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ISOBUTANE GAS SAMPLING
PROCEDURE REQUIRED FOR
ISOBUTANE ANALYSES

This procedure outlines the steps to be taken in order to draw samples of isobutane from commercial cylinders. For each cylinder, one sample is required in a one liter sample cylinder for outside analysis. An analysis must be completed and approved before a given cylinder of isobutane is certified for use.

Editorial Hand-Process Changes Other Than Spelling
Require PPD/CDF Operations Department Co-Head Approval

HPC Number	Date	Section Number	Initials
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____

Approval:

(PPD/CDF Operations Department Co-Head)

(Date)

1.0 Controlled Copies of This Procedure

Four controlled copies of this procedure will exist.

One at the CDF Department Office.

One on the CDF Web Page.

One on CDF ADMIN. Server.

One in the CDF Main Floor Cryo Control Room.

All other copies will be marked, "**INFORMATIONAL COPY ONLY**"

2.0 ISOBUTANE SAMPLING PROCEDURE

2.1 WARNINGS

Before beginning this procedure, the following safety precautions must be taken:

- ___ 1. The Gas Storage Shed is rated NEC Class I Div. 2. Be sure that there are no ignition sources in the area. The Gas Storage Shed also has a Fermilab Risk Class I assigned per the FESHM chapter 6020.3. The requirements for this classification must be adhered to.
- ___ 2. Be sure that combustibles and flammable materials are not present.
- ___ 3. Wear appropriate Personal Protective Equipment for this procedure.
- ___ 4. Only trained personnel are to be in the sampling area.
- ___ 5. The isobutane cylinder to be sampled shall not be stored in the Gas Storage Shed. Connect the cylinder to the sample station for the duration of this sampling procedure only. Promptly return cylinder to the CDF Storage Racks when the procedure is completed.

2.2 PREPARATION

- ___ 1. Record the following information:

_____	SCOTT GAS ISOBUTANE SERIAL NUMBER
_____	DATE/TIME SAMPLE IS TAKEN
_____	OUTDOOR TEMPERATURE
_____	NAME OF PERSON DRAWING THE SAMPLE

- ___ 2. Verify the cylinder contents:

- ___ Isobutane in Scott Gas Cylinder containing □ 35 lbs of product
- ___ Instrument Grade, 99.5% purity
- ___ Cylinder Size A with CGA 510 fitting
- ___ Certification by Scott Gas indicating Helium Content < 100 ppm

2.3 SAMPLING PROCESS STEPS

- ___ 1. Remove the isobutane cylinder (hereafter referred to as 'the cylinder') from the CDF Gas Storage Racks and connect it to the CV-2950/Flexhose assembly using the CGA-510/VCO fitting. Do not exceed the minimum bend radius of the flexhose. Be sure the O-ring is installed in the VCO fitting. Keep the cylinder valve closed.

- ___ 2. Install the cylinder heater, thermostat, and insulating blanket. The cylinder heater and blanket are stored on a shelf in the CDF main floor 'storage cage'. Check the electrical cord for damage. If damaged, do not use. Obtain another heater and continue. The cylinder heater should be installed at/near the bottom of the cylinder. Do not place the thermostat in direct contact with the heater. Do not apply power to the heater at this point.

- ___ 3. Install a one liter sample cylinder inside of the sampling enclosure. Verify that the O-rings are installed in the fittings and that they are in good condition.

- ___ 4. Verify the following nitrogen gas flow set points:
 - ___ Set FI-2962 to 0 SCFH by closing MV-2962;
 - ___ Verify that flow through FI-2963 is 25 SCFH by adjusting MV-2963.

- ___ 5. To begin, verify that the system valve positions are as follows:

___ Isobutane cylinder supply valve	CLOSED
___ MV-SAMP-IN	OPEN
___ MV-SAMP-OUT	OPEN
___ MV-2950	OPEN
___ MV-2960	CLOSED
___ MV-2961	CLOSED
___ MV-2964	CLOSED

- ___ 6. To verify the set points for FI-2950 and PRV-2950, follow these steps in the order listed:
 - ___ Open MV-2960
 - ___ Slowly Open MV-2961 to flow nitrogen gas through PRV-2950 to begin purging the supply line and sample cylinder.

