

Volume IV
Chapter 56

STATE OF MONTANA
AIR QUALITY CONTROL
IMPLEMENTATION PLAN

Subject: ~~Yellowstone County~~
Air Pollution
Control Program

56.9.4.1 EXHIBIT A - EMISSION LIMITATIONS AND OTHER CONDITIONS -
CENEX HARVEST STATES COOPERATIVES' PETROLEUM REFINERY,
LAUREL, MONTANA

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EXHIBIT A

EMISSION LIMITATIONS AND OTHER CONDITIONS

CENEX HARVEST STATES COOPERATIVES' Petroleum Refinery
Laurel, Montana

SECTION 1. AFFECTED FACILITIES

(A) Plant Location:

CENEX HARVEST STATES COOPERATIVES ("Cenex"), formerly Cenex, Inc., is located just south of Interstate 90 at Laurel, Montana. The plant is located in Yellowstone County, Township 2 South, Range 24 East, SE¼ Section 16.

(B) Affected Equipment and Facilities:

- (1) Main crude heater
- (2) FCC Regenerator/CO Boiler
- (3) #3 boiler
- (4) #4 boiler
- (5) #5 boiler
- (6) Old SRU tail gas oxidizer
- (7) HDS complex SRU
- (8) HDS complex fuel gas fired units: H-101 heater, H-201 heater, H-202 heater, SRU reheater E-407, and incinerator INC-401;
- (9) Pre-1990 fuel gas fired units: FCC CO boiler, Alkylation unit oil heater, crude preheater, platform heater-4 sections, #1 naphtha unit charge heater, #1 naphtha unit stripper heater, naphtha unit splitter heater, MDU stripper heater, MDU charge heater, PDA asphalt heater, #2 N.U. heater-2, platformer debutanizer heater, #2 crude heater, #2 vacuum heater, #1 vacuum heater, FCC preheater, #9 boiler, saturated gas concentration hot oil heater, asphalt loading heaters (2), tank BP-2 heater, tank 11 heater, and the fuel can heaters (2).
- (10) #10 Boiler

(C) Nonaffected Equipment and Facilities:

Any equipment or facilities which have no effect on the nature or quantity of emissions of sulfur-bearing gases including, but not limited to, combustion equipment which is fueled exclusively with natural gas or propane.

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SECTION 2. DEFINITIONS

(A) The following definitions apply throughout this Stipulation and Exhibit A.

- (1) "Annual Emissions" means the amount of SO₂ emitted in a calendar year, expressed in pounds per year rounded to the nearest pound.

Where:

$$[\text{Annual Emissions}] = \Sigma [\text{Daily Emissions}]$$

- (2) "Attachment #1" means the "Performance Specifications for Stack Flow Rate Monitors, Fuel Oil Flow Meters, and Fuel Oil Sulfur Analysis", attached to this Exhibit and incorporated herein by reference.
- (3) "Attachment #2" means the "Analytical Methods for Analyzing the Sour Water Stripper Overheads for Hydrogen Sulfide and Precision and Accuracy Methods for the Sour Water Stripper Flow Meter," attached to this Exhibit and incorporated herein by reference.
- (4) "Calendar Day" means a 24-hour period starting at 12:00 midnight and ending at 12:00 midnight, 24 hours later.
- (5) "Clock Hour" means one twenty-fourth (1/24) of a Calendar Day and refers to any of the standard 60-minute periods in a day which are generally identified and separated on a clock by the whole numbers one through twelve.
- (6) "Continuous Emission Monitoring System (CEMS)" means all equipment necessary to obtain an Hourly SO₂ Emission Rate, provided each SO₂ concentration, stack gas volumetric flow rate, fuel oil flowmeter, fuel gas flow rate and sour water flow rate monitor is designed to achieve a temporal sampling resolution of at least one concentration or flow rate measurement per minute and each hydrogen sulfide concentration monitor is designed to achieve a temporal sampling resolution of at least one concentration measurement per three minutes. Such equipment includes:
- (a) a continuous emission monitor (CEM) which determines SO₂ concentration in a stack gas, a continuous stack gas volumetric flow rate monitor which determines stack gas flow rate, and associated data acquisition equipment;
 - (b) a continuous fuel gas monitor which determines hydrogen sulfide (H₂S) concentration in the fuel gas in the refinery section, a fuel gas flow rate monitor that determines the combined fuel gas firing rate for the fuel gas combustion units listed in Section 1 (B)(9)

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and (10), and associated data acquisition equipment; and

a continuous fuel gas monitor which determines H₂S concentration in the fuel gas in the HDS complex, a fuel gas flow rate monitor that determines the combined fuel gas firing rate for the fuel gas combustion units listed in Section 1 (B)(8) and associated data acquisition equipment;

(c) a pair of fuel oil flow meters which in combination measure the combined fuel oil firing rate for fuel oil combustion units, and associated data acquisition equipment; or

(d) a continuous sour water flow rate monitor which determines the sour water flow rate to the "old" sour water stripper tower and associated data acquisition equipment.

(7) "Daily Emissions" means the amount of SO₂ emitted in a Calendar Day, expressed in pounds per day rounded to the nearest pound.

Where:

$$[\text{Daily Emissions}] = \Sigma [\text{Three Hour Emissions}]$$

Each Calendar Day is comprised of eight non-overlapping 3-hour periods. The Three Hour Emissions from all of the 3-hour periods in a Calendar Day shall be used to determine that day's emissions.

(8) "Hourly Average" means an arithmetic average of all Valid and complete 15-minute data blocks in a Clock Hour. Four (4) Valid and complete 15-minute data blocks are required to determine an Hourly Average for each monitor and source per Clock Hour.

Exclusive of the above definition, an Hourly Average may be determined with two (2) Valid and complete 15-minute data blocks, for two of the 24 hours in any Calendar Day.

A complete 15-minute data block for each sulfur dioxide continuous emission monitor, stack gas flow rate monitor, fuel oil flowmeter, hydrogen sulfide concentration monitor, sour water flow rate monitor, and fuel gas flow rate monitor, shall have a minimum of one (1) data point value; however, each monitor shall be operated such that all Valid data points acquired in any 15-minute block shall be used to determine that 15-minute block's reported concentration and flow rate.

(9) "Hourly SO₂ Emission Rate" means the pounds per Clock Hour of SO₂ emissions from a source (stack, fuel oil system, sour water system, or fuel gas system) determined using Hourly Averages and rounded to the nearest tenth of a pound.

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(a) For stack systems, SO₂ concentrations shall be measured in parts per million (PPM) on either a wet or dry basis.

(i) If the SO₂ concentration is measured on a wet basis, Cenex shall calculate the Hourly SO₂ Emission Rate using the following equation:

$$E_{ST} = K * C_H * Q_H$$

Where:

E_{ST} = Hourly SO₂ Emission Rate in pounds per hour and rounded to the nearest tenth of a pound;
K = 1.663 X 10⁻⁷ in (pounds/SCF)/PPM;
 C_H = Hourly Average SO₂ concentration in PPM; and
 Q_H = stack gas Hourly Average volumetric flow rate, measured on an actual wet basis, converted to Standard Conditions, and reported in standard cubic feet per hour (SCFH).

(ii) If the SO₂ concentration is measured on a dry basis, Cenex shall either install, operate, and maintain a continuous moisture monitor for measuring and recording the moisture content of the stack gases or determine the moisture content of the stack gases continuously (or on an hourly basis) and correct the measured hourly volumetric stack gas flow rates for moisture. Cenex shall calculate the Hourly SO₂ Emission Rate using the following equation:

$$E_{ST} = K * C_H * Q_H * \frac{(100 - \%H_2O)}{100}$$

Where:

E_{ST} = Hourly SO₂ Emission Rate in pounds per hour and rounded to the nearest tenth of a pound;
K = 1.663 X 10⁻⁷ in (pounds/SCF)/PPM;
 C_H = Hourly Average SO₂ concentration in PPM (dry basis);
 Q_H = stack gas Hourly Average volumetric flow rate, measured on an actual wet basis, converted to Standard Conditions, and reported in standard cubic feet per hour (SCFH); and
 $\%H_2O$ = Hourly Average stack gas moisture content, in percent by volume.

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(b) For refinery fuel gas systems:

- (i) H₂S concentrations are measured on an actual wet basis in PPM;
- (ii) the combustion unit fuel gas firing rates shall be measured on an actual wet basis in standard cubic feet per hour (SCFH); and
- (iii) the Hourly SO₂ Emission Rate shall be calculated using the following equation:

$$E_{FG} = K[(C_{HDS} * Q_{HDS}) + (C_R * Q_R)]$$

Where:

- E_{FG} = Hourly SO₂ Emission Rate in pounds per hour and rounded to the nearest tenth of a pound;
- K = 1.688 X 10⁻⁷ in (pounds/SCF)/PPM;
- C_{HDS} = Hourly Average fuel gas H₂S concentration in PPM from the H₂S CEM located in the HDS complex
- Q_{HDS} = Combined Hourly Average fuel gas firing rate expressed in SCFH from the fuel gas flow rate monitor located downstream and adjacent to the amine unit fuel gas absorber in the HDS complex;
- C_R = Hourly Average fuel gas H₂S concentration in PPM from the H₂S CEM located in the refinery section; and
- Q_R = Combined Hourly Average fuel gas firing rate expressed in SCFH from the fuel gas flow rate monitor located downstream and adjacent to the Zone A amine unit fuel gas absorber.

