OUTGASSING TEST PROCEDURE	IDENTIFICATION COUP-02			
		REFERENCE NO. 930212		SHT 6 OF 9	
		OFFICE RCE		REVISION 5	
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COMPLETE SYSTEM SHUTDOWN *IF REQUIRED*

Shut off the RGA . Close the chamber isolation valves (V-1 & V-2), close the hydrogen calibrated leak valve (V-7) and open the calibrated leak system isolation valve (V-4). Open the clean system turbomolecular pump isolation valve. Activate the system piping bake-out heaters and heat to a minimum temperature of 100° C. Shut off the cold cathode gage, close the calibrated leak system isolation valve (V-4), close the RGA isolation valve (V-8) and slightly open the cold trap purge gas valve (V-9) and allow nitrogen to sweep the vaporizing cold trap condensate out of the system. The vent valve should be opened sufficiently to allow the pressure increase above 0.3 torr. Set the turbomolecular pump controller to stand-by. Open the roughing pump ballast valve. Operate the system for a minimum of 12 hours with the bake-out and vent operating. Shut the turbomolecular pump isolation valves (V-5 & V-10), stop the turbomolecular pumps and stop the roughing pumps. Close the roughing pump ballast valves.

Sample Storage

The coupons shall be removed from the chamber and packaged in accordance with the sample packaging procedure. The sample bundle shall also be labeled to indicate the material heat and slab numbers, the test start date, the calculated outgassing rate and any other relevant information. Close the chamber.

OUTGASSING RATE CALCULATION

Nomenclature:

- Background outgassing flow rate (without coupons) Q_b (torr liters / sec)
- Total system outgassing flow rate (incl. coupons) Q_t (torr liters / sec)
- Coupon outgassing flow rate Q_c (torr liters / sec)
- Coupon outgassing rate(uncorrected) k_{cu} (torr liters / sec cm^2)
- Coupon outgassing rate(corrected) k_{cc} (torr liters / sec cm^2)
- System volume (including the chamber) V_s (liters)
- Outgassing accumulation time T_{ao} (sec.)
- Calibration accumulation time T_{ac} (sec.)
- System partial pressure after accumulation (W/ coupons) P_{st} (torr)
- System partial pressure after accumulation (background) P_{sb} (torr)
- Calibrated leak partial pressure after accumulation P_c (torr)
- Coupon surface area A_c
- Calibration factor - background CF_b
- Calibration factor - coupon CF_c
- Calibrated Leak Rate Q_x (torr liters / sec)

CBI PROPRIETARY	IDENTIFICATION: COUP-02			
TITLE: COUPON OUTGASSING TEST PROCEDURE	REFERENCE NO.: 930212		SHT 7 OF 9	
	OFFICE: RCE		REVISION: 5	
PRODUCT: LIGO BEAM TUBE MODULES QUALIFICATION TEST CALIFORNIA INSTITUTE OF TECHNOLOGY	MADE BY: WAC	CHKD BY: PM	MADE BY: WAC	CHKD BY: PM
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Note: the following procedure is used for the determination of hydrogen outgassing rate only

Calibration Correction Factor (background or coupon)

$CF_{b \text{ or } c}=(Q_X*T_{ac}/V_s)/(P_c)$

Background Outgassing Rate

$Q_b=P_{sb}*CF_b*V_s/T_{ao}$

Total System Outgassing Flow Rate

$Q_t=P_{st}*CF_c*V_s/T_{ao}$

Coupon Outgassing Flow Rate

$Q_c=Q_t-Q_b$

Coupon Outgassing Rate

$k_{cu}=Q_c/A_c$

MATERIAL SAMPLE ACCEPTANCE

The material will be accepatble if the coupon outgassing rate is calculated to be less than the outgassing acceptance value specified by Caltech. This value is currently 1×10^{-13} torr liters /cm^2 sec. Official notice of the acceptability, including calculated outgassing rate, shall be provided to the pipe manufacturer and Caltech as soon as possible after the completion of the test. Caltech shall confirm that the material shall be rejected.