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- ___ Verify a flow rate of 80 SCFH through FI-2950. MV-2961 should be open several turns. Adjust MV-2950 as well as PRV-2950 to achieve a stable flow. The pressure downstream of the regulator as read by PI-2951 should read slightly less than 20 psig when this flow rate is reached.
- ___ Check for leaks at the isobutane cylinder fitting connections.
- ___ Close MV-2960 and MV-2961. The sample cylinder should maintain approximately 5 psig of pressure (equal to the CV-2950 setting) over a five minute period. This may be monitored with PI-2951. If this pressure does not hold, double check that the O-rings on the sample cylinder fittings are installed and in good condition. It is important that the check valve seals as well. If the check valve is leaking, it must be repaired before continuing.
- ___ Close and secure the sampling enclosure door.
- ___ 7. Turn power on to warm the cylinder. Power is supplied via PP-B0-10 circuit 32. Note that this circuit provides power to the sampling enclosure heater as well. The enclosure temperature setting is preset by the factory. The cylinder temperature set point should be already set to 75 deg. F.
- ___ 8. Allow time for the cylinder and enclosure to warm up. The enclosure temperature may be read on TI-2950. Wait for the cylinder head to be warm to the touch and the enclosure temperature, TI-2950, to read \geq 70 deg. F.
- ___ 9. Open the cylinder supply valve to begin purging the sample line and sample cylinder with isobutane.
- ___ 10. In this step, five vent and fill cycles of the sample tubing will be completed as well as purging the sample cylinder. Check the flow rate of FI-2950. For a flow rate = 80 SCFH, the total purge time is equal to 15 minutes. The purging goal is to flow 20 cubic feet of gas from the isobutane cylinder. If the flow read by FI-2950 is not equal to 80 SCFH, calculate the total purge time using the following formula or refer to Table 1:

$$\frac{(20 \text{ ft}^3) \times (60 \text{ minutes} / \text{hour})}{(\text{Flow Rate, SCFH})} = \text{Total Purge Time Required, minutes}$$

If the outdoor temperature is too cold, use of an additional cylinder heater may be required or, a reduced flow rate is acceptable, i.e. flow at 60 SCFH for 20 minutes. If the flow meter is pegged, immediately close the cylinder supply valve and reduce the set points of FI-2950 using MV-2950 and/or PRV-2950. The purge rate should be set for 100 SCFH or less and the flow must be stable. Record the flow rate and purge time below.

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FI-2950 Flow Rate, scfh	Total Purge Time	Purge Time
100	12 min.	6 min.
90	13 min. 20 sec.	6 min. 25 sec.
80	15 min.	7 min. 30 sec.
70	17 min. 10 sec.	8 min. 20 sec.
60	20 min.	10 min.
50	24 min.	12 min.

Table 1. Sample Cylinder Required Purge Time

_____ Flow Rate

_____ Total Purge Time

- ___ 10a. After one half of the purge is complete; i.e. 10 cubic feet of isobutane has purged through the sample cylinder, stop the flow to the sample cylinder by fully closing the isobutane cylinder supply valve.
- ___ 10b. Perform five vent and fill cycles. Close MV-SAMP-IN for this portion of the procedure. The enclosure will need to be accessed. Note that the enclosure heater is powered. Keep the door closed during the vent and fill cycles so that the enclosure tubing and components remain warm. The pressure of the tubing may be monitored with PI-2951 during the vent and fill cycles.
- ___ i. Open MV-2964 to vent the supply line and then Close.
- ___ ii. Open the cylinder valve to fill the supply line and Close.
- ___ iii. Repeat 10b steps i. and ii. (2nd vent and fill)
- ___ iv. Repeat 10b steps i. and ii. (3rd vent and fill)
- ___ v. Repeat 10b steps i. and ii. (4th vent and fill)
- ___ vi. Repeat 10b steps i. and ii. (5th vent and fill)
- ___ 10c. Be sure MV-2964 is closed. Then, re-open MV-SAMP-IN. Close the enclosure door and secure. Wait for the enclosure temperature, TI-2950, to read ≥ 70 deg. F.
- ___ 10d. Open the isobutane cylinder supply valve and flow the remaining 10 cubic feet of isobutane through the sample cylinder. After the purging is complete, stop the flow to the sample cylinder by fully closing the isobutane cylinder supply valve.
- ___ 11. Allow time for the temperature of the sample cylinder to reach equilibrium with the enclosure. Be sure that no flow is detected on FI-2950 and that PI-2951 reads 5 PSIG. This pressure is the set point of CV-2951. If the outdoor temperature is below 32 deg. F, the sample cylinder should be left in the heated sample enclosure for one hour to be sure any liquid in the sample