- (iv) When the H₂S concentration in the fuel gas exceeds the upper range of the Zone A CEMS required by Section 6(B)(3), a Surrogate H₂S Concentration "S_R" shall be determined in accordance with the H₂S sampling and analytical requirements of Section 6(B)(3) and substituted for the CEMS-derived concentration "C_R" in the equation presented in Section 2(9)(b)(iii) above.

The Surrogate H₂S Concentration "S_R" determined in accordance with Section 6(B)(3) shall not be used to satisfy the QDRR requirements.

- (c) For fuel oil combustion with mass flow metering the following equation shall be used to calculate the Hourly SO₂ Emission Rate in pounds per hour:

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$$E_{FO} = 2.0 * M_o * \%S_o / 100$$

Where:

E_{FO} = Hourly SO₂ Emission Rate in pounds per hour and rounded to the nearest tenth of a pound;

M_o = mass of fuel oil consumed per hour in pounds per hour;

$\%S_o$ = percentage of sulfur by weight measured in the fuel oil; and
2.0 = ratio of pounds of SO₂ per pound of sulfur.

- (d) For the sour water stripper overheads (SWSOH) contribution to SO₂ emissions from the main crude heater stack or the flare:
- (i) the H₂S concentrations shall be determined in accordance with Attachment #2 (or another method approved by the Department and EPA) and expressed in milligrams per liter;
 - (ii) sour water flow rate shall be expressed in gallons per hour; and
 - (iii) the Hourly SO₂ Emission Rate shall be calculated using the following equation:

$$E_{SWS} = K * C_H * Q_H$$

Where:

E_{SWS} = Hourly SO₂ Emission Rate from the burning of the sour water stripper overheads in the main crude heater or the flare in pounds per hour rounded to the nearest tenth of a pound;
 K = 1.57 X 10⁻³ in [(pounds-liters)/(gallons-milligrams)];
 C_H = H₂S concentration in the sour water in milligrams per liter; and
 Q_H = sour water flow rate to the "old" sour water stripper tower in gallons per hour.

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- (e) The total Hourly SO₂ Emission Rate (E_T) for fuel oil combustion in the combined combustion units (#3, #4, and #5 boiler stacks, main crude heater stack), fuel gas combustion in the fuel gas-fired units [listed in Section 1 (B)(8), (9) and (10)], and for SWSOH burning in the main crude heater is calculated by the following equation: $E_T = E_{FO} + E_{FG} + E_{SWS}$

Where, for a given hour:

- E_T = total Hourly SO₂ Emission Rate for the combined sources in pounds per hour and rounded to the nearest tenth of a pound;
- E_{FO} = Hourly SO₂ Emission Rate for fuel oil combustion calculated using the equation in Section 2 (A)(9)(c) and rounded to the nearest tenth of a pound;
- E_{FG} = combined Hourly SO₂ Emission Rate (E_{FG}) for the fuel gas combustion units listed in Section 1 (B)(8) (9) and (10) calculated using the equation in Section 2 (A)(9)(b) and rounded to the nearest tenth of a pound; and
- E_{SWS} = Hourly SO₂ Emission Rate from burning of the sour water stripper overheads in main crude heater calculated using the equation in Section 2(A)(9)(d) in pounds per hour rounded to the nearest tenth of a pound.

- (10) "Operating" means whenever an affected facility is starting up, shutting down, using fuel, or processing materials, and SO₂ emissions are expected from the source or stack, except that:
- (a) for the FCC Regenerator/CO Boiler stack, start-up and shutting down shall only include time periods when gas-oil feedstock is being delivered to the FCC;
- (b) for the old SRU tail gas oxidizer stack and the HDS Complex SRU stack, start-up and shutting down shall only include time periods when sulfur-bearing gases are being delivered to the associated SRU; and
- (c) for the purpose of determining the Quarterly Data Recovery Rate for the sour water flow rate CEMS, Operating shall only include those periods when sour water stripper overheads are burned in the main crude heater or the flare and exhausted up the main crude heater stack or the flare.
- (11) "Quarterly Data Recovery Rate" (QDRR) means the percentage of hours in a calendar quarter when CEMS-derived Hourly SO₂ Emission Rate data are available for a source (stack, fuel oil combustion system, sour water system, or fuel gas system) in comparison to the number of corresponding Operating hours for that source.

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If Cenex demonstrates, through the CEMS manufacturer's specifications and stack measurements, that stack conditions during certain periods of startup or shutdown in the FCC Regenerator/CO boiler stack are beyond the design capabilities of the CEMS, then such periods shall not be considered Operating hours for determination of the QDRR.

The QDRR for a source shall be calculated in accordance with the following equation:

$$\text{QDRR} = \frac{\text{VH}}{\text{OH}} * 100\%$$

Where:

VH = number of hours of Hourly SO₂ Emission Rate data that are also source Operating hours in a calendar quarter;

OH = total number of source Operating hours in a calendar quarter; and

QDRR = Quarterly Data Recovery Rate.

(12) "Standard Conditions":

(a) means 20.0°C (293.2°K, 527.7°R, or 68.0 °F) and 1 atmosphere pressure (29.92" Hg) for stack gas emission calculations using the equation/method in Section 2(A)(9)(a); and

(b) means 15.6°C (288.7°K, 520.0°R, or 60.3 °F) and 1 atmosphere pressure (29.92" Hg) for refinery fuel gas emission calculations using the equation/method in Section 2(A)(9)(b).

(13) Surrogate H₂S Concentration" means an H₂S concentration expressed in parts per million (PPM) and used for purposes of demonstrating compliance with the SO₂ emission limits in Section 3(A)(1)(d) when the H₂S concentration in the Zone A fuel gas system exceeds the upper range of the CEMS required by Section 6(B)(3). The Surrogate H₂S Concentration shall be determined in accordance with the sampling and analytical requirements of Section 6(B)(3).

(14) "Three Hour Emissions" means the amount of SO₂ emitted in each of the eight non-overlapping three hour periods in a Calendar Day, expressed in pounds and rounded to the nearest pound.

Where:

$$[\text{Three Hour Emissions}] = \Sigma [\text{Hourly SO}_2 \text{ Emission Rates}]$$

Whenever Hourly SO₂ Emission Rates are unavailable and the facility is not Operating, zero

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pounds per hour shall be substituted for the missing Hourly SO₂ Emission Rates.

- (15) "Valid" means data that is obtained from a monitor or meter serving as a component of a CEMS which meets the applicable specifications, operating requirements, and quality assurance and control requirements of Section 6.

SECTION 3. EMISSION LIMITATIONS AND FACILITY MODIFICATIONS

(A) Emission limitations

(1) Affected Sources:

- ✓ (a) FCC Regenerator/CO boiler stack;
 - (i) Three Hour Emissions of SO₂ from the FCC Regenerator/CO boiler stack shall not exceed 2,142.3 pounds per three hour period,
 - (ii) Daily Emissions of SO₂ from the FCC Regenerator/CO boiler stack shall not exceed 17,138.4 pounds per Calendar Day, and
 - (iii) Annual Emissions of SO₂ from the FCC Regenerator/CO boiler stack shall not exceed 6,255,516 pounds per calendar year.
- ✓ (b) Old SRU tail gas oxidizer stack;
 - (i) Three Hour Emissions of SO₂ from the old SRU tail gas oxidizer stack shall not exceed 2,916.3 pounds per three hour period,
 - (ii) Daily Emissions of SO₂ from the old SRU tail gas oxidizer stack shall not exceed 23,330.4 pounds per Calendar Day, and
 - (iii) Annual Emissions of SO₂ from the old SRU tail gas oxidizer stack shall not exceed 8,515,596 pounds per calendar year.
- ✓ (c) HDS Complex SRU stack;
 - (i) Three Hour Emissions of SO₂ from the HDS complex SRU stack shall not exceed 42.6 pounds per three hour period,
 - (ii) Daily Emissions of SO₂ from the HDS complex SRU stack shall not

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- exceed 340.8 pounds per Calendar Day, and
- (iii) Annual Emissions of SO₂ from the HDS complex SRU stack shall not exceed 124,392 pounds per calendar year.
 - (d) Combustion sources (#3, #4, and #5 boiler stacks, and main crude heater stack) and the fuel gas-fired units [listed in Section 1 (B)(8), (9) and (10)];
 - (i) Combined Three Hour Emissions of SO₂ from the combustion sources (#3, #4, and #5 boiler stacks, and main crude heater stack), the fuel gas fired sources [listed in Section 1 (B)(8), (9) and (10)], and the combustion of sour water stripper overhead gases in the main crude heater shall not exceed 3,014.7 pounds per three hour period,
 - (ii) Combined Daily Emissions of SO₂ from the combustion sources (#3, #4, and #5 boiler stacks, and main crude heater stack), the fuel gas fired sources [listed in Section 1 (B)(8), (9) and (10)], and the combustion of sour water stripper overhead gases in the main crude heater shall not exceed 24,117.6 pounds per Calendar Day, and
 - (iii) Combined Annual Emissions of SO₂ from the combustion sources (#3, #4, and #5 boiler stacks, and main crude heater stack), the fuel gas fired sources [listed in Section 1 (B)(8), (9) and (10)], and the combustion of sour water stripper overhead gases in the main crude heater shall not exceed 8,802,924 pounds per calendar year.
 - (e) Other Minor Sources;
 - (i) Cenex shall utilize appropriate maintenance, repair, and operating practices to control emissions of sulfur bearing gases from minor sources such as ducts, stacks, valves, vents, vessels, and flanges which are not otherwise subject to this Stipulation and Exhibit A.
 - (ii) Cenex shall use good engineering judgement and appropriate engineering calculations to quantify emissions from activities that are not otherwise addressed by this Stipulation and Exhibit A but are known to contribute to emissions from sources listed in Section 1(B). In addition, Cenex shall account for such emissions in determining compliance with all applicable emission limits contained in Section 3.