CBI PROPRIETARY

TITLE	COUPON OUTGASSING TEST PROCEDURE	IDENTIFICATION: COUP-02			
		REFERENCE NO.: 930212		SHT 8 OF 9	
		OFFICE: RCE		REVISION: 5	
PRODUCT	LIGO BEAM TUBE MODULES QUALIFICATION TEST CALIFORNIA INSTITUTE OF TECHNOLOGY	MADE BY: WAC	CHKD BY: PM	MADE BY: WAC	CHKD BY: PM
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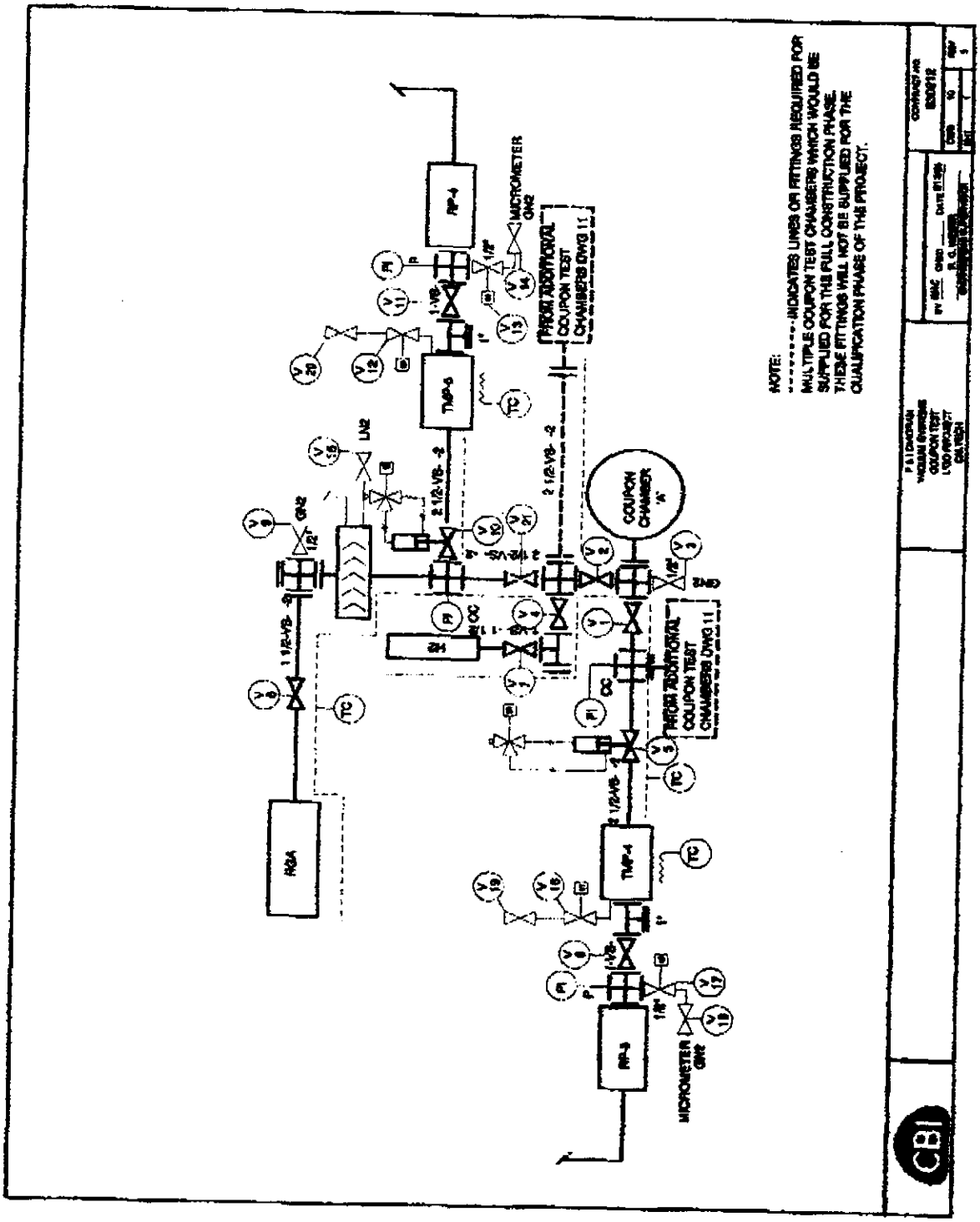

CBI PROPRIETARY

TITLE	COUPON OUTGASSING TEST PROCEDURE	IDENTIFICATION COUP-02			
		REFERENCE NO. 930212		SHT 9 OF 9	
		OFFICE RCE		REVISION 5	
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