

## ETHANE GAS ANALYSIS PROCEDURE REQUIRED FOR ETHANE CERTIFICATION

This procedure outlines the steps to be taken to perform the Gas Analyses for detection of Oxygen, Hydrogen Sulfide, Ethylene, and Hydrochloric Acid as impurities in ethane. This analysis is required before a given trailer of ethane 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.	_____	_____	_____
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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 at the Test Room #114 Ethane Analysis Stand in the CDF Assembly Building.

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

## **2.0 ETHANE GAS ANALYSIS PROCEDURE**

### **2.1 WARNINGS:**

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

- a) Be sure that the Test Room Blower is running.
- b) Be sure that combustibles and flammable materials (alcohol, cardboard, etc.) are not present within 10 feet of the testing apparatus.
- c) Minimize ignition sources within 10 feet of the testing apparatus.
- d) Welding, Grinding, and Brazing are not permitted inside the test room while flammable gas is present.
- e) Be sure that the sample cylinder is warmed to room temperature before beginning the analyses.
- f) Wear Safety Glasses while performing the analyses.
- g) Keep doors to the test room closed.
- h) Wear gloves while handling the Draeger tubes.

### **2.2 TEST PROCEDURE:**

The ethane gas testing consists of six basic steps. The first is the preparation of the test. The second, third, fourth and fifth are an oxygen analysis, a hydrogen sulfide test, an ethylene test, and a hydrochloric acid test, respectively. The final step is the test completion. The following are the procedures required for these steps.

#### **A. Test Preparation**

- 1. Record the following information:
  - \_\_\_\_\_ TRAILER NUMBER
  - \_\_\_\_\_ DATE SAMPLE WAS TAKEN
  - \_\_\_\_\_ DATE OF ANALYSIS
  - \_\_\_\_\_ NAME OF PERSON PERFORMING THE ANALYSIS
- 2. Close MV-OUT, the sample cylinder outlet valve. MV-IN is already closed. Remove the 'Sampler Venting Attachment' from MV-OUT. Connect the Sample Cylinder Outlet (at MV-OUT) to the Analysis Stand Inlet at MV-A1.

- \_\_\_ 3. Verify that the Analysis Stand Manual Valves are in the following positions:
  - \_\_\_ MV-IN Closed
  - \_\_\_ MV-OUT Closed
  - \_\_\_ MV-A1 Open
  - \_\_\_ MV-A2 Closed
  - \_\_\_ MV-A3 Closed
  - \_\_\_ MV-A4 Closed
  - \_\_\_ MV-A5 Vent Position
  - \_\_\_ MV-A6 Closed
  - \_\_\_ MV-A7 Closed
  - \_\_\_ MV-A8 Open
  
- \_\_\_ 4. Open the Nitrogen Cylinder Supply Valve.
  
- \_\_\_ 5. Inert the Analysis Stand Tubing by following these steps:
  - \_\_\_ a) Open MV-A2 momentarily to pressurize the Analysis Stand tubing with the regulators set to their nominal values. Close MV-A2.
  - \_\_\_ b) Open MV-A4 to depressurize the tubing. The pressure may be monitored with PI-A3. Close MV-A4.
    - \_\_\_ Pressure Cycle #1 completed.
  
  - \_\_\_ c) Repeat steps II.5. a.& b. two more times.
    - \_\_\_ Pressure Cycle #2 completed.
    - \_\_\_ Pressure Cycle #3 completed.
  
- \_\_\_ 6. Close MV-A8.

## B. Oxygen Analysis

With the Neutronics Oxygen Analyzer attached to the Analysis Stand, complete the following steps:

- \_\_\_ 1. Open the manual valves in the order listed.
  - \_\_\_ MV-A2
  - \_\_\_ MV-A3
  - \_\_\_ Neutronics Inlet Valve
  - \_\_\_ Neutronics Outlet Valve
  - \_\_\_ MV-A9
  - \_\_\_ MV-A6
  
- \_\_\_ 2. Turn on the Neutronics PPM Oxygen display. The pump is not required. Set the flowrate to 0.3 scfh with FI-A2 (MV-A9). The flow may be fine tuned with the Sample Inlet Valve. Continue to flow nitrogen through the analyzer until the readout is zero. Allow a minimum of one hour for the Neutronics to warm up and zero. The display should be within 10 ppm of zero before continuing.
  