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(B) Facility Modifications

- (1) By November 1, 1997, Cenex shall remove the fuel oil guns from the crude preheater unit, alkylation oil heater, and MDU charge heater and install a blind insert at the fuel oil header to the unit.
- (2) Beginning January 1, 1998, Cenex shall burn the sour water stripper overheads from the "old" sour water stripper (SWS) in the FCC CO boiler and exhaust those emissions through the FCC Regenerator/CO boiler stack, except that those sour water stripper overheads may be burned in the main crude heater (and exhausted through the main crude heater stack) ~~or in the flare~~ during periods when the FCC CO boiler is unable to burn the sour water stripper overheads from the "old" SWS, provided that such periods do not exceed 55 days per calendar year and 65 days for any two consecutive calendar years.
- (3) By June 1, 1998, Cenex shall install a chain and lock on the valve which supplies sour water stripper overheads from the "old" SWS to the main crude heater or the flare to insure that the valve cannot be opened (sour water stripper overheads to the main crude heater or the flare) unless the chain and lock are removed. When sour water stripper overheads are no longer being burned in the main crude heater or the flare the valve shall once again be chained and locked. Cenex shall log each date and time that the valve is opened and closed.

SECTION 4. COMPLIANCE DETERMINATIONS

- (A) Compliance with the emission limitations contained in Section 3 (A)(1)(a) through (c) shall be determined using data from the CEMS required by Section 6 (B)(1) and (2) and in accordance with the appropriate equation(s) in Section 2(A)(1), (7), (9), and (14) except when CEMS data is not available as provided in Section 2(A)(14). Although the CEMS data is the method of demonstrating compliance on a continuous basis, the data from the testing required by Sections 5(A) or 6(C) and (D) shall also be used to demonstrate compliance.
- (B) Compliance with the emission limitations contained in Section 3 (A)(1)(d) shall be determined by summing the Hourly SO₂ Emission Rates for fuel oil combustion, fuel gas combustion, and SWSOH burning in the main crude heater and using the result to calculate the corresponding emission rate for each of the averaging periods (for which an emission limit in Section 3(A)(1)(d) applies) in accordance with the equations in Section 2(A)(1), (7), (9), and (14). The Hourly SO₂ Emission Rate for fuel oil combustion shall be determined by using the total hourly mass of fuel oil consumed as measured by the fuel oil flowmeters and fuel oil sulfur content determined in accordance with Section 6(F). The Hourly SO₂ Emission Rate for the fuel gas combustion units shall be determined by using the H₂S concentrations and fuel gas flow rates measured by the CEMs required by Section 6 (B)(3) and (4) and the sampling required by Section 6(B)(3).

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The Hourly SO₂ Emission Rate for the burning of SWSOH in the main crude heater shall be determined by using the H₂S concentration in the SWS feed stream determined in accordance with sampling required by Section 4(D) and the sour water flow rate measured by the CEMS required by Section 6(B)(6).

All calculations shall be made in accordance with the appropriate equation(s) in Section 2(A)(1), (7), (9), and (14), except when CEMS data is not available as provided in Section 2(A)(14).

- (C) By November 1, 1997, Cenex shall certify for the Department that the facility modifications described in Section 3(B)(1) have been completed and are permanent in nature. Similarly, by July 1, 1998, Cenex shall certify for the Department that the facility modifications described in Section 3(B)(2) and (3) have been completed and are permanent in nature.
- (D) Whenever sour water stripper overheads are being burned in the main crude heater (and exhausted through the main crude heater stack) ~~or in the flare~~, compliance with the emission limitations contained in Section 3(A)(1)(d) shall be determined using flow rate monitoring data from the CEMS required by Section 6(B)(6) and from sampling and analysis of the sour water feed to the "old" sour water stripper tower. Except for the first two hours after sour water stripper overheads are directed to the main crude heater ~~or the flare~~, Cenex shall collect at least one sample from the sour water feed to the "old" sour water stripper tower for each of the eight nonoverlapping three hour periods in a Calendar Day. In addition, the time elapsed before collection of the first sample shall not exceed four hours. Cenex shall analyze the sample for H₂S in accordance with the procedures contained in Attachment #2 (or another method approved by the Department and EPA) and Cenex shall use the results to calculate the Hourly SO₂ Emission Rate for each of the hours in the three hour period in accordance with the equations in Section 2(A)(9). Such emission rate shall be counted against the emission limitations contained in Section 3 (A)(1)(d).
- (E) Compliance with the facility modifications contained in Section 3(B) shall be determined through inspection by the Department.
- (F) Compliance with the Quarterly Data Recovery Rate requirements.
 - (1) Compliance with the Quarterly Data Recovery Rate requirements contained in Section 6(A)(2) shall be determined in accordance with Section 2 (A)(11), with no exceptions for out-of-specification data or monitor downtime, except as provided in Section 6(A)(2).
 - (2) For quarters in which Operating hours are reduced (short quarters), a determination of whether Cenex has violated the minimum Quarterly Data Recovery Rate (QDRR) requirements in Section 6(A)(2)(b) shall include consideration of whether the reduced Operating hours made compliance with Section 6(A)(2)(b) unreasonable.
 - (3) Upon determination that the CEMS is not functioning properly, Cenex shall implement short-term

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corrective measures and if necessary, long term corrective measures to accomplish, as expeditiously as practicable either:

- (a) correction of the failure; or
- (b) development, installation (if necessary), testing, maintenance, and operation of a new CEMS or appropriate replacement portions of the affected CEMS.

SECTION 5. EMISSION TESTING

- (A) In order to accurately determine the sulfur dioxide emission rates in pounds per hour for the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, and HDS complex SRU stack, Cenex shall perform annual source testing using EPA-approved methods (40 CFR Part 60, Appendix A, Methods 1-4 and 6/6c as appropriate for this Stipulation and Exhibit A) or an equivalent method approved by the Department and EPA, and in accordance with the Montana Source Testing Protocol (ARM 17.8.106). The annual Relative Accuracy Test Audits (RATAs) required by Sections 6(C) and (D) may substitute for the annual source tests provided that the flow rate RATA and the concentration RATA are performed simultaneously and additional calculations are made to determine and report the data in pounds per hour of sulfur dioxide.
- (B) In order to accurately determine the hydrogen sulfide concentration in parts per million for the HDS complex fuel gas-fired units and the pre-1990 fuel gas-fired units, Cenex shall perform annual source testing using EPA-approved methods (40 CFR Part 60, Appendix A, Method 11) or an equivalent method approved by the Department and EPA, and in accordance with the Montana Source Testing Protocol (ARM 17.8.106).
- (C) Cenex shall notify the Department in writing of each annual source test a minimum of twenty-five (25) working days prior to the actual testing (unless otherwise specified by the Department).

SECTION 6. CONTINUOUS MONITORING AND FUEL OIL FLOWMETERING

- (A) CEM Quarterly Data Recovery Rates
 - (1) "Unusual Circumstances" means circumstances which are unforeseeable, beyond Cenex's control, and which could not reasonably have been prevented or mitigated by Cenex. Such circumstances may include but are not limited to earthquakes, power outages, or fire, but do not include failures of any monitoring or metering equipment or associated data acquisition equipment unless such failures meet the following conditions:
 - (a) prior to the failure, the equipment was installed, operated, and maintained in accordance

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with the requirements of Section 6;

- (b) upon failure, Cenex initiates the short term corrective measures and, as necessary, the long term corrective measures required by Section 4 (F)(3);
- (c) within two working days of occurrence, Cenex notifies the Department's Permitting and Compliance Division by telephone of the occurrence of Unusual Circumstances, as defined herein; and
- (d) Cenex demonstrates, by utilizing properly signed contemporaneous CEMS operating logs and other relevant evidence, in the first quarterly report following the failure that the failure meets the above conditions.