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cylinder will have time to vaporize. In warmer weather, keep the cylinder in the enclosure for a minimum of 20 minutes. Be sure the sampling enclosure temperature is ≥ 70 deg. F as read by TI-2950.

- 12. Open the enclosure door and close MV-SAMP-IN and MV-SAMP-OUT. Do not remove or disconnect the sample cylinder at this point.
- 13. Turn off power to the cylinder heater and the sampling enclosure heater (using PP-B0-10 circuit 32).
- 14. Open MV-2964 and MV-2960. Then open MV-2961 to purge the sample line with nitrogen gas at 60 SCFH for 30 seconds.
- 15. Close the following valves in the order listed.
 - MV-2960
 - MV-2961
 - MV-2964
- 16. Remove the sample cylinder and plug/cap both the inlet and outlet ports of the sample cylinder and the tubing inside of the sampling enclosure. Be sure that the O-rings remain in place. Note that the sample line, inlet and outlet, will be slightly pressurized.
- 17. Close and secure the enclosure door. Turn on the enclosure purge at 5 SCFH as read on FI-2962 using MV-2962. Also, keep the flow on to the vent line at 25 SCFH as read on FI-2963.
- 18. Disconnect the CGA-510 fitting from the isobutane cylinder. Remove the CGA-510 from the CV-2950/Flexhose assembly at the VCO fitting. Do not disconnect CV-2950 from the flexhose. Plug the inlet to CV-2950 (check that the VCO O-ring remains in place) and secure the CV/Flexhose assembly. Cap the isobutane cylinder. Store the CGA-510/VCO fitting in the sampling enclosure.
- 19. Immediately return the isobutane cylinder to the Gas Storage Racks (outdoors). Store the cylinder heater and insulating blanket in the 'storage cage' on the designated shelf.
- 20. Notify the Operations Manager that the sample is ready for analysis. Appropriately tag the cylinder contents and note the isobutane cylinder serial number on the tag and the date the sample was drawn. Store the sample cylinder in Test Room 114 until it is ready to be delivered to the Shipping Department.

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3.0 CHECKLISTS

The checklists for this procedure are included in, and are integral to, the above Procedure section. A copy of this procedure will be kept in the First Floor Cryo Control Room in the CDF Assembly Building.

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4.0 DEVIATIONS

None are allowed.

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5.0 Required Training and Authorized Training Personnel

CDF Gas Systems Engineer
CDF Gas Systems Manager

The training should be documented on a standard Fermilab Training Form and the Training Expiration date should be tied to the end date of the Collider Run (e.g. "the end of Collider Run II"). The completed forms must be inserted in the CDF Department Office copy of this procedure.

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6.0 Training Materials

This procedure covering the operation of the isobutane sampling system must be read and understood.

One of the authorized training personnel must give a training lecture on the use of the isobutane sampling system in the Gas Storage Shed.

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7.0 List of Trained People for this Procedure

A list of trained personnel for this procedure should be kept in a separate section at the end of the CDF Department copy of the procedure.

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8.0 References and Supporting Documentation

Fermilab Piping and Instrumentation Drawing # 2563.372-MD-382228.