- \_\_\_ 3. When the Neutronics Analyzer readout is Zero, Close MV-A2.
  
- \_\_\_ 4. When PI-A3 indicates that the tubing is depressurized, Perform the Oxygen Analysis:
  - \_\_\_ a) To flow ethane, Open MV-OUT on the sample cylinder and begin the TIMER.
  - \_\_\_ b) Verify that the flowrate is 0.3 scfh on FI-A2.
  - \_\_\_ c) When the TIMER = 15 minutes, Record the Oxygen Readout. The CDF specification requires < 50 PPM O<sub>2</sub> in Ethane.  
  
\_\_\_\_\_ PPM Oxygen.
  
- \_\_\_ 5. The Neutronics PPM Oxygen display may be left in the ON position, but valves MV-A3 and MV-A6 must be closed at this time.

### C. Draeger Tube Testing

#### \_\_ 1. HYDROGEN SULFIDE TESTING

- \_\_ a) Break off both tips of the H<sub>2</sub>S Draeger tube in the tube opener.
- \_\_ b) Insert each end of the Draeger tube slightly into the ends of the flexible vinyl tubing. The arrow on the Draeger tube must point towards the exhaust.
- \_\_ c) Set MV-A5 to the Draeger Testing position.
- \_\_ d) Open MV-A7.
- \_\_ e) Open MV-A4. Slowly open MV-A8 to achieve flow through the Draeger Tube at a rate of 250 sccm as read on FI-A1. Begin the TIMER.
- \_\_ f) Stop the flow (Close MV-A4 and MV-A8) at TIMER = 4 minutes.
- \_\_ g) Record the level of H<sub>2</sub>S. Read the entire length of discoloration. Color change is white to brown. Tube range is 5 to 60 PPM H<sub>2</sub>S. The CDF specification requires < 10 PPM H<sub>2</sub>S in Ethane.  
  
\_\_\_\_\_ PPM H<sub>2</sub>S
- \_\_ h) Set MV-A5 to the Vent Position and Close MV-A7.
- \_\_ i) Remove the Draeger Tube and dispose of it.

\_\_ 2. ETHYLENE TESTING

- \_\_ a) Break off both tips of the C<sub>2</sub>H<sub>4</sub> Draeger tube in the tube opener.
- \_\_ b) Insert each end of the Draeger tube slightly into the ends of the flexible vinyl tubing. The arrow on the Draeger tube must point towards the exhaust.
- \_\_ c) Set MV-A5 to the Draeger Testing position.
- \_\_ d) Open MV-A7.
- \_\_ e) Open MV-A4. Slowly open MV-A8 to achieve flow through the Draeger Tube at a rate of 50 sccm as read on FI-A1. Begin the TIMER.
- \_\_ f) Stop the flow (Close MV-A4 and MV-A8) at TIMER = 6 minutes.
- \_\_ g) Record the level of C<sub>2</sub>H<sub>4</sub>. Read the entire length of discoloration. Color change is yellow to blue. Tube range is 50 to 2500 PPM C<sub>2</sub>H<sub>4</sub>. The CDF specification requires < 1000 PPM C<sub>2</sub>H<sub>4</sub> in Ethane.  
  
\_\_\_\_\_ PPM C<sub>2</sub>H<sub>4</sub>
- \_\_ h) Set MV-A5 to the Vent Position and Close MV-A7.
- \_\_ i) Remove the Draeger Tube and dispose of it.