✓ (2) Quarterly Data Recovery Rates

- (a) Notwithstanding the QDRR requirements specified in Section 6(A)(2)(b), whenever a source or stack is Operating, Cenex shall use best efforts to operate the associated CEMS in a manner to achieve the highest Quarterly Data Recovery Rate (QDRR) that is technically feasible.
- (b) At a minimum, Cenex shall achieve the following QDRR requirements, unless prevented by Unusual Circumstances or by reduced Operating hours as provided in Section 4(F)(2):
 - (i) for the FCC Regenerator/CO boiler stack CEMS, old SRU tail gas oxidizer stack CEMS, and HDS complex SRU stack CEMS, Cenex shall achieve a QDRR for each CEMS of equal to or greater than 90%;
 - (ii) for the Fuel Oil Combustion Unit (#3, #4 and #5 boilers and main crude heater) CEMS, Cenex shall achieve a QDRR for the Fuel Oil Combustion Unit CEMS of equal to or greater than 94%;
 - (iii) for the Fuel Gas Combustion Unit CEMS:
 - (I) if the Three Hour Emissions from the fuel gas combustion units never exceed 300 pounds at any time during a calendar quarter or if the only exceedances are caused by Malfunctions, Cenex shall achieve a QDRR for each pair of H₂S concentration and fuel gas flow rate monitors of equal to or greater than 90%; or
 - (II) if the Three Hour Emissions from the fuel gas combustion units

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exceed 300 pounds at any time during a calendar quarter and one or more of the exceedances are not caused by a Malfunction, Cenex shall achieve a QDRR for each pair of H₂S concentration and fuel gas flow rate monitors of equal to or greater than 94%; and

(iv) for the sour water system CEMS (measures sour water flow rate to the "old" sour water stripper tower), Cenex shall achieve a QDRR of equal to or greater than 94%.

(c) In its evaluation of whether Cenex used best efforts to achieve the highest QDRR technically feasible, the Department will consider:

(i) the design capabilities of the CEMS, including a demonstration made by Cenex (using manufacturer's specifications and stack measurements), that stack conditions during certain periods of startup or shutdown in the FCC Regenerator/CO boiler stack are beyond the design capabilities of the CEMS; and whether:

(ii) Cenex has properly operated and maintained the CEMS, including the maintenance of an adequate spare parts inventory;

(iii) Cenex has complied with the quality assurance requirements described in Section 6;

(iv) Cenex has taken timely and appropriate action to correct a failure in the CEMS; and

(v) Unusual Circumstances have occurred, as defined in Section 6 (A)(1).

(d) Any time that a CEMS, including the associated data acquisition system, is not functioning properly, Cenex shall implement the short term corrective measures, and, if necessary, the long term corrective measures required by Section 4 (F)(3).

(B) Affected Sources

(1) By July 1, 1997, Cenex shall install, operate, and maintain continuous emission monitors to measure SO₂ from the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, and HDS complex SRU stack.

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- (2) By July 1, 1997, Cenex shall install, operate, and maintain continuous stack flow rate monitors to measure the stack gas flow rates from the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, and HDS complex SRU stack.
 - ✓(3) By January 1, 1997, Cenex shall install, operate, and maintain hydrogen sulfide (H₂S) concentration monitors downstream but adjacent to the Zone A amine unit fuel gas absorber for the refinery fuel gas system in the refinery section and downstream but adjacent to the amine unit fuel gas absorber for the refinery fuel gas system in the HDS complex. Within 4 hours of the initial determination that the H₂S concentration in the fuel gas stream has exceeded the upper range of the Zone A CEMS required by this paragraph, Cenex shall initiate sampling of the Zone A fuel gas stream on a once-per-three-hour-period frequency using the Tutwiler method (40 CFR 60.648) or another method approved by the Department and EPA to determine the H₂S concentration.
 - ✓(4) By January 1, 1997, Cenex shall install, operate, and maintain continuous fuel gas flow rate monitors downstream but adjacent to the Zone A amine unit fuel gas absorber for the refinery fuel gas system in the refinery section and downstream but adjacent to the amine unit fuel gas absorber for the refinery fuel gas system in the HDS complex.
 - ✓(5) By March 1, 1997, Cenex shall install, operate, and maintain two in-line fuel oil flowmeters on the fuel loop, one immediately before the fuel oil tank in use and one before the first fuel oil loop in use.
 - ✓(6) Before SWSOH may be burned in the main crude heater or the refinery flare, Cenex shall install, operate and maintain a continuous flow rate monitor to determine the sour water flow rate to the "old" sour water stripper tower.
- (C) CEM Performance Specifications
- ✓(1) All continuous SO₂ concentration monitors and hydrogen sulfide concentration monitors required by this control plan shall:
 - (a) be installed, certified (on a concentration basis), and operated in accordance with the performance specifications in 40 CFR Part 60, Appendix B, Performance Specification 2 and 7; and
 - (b) be subject to and meet the quality assurance and quality control requirements (on a concentration basis) of 40 CFR Part 60 Appendix F including but not limited to:
 - (i) daily calibration drift checks (zero/span or Z/S) using either electro-optical methods or certified calibration gas (however, in addition to the

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requirements of Appendix F at least one Z/S per calendar week must be conducted using a certified calibration gas),

- (ii) quarterly Cylinder Gas Audits (CGA) or Relative Accuracy Audits (RAA), and
- (iii) the annual Relative Accuracy Test Audit (RATA).

- (2) Cenex shall notify the Department in writing of each Relative Accuracy Test Audit a minimum of twenty-five (25) working days prior to the actual testing (unless otherwise specified by the Department).

✓(D) Stack Gas Flow Rate Monitor Performance Specifications

- (1) All continuous stack gas flow rate monitors required by this control plan shall:
 - (a) be installed, certified (on a flow rate basis), and operated in accordance with Department Method A-1 of Attachment #1; and
 - (b) be subject to and meet (on a flow rate basis) the quality assurance and quality control requirements of Department Method B-1 of Attachment #1.
- (2) Cenex shall notify the Department in writing of each Relative Accuracy Test Audit a minimum of twenty-five (25) working days prior to the actual testing (unless otherwise specified by the Department).

(E) Refinery fuel gas flow rate monitor accuracy determinations shall be required at least once every 48 months or more frequently as routine refinery turn-arounds allow. Accuracy determinations for the sour water flow rate monitor shall be conducted every 48 months and within three months prior to any scheduled shutdown of the FCC CO Boiler which coincides with the scheduled operation of the "old" sour water stripper. Accuracy determinations for the sour water flow rate monitor shall be conducted in accordance with Attachment #2 or another method approved by the Department and EPA.

✓(F) Fuel Oil Flow metering and Analysis Specifications

- (1) Cenex shall operate and maintain all fuel oil flowmeters required by this control plan in accordance with Method C-1 of Attachment #1.
- (2) Cenex shall conduct daily fuel oil sampling in accordance with Method C-1 of Attachment #1.

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- (3) Cenex shall analyze all fuel oil samples collected, as required by Section 6 (F)(2), for sulfur content in accordance with Method C-1 of Attachment #1.
- (4) Each fuel oil flowmeter required by this control plan shall demonstrate a flowmeter accuracy of 2.0 percent of the upper range value (i.e. maximum calibrated oil flow rate) as measured under laboratory conditions by the manufacturer or by the owner or operator, and pursuant to the calibration procedures as specified by Method C-1 of Attachment #1.
- (5) Cenex shall archive a split (at least 200 cc) of each fuel oil sample collected, as required by Section 6 (F)(2), in accordance with Method C-1 of Attachment #1.

SECTION 7. DATA REPORTING

- (A) Cenex shall submit quarterly reports on a calendar year basis, beginning with the first calendar quarter of 1998. The quarterly reports shall be submitted within 30 days of the end of each calendar quarter. The quarterly reports shall be submitted to the Department's Permitting and Compliance Division office in Helena and the Billings Regional Office. The quarterly report format shall consist of both a comprehensive electronic-magnetic report and a written or hard copy data summary report.
- (B) The electronic report format and records structure shall require hourly CEMS data, hourly mass of fuel oil consumed data, daily fuel oil sulfur content, stack temperature, and calibration data to be submitted to the Department as required in Section 7(A). The data shall be submitted to the Department on magnetic or optical media compatible with the Department's existing data management system. The data shall be submitted to the Department on magnetic or optical media, and such submittal shall follow the reporting format specified by the Department in 1996, as may be subsequently amended. The Department shall reserve the right to call for any necessary future revisions to the reporting format delineated in this Section.
 - (1) The electronic report shall contain the following:
 - (a) Hourly Average SO₂ concentrations in PPM from the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, and HDS complex SRU stack;
 - (b) Hourly Average stack volumetric flow rates in SCFH from the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, and HDS complex SRU stack;
 - (c) Hourly Average stack gas temperature in degrees Fahrenheit from the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, and HDS complex SRU stack;

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- (d) Hourly SO₂ Emission Rates in pounds per Clock Hour from the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, HDS complex SRU stack, and a combined Hourly SO₂ Emission Rate from fuel oil combustion, fuel gas fired units, and burning of sour water stripper overheads in the main crude heater;
 - (e) Hourly Average refinery section and HDS complex fuel gas system H₂S concentrations in PPM;
 - (f) Hourly Average refinery section and HDS complex fuel gas systems fuel gas flow rates in SCFH;
 - (g) total hourly mass of fuel oil consumed in pounds per Clock Hour;
 - (h) Hourly Average SWS flow rate in gal/hr whenever SWSOH are being burned in the main crude heater or the refinery flare; and
 - (i) daily calibration data from CEMS required by Section 6(B)(1) through (4).
- (2) In addition to submitting the electronic-magnetic quarterly reports to the Department, Cenex shall also record, organize and archive for at least five years the same data, and upon request by the Department, Cenex shall provide the Department with any data archived in accordance with this Section.
- (C) The quarterly written report shall consist of summarized Calendar Day CEMS data, Three Hour Emissions, fuel oil flowmeter data, Quarterly Data Recovery Rates and text regarding excess emissions. The quarterly written report shall identify each time period when the sour water stripper overheads were burned in the main crude heater (and exhausted through the main crude heater stack) or in the refinery flare.
- (1) The following data shall be recorded, organized, reported, and archived for a minimum of five years:
- (a) Three Hour Emissions of SO₂ in pounds per three hour period from the FCC Regenerator/CO boiler stack; old SRU tail gas oxidizer stack; HDS complex SRU stack; and combined Three Hour Emissions from the fuel gas fired units, fuel oil combustion, and burning of sour water stripper overheads in the main crude heater;
 - (b) Daily Emissions of SO₂ in pounds per Calendar Day from the FCC Regenerator/CO boiler stack; old SRU tail gas oxidizer stack; HDS complex SRU stack; and combined Daily Emissions from fuel oil combustion, the fuel gas fired units, and burning of sour water stripper overheads in the main crude heater;

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- (c) daily fuel oil sulfur content in percent sulfur by weight;
 - (d) the Quarterly Data Recovery Rate for each CEMS required by Section 6 (B)(1) through (6) expressed in percent;
 - (e) the Operating hours during the calendar quarter for the source or units associated with the FCC Regenerator/CO boiler stack, old SRU tail gas oxidizer stack, HDS complex SRU stack, main crude heater stack (when sour water stripper overheads are being burned in the main crude heater or the refinery flare), and the combined fuel oil and fuel gas combustion units;
 - (f) the date and time identifying each period of continuous monitoring system downtime during the reporting period, including quality control and quality assurance checks, and the nature of system repairs or adjustments;
 - (g) the results of the quarterly CGAs or RAAs and flow rate checks, the annual source tests required by Section 5 (A) and (B), and the annual RATAs required in Section 6(C) and (D);
 - (h) any documentation which demonstrates that a CEMS failure meets the conditions of Unusual Circumstances; and
 - (i) the dates and times that sour water stripper overheads are diverted from the FCC CO Boiler to the main crude heater or the refinery flare, the reasons for the diversions, and corrective actions taken, as appropriate, to avoid future recurrence.
- (2) For each Calendar Day on which any emission limitations are exceeded, the written report shall identify the source or unit with excess emissions and include the following information in a report submittal as specified in Section 7(A):
- (a) total hours of operation with excess emissions, the Hourly SO₂ Emission Rates, and Three Hour Emissions;
 - (b) all information regarding reasons for operating with excess emissions; and
 - (c) corrective actions taken to mitigate excess emissions.
- (D) Upon request from a representative of the Department, EPA or Yellowstone County Air Pollution Control, Cenex shall provide Hourly SO₂ Emissions Rate data for any prior day not covered by the latest quarterly report for the sources or units covered by this control plan and listed in Section 1(B).

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- (E) By January 1, 2000, the Department shall reevaluate the reporting requirements of this Section and determine if revisions are necessary or desirable. The purpose of the reevaluation is to determine if the reporting requirements should be modified to more closely meet the informational needs of the Department and the public, and to reduce or simplify the requirements for Cenex while still providing the necessary information. Any revisions shall be made only after consultation with Cenex, consideration of the number and type of data requests made by the public, and the Department's emission inventory and compliance needs.

SECTION 8. ADDITIONAL REQUIREMENTS AND CONDITIONS

Except as otherwise provided herein, nothing in this Stipulation, Exhibit A, or Attachments shall be construed to alter Cenex's obligations under any other applicable state, federal and local laws and regulations, orders, and permit conditions. In any enforcement proceeding pertaining to such other requirements, Cenex reserves the right to raise any and all available equitable or legal defenses.

SECTION 9. GENERAL CONDITIONS

- (A) Inspection - For purposes of ensuring compliance with this Stipulation, Exhibit A, and Attachments, Cenex shall, pursuant to 75-2-403, MCA, allow the Department representative(s) access to all SO₂ emitting sources at the Cenex facility such that, the Department representative(s) may, pursuant to 75-2-403, MCA, enter and inspect, at any reasonable time, any property, premises, or place, except a private residence, on or at which an SO₂ emitting source is located or is being constructed or installed. The Department representatives shall be allowed to conduct surveys, collect samples, obtain emissions data, audit any monitoring equipment (CEMS), or observe any monitoring or testing, and conduct all other necessary functions related to this control plan.

As provided in Section 75-2-105, MCA, Cenex may seek a court order declaring certain trade secret information as confidential and not a matter of public record. If Cenex claims that certain information is entitled to trade secret protection, the Department shall maintain such information as confidential pending issuance of a court order under Section 75-2-105, MCA, provided that Cenex initiates such court action within 14 days of delivering the information to the Department.

- (B) Enforcement - Any violation of a limitation, condition or other requirement contained herein ("Stipulation Requirement") constitutes grounds for judicial or administrative enforcement action. If the incident causing the violation would also form the basis of a violation of ARM Title 17, Chapter 8, or of Title 75, Chapter 2, MCA, the Department shall not count the violation of the Stipulation Requirement as an additional or separate violation incident for penalty calculation and assessment purposes.

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ATTACHMENT 1
PERFORMANCE SPECIFICATIONS FOR STACK FLOW RATE MONITORS,
FUEL OIL FLOWMETERS, AND FUEL OIL SULFUR ANALYSIS
(Includes Methods A-1, B-1, & C-1)

METHOD A-1
INSTALLATION AND INITIAL CERTIFICATION
IN-STACK OR IN-DUCT FLOW MONITORS

1.0 FLOW MONITOR INSTALLATION AND MEASUREMENT LOCATION

Install the flow monitor in a location that provides representative volumetric flow for all operating conditions. Such a location provides an average velocity of the flue gas flow over the stack or duct cross section, provides a representative SO₂ emission rate (in lb/hr), and is representative of the pollutant concentration monitor location. Where the moisture content of the flue gas affects volumetric flow measurements, use the procedures in both Reference Methods 1 and 4 of 40 CFR Part 60, Appendix A to establish a proper location for the flow monitor.

The department recommends (but does not require) performing a flow profile study following the procedures in 40 CFR Part 60, Appendix A, Test Method 1, Section 2.5 to determine the acceptability of the potential flow monitor location and to determine the number and location of flow sampling points required to obtain a representative flow value. The procedure in 40 CFR part 60, Appendix A, Test Method 1, Section 2.5 may be used even if the flow measurement location is greater than or equal to 2 equivalent stack or duct diameters downstream or greater than or equal to 1/2 duct diameter upstream from a flow disturbance. If a flow profile study shows that cyclonic (or swirling) or stratified flow conditions exist at the potential flow monitor location that are likely to prevent the monitor from meeting the performance specifications of this Method, then the department recommends either (1) selecting another location where there is no cyclonic (or swirling) or stratified flow condition, or (2) eliminating the cyclonic (or swirling) or stratified flow condition by straightening the flow, e.g., by installing straightening vanes. The department also recommends selecting flow monitor locations to minimize the effects of condensation, coating, erosion, or other conditions that could adversely affect flow monitor performance.

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1.1 Acceptability of Flow Monitor Location

The installation of a flow monitor is acceptable if (1) the location satisfies the minimum siting criteria of Method 1 in Appendix A to 40 CFR Part 60 (i.e., the location is greater than or equal to eight stack or duct diameters downstream and two diameters upstream from a flow disturbance; or, if necessary, two stack or duct diameters downstream and one-half stack or duct diameter upstream from, a flow disturbance), (2) the results of a flow profile study, if performed, are acceptable (i.e., there are no cyclonic (or swirling) or stratified flow conditions), and (3) the flow monitor satisfies the performance specifications of this Method. If the flow monitor is installed in a location that does not satisfy these physical criteria, but the monitor achieves the performance specifications of this Method, then the department and EPA may certify the location as acceptable.

1.2 Alternative Flow Monitoring Location

Whenever the flow monitor is installed in a location that is greater than or equal to two stack or duct diameters downstream and greater or equal to one-half diameter upstream from a flow disturbance, and/or in a location that is acceptable based on a flow profile study, but nevertheless the monitor does not achieve the performance specifications of this Method, perform another flow profile study (the procedures described in 40 CFR Part 60, Appendix A, Method 1, Section 2.5 may be used) to select an alternative flow monitoring installation site.

Whenever the owner or operator successfully demonstrates that modifications to the exhaust duct or stack (such as installation of straightening vanes, modifications of ductwork, and the like) are necessary for the flow monitor to meet the performance specifications, the department and EPA may approve an interim alternative flow monitoring methodology and an extension to the required certification date for the flow monitor.

Where no location exists that satisfies the physical siting criteria in section 1.1, where the results of flow profile studies performed at two or more alternative flow monitor locations are unacceptable, or where installation of a flow monitor in either the stack or the ducts is demonstrated to be technically infeasible, the owner or operator may petition the department and EPA for an alternative method for monitoring flow.

2.0 FLOW MONITOR EQUIPMENT SPECIFICATIONS

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2.1 Instrument Span - General Requirements

In implementing Section 2.1.1 of this Method, to the extent practicable, measure at a range such that the majority of readings obtained during normal operation are between 25 and 75 percent of full-scale range of the instrument.