\_\_ 3. HYDROCHLORIC ACID TESTING

- \_\_ a) Break off both tips of the HCl Draeger tube in the tube opener.
- \_\_ b) Insert each end of the Draeger tube slightly into the ends of the flexible vinyl tubing. The arrow on the Draeger tube must point towards the exhaust.
- \_\_ c) Set MV-A5 to the Draeger Testing position.
- \_\_ d) Open MV-A7.
- \_\_ e) Open MV-A4. Slowly open MV-A8 to achieve flow through the Draeger Tube at a rate of 500 sccm as read on FI-A1. Begin the TIMER.
- \_\_ f) Stop the flow (Close MV-A4 and MV-A8) at TIMER = 2 minutes.
- \_\_ g) Record the level of HCl. Read the entire length of discoloration. Color change is blue to yellow. Tube range is 1 to 10 PPM HCl. The CDF specification requires < 10 PPM HCl in Ethane.  
  
\_\_\_\_\_ PPM HCl
- \_\_ h) Set MV-A5 to the Vent Position and Close MV-A7.
- \_\_ i) Remove the Draeger Tube and dispose of it.

#### D. TEST COMPLETION

- \_\_\_ 1. When testing is complete, the tubing and sample cylinder must be inerted. Set the manual valves in the position indicated:
- |     |        |               |
|-----|--------|---------------|
| ___ | MV-IN  | Closed        |
| ___ | MV-OUT | Open          |
| ___ | MV-A1  | Open          |
| ___ | MV-A2  | Closed        |
| ___ | MV-A3  | Closed        |
| ___ | MV-A4  | Closed        |
| ___ | MV-A5  | Vent Position |
| ___ | MV-A6  | Closed        |
| ___ | MV-A7  | Closed        |
- \_\_\_ 2. Open MV-A4 and MV-A8 and wait until PI-A3 indicates that the tubing and sample cylinder are depressurized. Close MV-A4.
- \_\_\_ 3. Inert the tubing and sample cylinder with the following steps:
- \_\_\_ a) Open MV-A2 until PI-A3 reads 15 psig. Close MV-A2.
- \_\_\_ b) Open MV-A4 to relieve the pressure. Close MV-A4.
- \_\_\_ Pressure Cycle #1 completed.
- \_\_\_ c) Repeat steps V.3. a.& b. two more times.
- \_\_\_ Pressure Cycle #2 completed.
- \_\_\_ Pressure Cycle #3 completed.
- \_\_\_ 4. Close MV-OUT, MV-A1, and MV-A8.
- \_\_\_ 5. Open MV-A2, MV-A3, and MV-A6.
- \_\_\_ 6. With the Neutronics PPM Oxygen display turned ON, set the Neutronics flowrate to 0.3 scfh with FI-A2 (MV-A9). Continue to flow nitrogen through the analyzer until the readout is less than 100 ppm O<sub>2</sub>.

- \_\_\_ 7. When the Neutronics Analyzer readout is near Zero (<100 ppm), set the following manual valves in the order listed:
- |     |                         |        |
|-----|-------------------------|--------|
| ___ | MV-A6                   | Closed |
| ___ | MV-A9                   | Closed |
| ___ | Neutronics Outlet Valve | Closed |
| ___ | Neutronics Inlet Valve  | Closed |
| ___ | MV-A3                   | Closed |
| ___ | MV-A2                   | Closed |
- \_\_\_ 8. Turn off the Neutronics PPM Oxygen display. Close the Nitrogen Cylinder Supply Valve.
- \_\_\_ 9. Disconnect the sample cylinder from the Analysis Stand. Plug/Cap the ends of the cylinder and the inlet to the Analysis Stand at MV-A1.
- \_\_\_ 10. If no other source of flammable gas is present in the Test Room, the Test Room Blower may be turned off.

### **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 a binder by the Ethane Analysis Stand.

**4.0 DEVIATIONS**

None are allowed.

## **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.

## **6.0 Training Materials**

This procedure covering the operation of the oxygen analyzer and the Draeger Tube tests must be read and understood.

One of the authorized training personnel must give a training lecture on the use of the oxygen analyzer and the Draeger Tube test at the analysis stand.

**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.

## **8.0 References and Supporting Documentation**

For a layout of the Ethane Analysis Stand, see Fermilab drawing # 2563.327–MC405089, which is posted on the wall above the Ethane Analysis Stand.