2.1.1 Instrument Span for Flow Monitors

Select the full-scale range of the flow monitor so that it is consistent with Section 2.1 of this Method, and can accurately measure all potential volumetric flow rates at the flow monitor installation site. Establish the span value of the flow monitor at a level which is approximately 80% of the full-scale range and 125% of the maximum expected flow rate. Based upon the span value, establish reference values for the calibration error test in accordance with Section 2.2.1.

If the volumetric flow rate exceeds the flow monitor's ability to accurately measure and record values, adjust the full-scale range, span value, and reference values as described above and in Section 2.2.1. Record the new span value and report the new span value and reference values as parts of the results of the calibration error test required by Method B-1. Whenever the span value is adjusted, use reference values for the calibration error test based on the new span value.

2.2 Flow Monitor Design for Quality Control Testing

Design all flow monitors to meet the applicable performance specifications of this Method.

2.2.1 Flow Monitor Calibration Error Test

Design and equip each flow monitor to allow for a daily calibration error test consisting of at least two reference values: (1) Zero to 20 percent of span or an equivalent reference value (e.g., pressure pulse or electronic signal) and (2) 50 to 70 percent of span. Flow monitor response, both before and after any adjustment, must be capable of being recorded by the data acquisition and handling system. Design each flow monitor to allow a daily calibration error test of (1) the entire flow monitoring system, from and including the probe tip (or equivalent) through and including the data acquisition and handling system, or (2) the flow monitoring system from and including the transducer through and including the data acquisition and handling system.

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2.2.2 Flow Monitor Interference Check

Design and equip each flow monitor in a manner to minimize interference due to moisture. Design and equip each flow monitor with a means to detect, on at least a daily basis, pluggage of each sample line and sensing port, and malfunction of each resistance temperature detector (RTD), transceiver or equivalent.

Design and equip each differential pressure flow monitor to provide (1) an automatic, periodic back purging (simultaneously on both sides of the probe) or equivalent method of sufficient force and frequency to keep the probe and lines sufficiently free of obstructions on a least a daily basis to prevent velocity sensing interference, and (2) a means for detecting leaks in the system on a least a quarterly basis (manual check is acceptable).

Design and equip each thermal flow monitor with a means to ensure on at least a daily basis that the probe remains sufficiently clean to prevent velocity sensing interference.

Design and equip each ultrasonic flow monitor with a means to ensure on at least a daily basis that the transceivers remain sufficiently clean (e.g., backpurging system) to prevent velocity sensing interference.

3.0 FLOW MONITOR PERFORMANCE SPECIFICATIONS

3.1 Flow Monitor Calibration Error

The calibration error of flow monitors shall not exceed 3.0 percent based upon the span of the instrument as calculated using Equation A-1 of this Method.

3.2 Flow Monitor Relative Accuracy

Except as provided in this Section, the relative accuracy for flow monitors, where volumetric gas flow is measured in scfh, shall not exceed 20.0 percent. For affected units where the average of the flow monitor measurements of gas velocity during the relative accuracy test audit is less than or equal to 10.0 fps, the mean value of the flow monitor velocity measurements shall not exceed ± 2.0 fps of the reference method mean value in fps wherever the relative accuracy specification above is not achieved.

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4.0 DATA ACQUISITION AND HANDLING SYSTEMS

Automated data acquisition and handling systems shall: (1) read and record the full range of pollutant concentrations and volumetric flow from zero through span; and (2) provide a continuous record of all measurements and required information in an electronic format specified by the department and capable of transmission via an IBM-compatible personal computer diskette or other electronic media. These systems also shall have the capability of interpreting and converting the individual output signals from a pollutant concentration monitor and a flow monitor to produce a continuous readout of pollutant mass emission rates in pounds per hour.

Data acquisition and handling systems shall also compute and record monitor calibration error .

5.0 INITIAL FLOW MONITOR CERTIFICATION TESTS AND PROCEDURES

5.1 Flow Monitor Pretest Preparation

Install the components of the continuous flow monitor as specified in Sections 1.0, 2.0, and 3.0 of this Method, and prepare each system component and the combined system for operation in accordance with the manufacturer's written instruction. Operate the unit(s) during each period when measurements are made.

5.2 7-Day Calibration Error Test for Flow Monitors

Measure the calibration error of each flow monitor according to the following procedures.

Introduce the reference signal corresponding to the values specified in Section 2.2.1 of this Method to the probe tip (or equivalent), or to the transducer. During the 7-day certification test period, conduct the calibration error test once each day while the unit is operating (as close to 24-hour intervals as practicable). Record the flow monitor responses by means of the data acquisition and handling system. Calculate the calibration error using Equation A-1 of this Method.

Do not perform any corrective maintenance, repair, replacement or manual adjustment to the flow monitor during the 7-day certification test period other than that required in the monitor operation and maintenance manual. If the flow monitor operates within the calibration error performance specification, (i.e., less than or

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equal to 3 percent error each day and requiring no corrective maintenance, repair, replacement or manual adjustment during the 7-day test period) the flow monitor passes the calibration error test portion of the certification test. Whenever automatic adjustments are made, record the magnitude of the adjustments. Record all maintenance and required adjustments. Record output readings from the data acquisition and handling system before and after all adjustments.

5.3 Flow Monitor Relative Accuracy

Within 90 days of installation concurrent relative accuracy test audits may be performed by conducting simultaneous SO₂ concentration and volumetric flow relative accuracy test audit runs, or by alternating an SO₂ relative accuracy test audit run with a flow relative accuracy test audit run until all relative accuracy test audit runs are completed. Where two or more probes are in the same proximity, care should be taken to prevent probes from interfering with each other's sampling. For each SO₂ pollutant concentration monitor and each flow monitor, calculate the relative accuracy with data from the relative accuracy test audits.

Perform relative accuracy test audits for each flow monitor at normal operating load expressed in terms of percent of flow monitor span. If a flow monitor fails the relative accuracy test, the relative accuracy test audit must be repeated.

Complete each relative accuracy test audit within a 7-day period while the unit is operating in a normal condition. Do not perform corrective maintenance, repairs, replacements or adjustments during the relative accuracy test audit other than as required in the operation and maintenance manual.

5.3.1 Calculations

Using the data from the relative accuracy test audits, calculate relative accuracy in accordance with the procedure and equations specified in Section 6 of this Method.

5.3.2 Reference Method Measurement Location

Select a location for reference method measurements that is (1) accessible; (2) in the same proximity as the monitor or monitoring system location; and (3) meets the requirements of Method 1 (or 1A) of 40 CFR Part 60, Appendix A for volumetric flow, except as otherwise indicated in this Section.

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5.3.3 Reference Method Traverse Point Selection

Select traverse points that (1) ensure acquisition of representative samples of pollutant concentration, moisture content, temperature, and flue gas flow rate over the flue cross section; and (2) meet the requirements of Method 1 (or 1A) (for volumetric flow), and Method 4 (for moisture determination) in 40 CFR part 60, Appendix A.

5.3.4 Sampling Strategy

Conduct the reference method tests so they will yield results representative of the moisture content, temperature, and flue gas flow rate from the unit and can be correlated with the flow monitor measurements. Conduct any moisture measurements that may be needed simultaneously with the flue gas flow rate measurements. To properly correlate volumetric flow rate data with the reference method data, mark the beginning and end of each reference method test run (including the exact time of day) on the individual chart recorder(s) or other permanent recording device(s).

5.3.5 Correlation of Reference Method and Continuous Emission Monitoring System

Confirm that the monitor or monitoring system and reference method test results are on consistent moisture, pressure, and temperature basis (e.g., since the flow monitor measures flow rate on a wet basis, Method 2 test results must also be on a wet basis). Compare flow-monitor and reference method results on a scfh basis. Also consider the response time of the flow monitoring system to ensure comparison of simultaneous measurements. For each relative accuracy test audit run, compare the measurements obtained from the flow monitor against the corresponding reference method values. Tabulate the paired data in a table similar to the one shown in Figure 1.

5.3.6 Number of Reference Method Tests

Perform a minimum of nine sets of paired monitor (or monitoring system) and reference method test data for every required relative accuracy test audit. Conduct each set within a period of 30 to 60 minutes.

The tester may choose to perform more than nine sets of reference method tests. If this option is chosen, the tester may reject a maximum of three sets of the test results as long as the total number of test results used to determine the relative accuracy is greater than or equal to nine. Report all data, including the rejected

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data, and reference method test results.

5.3.7 Reference Methods

The following methods from 40 CFR Part 60, Appendix A or their approved alternatives are the reference methods for performing relative accuracy test audits: Method 1 or 1A for siting; Method 2 (or 2A, 2C, or 2D as appropriate) for velocity; and Method 4 for moisture.

6.0 CALCULATIONS

6.1 Flow Monitor Calibration Error (Drift)

For each reference value, calculate the percentage calibration error based upon span using the following equation:

$$CE = \frac{(R-A)}{S} \times 100 \quad (EQ.A-1)$$

Where:

CE = Calibration error;
R = Low or high level reference value specified in Section 2.2.1 of this Method;
A = Actual flow monitor response to the reference value; and
S = Flow monitor span.

Whenever the flow rate exceeds the monitor's ability to measure and record values accurately, adjust the span to prevent future exceedances. If process parameters change or other changes are made such that the expected flue gas velocity may change significantly, adjust the span to assure the continued accuracy of the monitoring system.

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6.2 Relative Accuracy for Flow Monitors

Analyze the relative accuracy test audit data from the reference method tests for flow monitors using the following procedures. Summarize the results on a data sheet. An example is shown in Figure 1. Calculate the mean of the monitor or monitoring system measurement values. Calculate the mean of the reference method values. Using data from the automated data acquisition and handling system, calculate the arithmetic differences between the reference method and monitor measurement data sets. Then calculate the arithmetic mean of the difference, the standard deviation, the confidence coefficient, and the monitor or monitoring system relative accuracy using the following procedures and equations.

6.2.1 Arithmetic Mean

Calculate the arithmetic mean of the differences, \bar{d} , of a data set as follows.

$$\bar{d} = \frac{1}{n} \sum_{i=1}^n d_i$$

Where:

n=Number of data points

$\sum_{i=1}^n d_i$ = Algebraic sum of the individual differences d_i

d_i = The difference between a reference method value and the corresponding continuous flowrate monitoring system value ($RM_i - FR_i$) at a given point in time i .

When calculating the arithmetic mean of the difference of a flow monitor data set, be sure to correct the monitor measurements for moisture if applicable.

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6.2.2 Standard Deviation

Calculate the standard deviation, S_d of a data set as follows:

$$S_d = \sqrt{\frac{\sum_{i=1}^n d_i^2 - \left[\frac{(\sum_{i=1}^n d_i)^2}{n} \right]}{n-1}} \quad (\text{Eq. A-3})$$

6.2.3 Confidence Coefficient

Calculate the confidence coefficient (one-tailed), cc , of a data set as follows.

$$CC = t_{0.025} \frac{S_d}{\sqrt{n}} \quad (\text{Eq. A-4})$$

where:

$t_{0.025}$ = *t value (see Table 2)*

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TABLE 2 T-VALUES

n-1	t _{0.025}	n-1	t _{0.025}	n-1	t _{0.025}
1.....	12.706	12	2.179	23	2.069
2.....	4.303	13	2.160	24	2.064
3.....	3.182	14	2.145	25	2.060
4.....	2.776	15	2.131	26	2.056
5.....	2.571	16	2.120	27	2.052
6.....	2.447	17	2.110	28	2.048
7.....	2.365	18	2.101	29	2.045
8.....	2.306	19	2.093	30	2.042
9.....	2.262	20	2.086	40	2.021
10.....	2.228	21	2.080	60	2.000
11.....	2.201	22	2.074	>60	1.960

6.2.4 Relative Accuracy

Calculate the relative accuracy of a data set using the following equation.

$$RA = \frac{|\bar{d}| + |cc|}{RM} \times 100 \quad (\text{Eq. A-5})$$

where:

RM = Arithmetic means of the reference method values.

$|\bar{d}|$ = The absolute value of the mean difference between the reference method values and the corresponding continuous flow monitor values.

$|cc|$ = The absolute value of the confidence coefficient.

FIGURE 1.-RELATIVE ACCURACY DETERMINATION (FLOW MONITORS)

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Run No.	Date & Time	Flow rate (Normal) (scf/hr)*		
		RM	M	Diff
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
Mean or mean of differences				
		Confidence coefficient		
		Relative accuracy		

* Make sure RM and M are on a consistent moisture basis.

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METHOD B-1
ON-GOING QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES
FOR IN-STACK AND IN-DUCT FLOW MONITORS

1.0 FREQUENCY OF FLOW MONITOR TESTING

A summary chart showing each quality assurance test and the frequency at which each test is required is located at the end of this Method in Table 1.

1.1 Daily Flow Monitor Assessments

For each flow monitor perform the following assessments during each day in which the unit is operating. These requirements are effective as of the date when the monitor or continuous emission monitoring system completes certification testing.

1.1.1 Calibration Error Test for Flow Monitors

Test, compute, and record the calibration error of each flow monitor at least once on each operating day. Introduce the reference values (specified in section 2.2.1 of Method A-1) to the probe tip (or equivalent) or to the transducer. Record flow monitor output from the data acquisition and handling system before and after any adjustments to the flow monitor. Keep a record of all maintenance and adjustments. Calculate the calibration error using Equation A-1 in Method A-1.

1.1.2 Flow Monitor Interference Check

Perform the daily flow monitor interference checks specified in section 2.2.2 of Method A-1 at least once per operating day (when the unit(s) operate for any part of the day).

1.1.3 Flow Monitor Recalibration

Adjusts the calibration, at a minimum, whenever the daily calibration error exceeds the limits of the applicable performance specification for the flow monitor in Method A-1. Repeat the calibration error test procedure following the adjustment or repair to demonstrate that the corrective actions were effective.

1.1.4 Flow Monitor Out-of-Control Period

An out-of-control period occurs when either the low or high level reference

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value calibration error exceeds 6.0 percent based upon the span value for five consecutive daily periods or 12.0 percent for any daily period. The out-of-control period begins with the hour of completion of the failed calibration error test and ends with the hour of completion following an effective recalibration. Whenever the failed calibration, corrective action, and effective recalibration occur within the same hour, the hour is not out of control if two or more complete and valid readings are obtained during that hour. An out-of-control period also occurs whenever interference of a flow monitor is identified. The out-of-control period begins with the hour of completion of the failed interference check and ends with the hour of completion of an interference check that is passed. During any period that the flow monitor is out-of-control, the data may not be used in calculating emission compliance nor be counted towards meeting minimum data recovery requirements.

1.1.5 Flow Monitor Data Recording

Record and tabulate all calibration error test data according to month, day, clockhour, and magnitude in scfh. Program monitors that automatically adjust data to the corrected calibration values (e.g., microprocessor control) to record either: (1) The unadjusted flow rate measured in the calibration error test prior to resetting the calibration or (2) the magnitude of any adjustment. Record the following applicable flow monitor interference check data: (1) Sample line/sensing port pluggage, and (2) malfunction of each RTD, transceiver, or equivalent.

1.2 Quarterly Flow Monitor Assessments

For each flow monitor, conduct a quarterly stack velocity and flow rate check by performing a velocity traverse and visual inspection of the pitot tubes. Perform the following assessments during each calendar quarter in which the unit operates. This requirement is effective as of the calendar quarter following the calendar quarter in which the flow monitor is provisional certified.

1.2.1 Flow Monitor Leak Check

For differential pressure flow monitors, perform a leak check of all sample lines (a manual check is acceptable) at least once during each unit operating quarter. Conduct the leak checks no less than two months apart.

1.2.2 Flow Monitor Flow Rate Check

Once during each operating quarter and for each flow monitor, perform a flow rate check by completing a single velocity traverse, calculating the associated

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average flow rate, and comparing the average flow with the concurrent flow measured by the continuous flow monitor. The flow rate check shall be performed at normal operating rates or load level. The flow rate check shall be performed in accordance with Section 5.3 of Method A-1 as appropriate for a single traverse. The difference (PD) between the average flow rate determined by the single velocity traverse and the continuous flow monitor shall not exceed 20 percent as determined by equation B-1. If the single velocity traverse fails to meet the 20% difference specification, the owner/operator may conduct an additional single velocity traverse or a complete Relative Accuracy Test Audit (RATA) in accordance with Section 5.3 of Method A-1 in order to demonstrate compliance with the 20% difference or 20% relative accuracy requirements.

$$PD = \frac{TF - FR}{TF} \times 100 \quad (\text{Eq. B-1})$$

Where:

PD = Percent Difference;
TF = Traverse Flow (scfh);
FR = Continuous Flow Monitor Flow (scfh); and
TF and FR are on a consistent moisture basis.

If the Relative Accuracy of the latest annual Relative Accuracy Test Audit (RATA) conducted pursuant to Section 1.3.1 is less than 10%, the single velocity traverse flow rate check may be discontinued. However, if future RATAs indicate a Relative Accuracy of 10% or greater, performance of the single velocity traverse flow rate check shall resume.

1.2.3 Flow Monitor Out-of-Control Period

An out-of-control period occurs when a flow monitor fails the quarterly flow rate check (the difference between the average flow rate determined by the velocity traverse and the continuous flow monitor exceeds 20%), the visual inspection of the pitot tube indicates pluggage or wear, or if a sample line leak is detected. The out-of-control period begins with the hour of the failed flow rate check, visual inspection, or leak check and ends with the hour of a satisfactory flow rate check, RATA, leak check, or cleaning or replacement of the pitot tube. During any period that the flow monitor is out-of-control, the data may not be used in calculating emission compliance nor be counted towards meeting minimum data recovery requirements.

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~~1.3 Annual Flow Monitor Assessments~~

~~For each flow monitor, perform the following assessments once annually. This requirement is effective as of the calendar quarter in which the monitor or continuous emission monitoring system is provisionally certified.~~

~~1.3.1 Flow Monitor Relative Accuracy Test Audit~~

~~For flow monitors, relative accuracy test audits shall be performed annually. The relative accuracy audit shall be performed at the normal operating rate or load level (with a minimum of 9 paired velocity traverses). The relative accuracy test audit shall be conducted according to the procedures and specifications of Method A-1.~~

~~1.3.2 Flow Monitor Out-of-Control Period~~

~~An out-of-control period occurs under any of the following conditions: (1) the relative accuracy of a flow monitor exceeds 20.0 percent or (2) for low flow situations (≤ 10.0 fps), the flow monitor mean value (if applicable) exceeds ± 2.0 fps of the reference method mean whenever the relative accuracy is greater than 20.0 percent. For flow relative accuracy test audits, the out-of-control period begins with the hour of completion of the failed relative accuracy test audit and ends with the hour of completion of a satisfactory relative accuracy test audit. During any period that the flow monitor is out-of-control, the data may not be used in calculating emission compliance nor be counted towards meeting minimum data recovery requirements.~~

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TABLE 1.-FLOW MONITOR QUALITY ASSURANCE TEST REQUIREMENTS

Test	QA test frequency requirements		
	Daily	Quarterly	Annual
Calibration Error (2 pt.)	x		
Interference (flow)	x		
Visual probe check		x	
Flow rate check (single traverse)		x ¹	
Leak (flow)		x ²	
RATA (flow)			x

¹ The owner/operator has an option to perform a RATA if the quarterly flow rate check (single traverse) fails specifications. In addition, if the Relative Accuracy determined by the latest RATA is less than 10%, the quarterly single velocity traverse flow rate check may be discontinued. However, if future RATAs indicate a Relative Accuracy of 10% or greater, performance of the quarterly single velocity traverse flow rate check shall resume.

² The leak check requirement only applies to differential pressure flow rate monitors and does not apply to thermal or ultrasonic flow rate monitors.

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METHOD C-1
FUEL OIL FLOWMETERING AND ANALYSIS SPECIFICATIONS

1.0 FLOWMETER SPECIFICATIONS

Cenex shall measure and record the fuel oil consumption rate within the fuel oil loop on an hourly basis. Cenex shall measure the flow of fuel oil with in-line fuel oil flowmeters, as required by Section 6 (B) (5) of Exhibit A.

1.1 Initial Calibration and Certification

Design and equip each fuel oil flowmeter used to demonstrate a flowmeter accuracy of 2.0 percent of the upper range value (i.e., maximum calibrated oil flow rate) as measured under laboratory conditions by the manufacturer or by the owner or operator. Use the procedures in the following ASME codes for flow measurement for use in the laboratory, as appropriate to the type of flowmeter: ASME MFC-3M-1989 with September 1990 Errata (Measurement of Fluid Flow in Pipes Using Orifice, Nozzle, and Venturi), ASME MFC-5M-1985 (Measurement of Liquid Flow in Closed Conduits Using Transit-Time Ultrasonic Flowmeters), ASME MFC-6M-1987 with June 1987 Errata (Measurement of Fluid Flow in Pipes Using Vortex Flow Meters), or ASME MFC-9M-1988 with December 1989 Errata (Measurement of Liquid Flow in Closed Conduits by Weighing Method) for all other flowmeter types. More current ASME or NIST (National Institute of Standards and Technology) procedures or other ASME or NIST procedures which are appropriate to flowmeter construction may, upon Department approval, be substituted. If the flowmeter accuracy exceeds 2 percent of the upper range value, the flowmeter does not qualify for certification.

1.2 Annual Calibration

Recalibrate each fuel oil flowmeter to a flowmeter accuracy of 2.0 percent of the upper range value at least annually, or more frequently if required by manufacturer specifications using the same ASME procedures required for initial calibration and certification.

1.2.1 Alternative Annual Calibration Method

Alternatively, the fuel oil flowmeter may be recalibrated to a flowmeter accuracy of 2.0 percent of the upper range value at least annually by comparing the measured flow of a flowmeter to the measured flow from another flowmeter which has been calibrated or recalibrated during the previous 365 days using the procedures in

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ASME MFC-9M-1988 with December 1989 Errata, "Measurement of Liquid Flow in Closed Conduits by Weighing Method", or which has been recalibrated by the manufacturer. Perform the comparison over a period of no more than seven consecutive facility operating days. Compare the average of three fuel oil flow readings for each meter at three different flow levels: (1) a frequently used low operating level selected within the range between the minimum safe and stable operating level and 50% of maximum operating level; (2) a frequently used high operating level selected within the range between 80% of maximum operating level and maximum operating level; and (3) normal operating level. Calculate the flowmeter accuracy using the following equation:

$$ACC = \frac{|R - A|}{URV} \times 100 \quad (\text{Eq. C-1})$$

Where:

- ACC = Flow meter accuracy as a percentage of the upper range value.
- R = Average of the three low-, mid-, or high-level flow measurements of the reference flowmeter.
- A = Average of the three measurements of the flowmeter being tested.
- URV = Upper range value of fuel flowmeter being tested (i.e. maximum measurable flow).

If the flowmeter accuracy exceeds 2% of the upper range value, either recalibrate the flowmeter until the accuracy is within the performance specification, or replace the flowmeter with another one that is within the performance specification.

2.0 FUEL OIL SAMPLING AND ANALYSIS

Cenex shall perform sampling and analysis of as-fired fuel oil from the fuel oil loop to determine the percentage of sulfur by weight in the fuel oil.

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2.1 Sampling Frequency and Methods

Cenex shall perform daily fuel oil sampling using either the flow proportional method described in Section 2.2 or the daily manual method described in Section 2.3.

2.2 Flow Proportional Sampling Method

Cenex shall conduct flow proportional fuel oil sampling or continuous drip fuel oil sampling in accordance with ASTM D4177-82 (Reapproved 1990), "Standard Practice for Automatic Sampling of Petroleum and Petroleum Products", every day the facility is combusting fuel oil within the fuel oil loop. Extract fuel oil at least once every hour and blend into a daily composite sample. The sample compositing period may not exceed 24 hours.

2.3 Daily Manual Sampling Method

Representative as-fired fuel oil samples may be taken manually every 24 hours according to ASTM D4057-88, "Standard Practice for Manual Sampling of Petroleum and Petroleum Products", provided that the highest fuel oil sulfur content recorded at that facility from the most recent 30 daily samples is used for the purposes of calculating SO₂ emissions.

2.4 Sample Archiving

Split and label each daily fuel oil sample. Maintain a portion (at least 200 cc) of each daily sample for not less than 150 calendar days after the submittal to the Department of the quarterly data report for the calendar quarter during which the sample was collected. Analyze fuel oil samples for percent sulfur content by weight in accordance with ASTM D129-91, "Standard Test Method for Sulfur in Petroleum Products (General Bomb Method)," ASTM D1552-90, "Standard Test Method for Sulfur in Petroleum Products (High Temperature Method)," ASTM D2622-92, "Standard Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry," or ASTM D4294-90, "Standard Test Method for Sulfur in Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectroscopy".

3.0 VOLUMETRIC FLOW MEASUREMENT

3.1 Fuel Oil Density

Where the flowmeter records volumetric flow rather than mass flow, analyze daily

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fuel oil samples to determine the density or specific gravity of the fuel oil (not required where the flowmeter records mass flow). Determine the density or specific gravity of the fuel oil sample in accordance with ASTM D941-88, "Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer," ASTM D1217-91, "Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer," ASTM D1481-91, "Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary," ASTM D1480-91, "Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer," ASTM D1298-85 (Reapproved 1990), "Standard Practice for Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method," or ASTM D4052-91, "Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter".

3.2 Calculation Of Mass Flow From Volumetric Flow

Where the flowmeter records volumetric flow rather than mass flow, calculate and record the fuel oil mass for each hourly period using hourly fuel oil flow measurements and the density or specific gravity of the daily oil sample.

Convert density, specific gravity, or API gravity of the fuel oil sample to density of the fuel oil sample at the sampling location's temperature using ASTM D1250-80 (Reapproved 1990), "Standard Guide for Petroleum Measurement Tables".

Where density of the fuel oil is determined by the applicable ASTM procedures from Section 3.1 of Department Method C-1, use the following equation to calculate the mass of fuel oil consumed (in lb/hr).

$$Moil = Voil \times Doil \quad (\text{Eq. C-2})$$

Where:

Moil = Mass of oil consumed per hr, lb/hr.

Voil = Volume of oil consumed per hr, measured in scf, gal, barrels, or m³.

Doil = Density of oil, measured in lb/scf, lb/gal, lb/barrel, or lb/m³.

When the mass of fuel oil consumed is determined, in accordance with Section 3.0 of Department Method C-1, such data can be used in the equation in Section 2

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(A) (12) (c) of Exhibit A to determine SO₂ emissions from fuel oil combustion.

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METHOD #6A-1

**ANALYTICAL METHOD FOR ANALYZING THE SOUR WATER STRIPPER FEED FOR
HYDROGEN SULFIDE (H₂S)
(October 1999)**

1.0 SCOPE AND APPLICATION

This method is applicable to the measurement of total and dissolved sulfides in sour water produced by the refinery. Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class).

2.0 SUMMARY OF METHOD

Excess iodine is added to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3.0 COMMENTS

Reduced sulfur compounds, such as sulfite, thiosulfate and hydrosulfite, which decompose in acid may yield erratic results. Also, volatile iodine-consuming substances such as mercaptans will give high results.

The sample source is hot and under pressure.

The volumes of preservative and the normality of the reagents have been modified from the referenced methods. The modifications are to make the method appropriate for the expected high concentrations of sulfide in the samples. The method calculations are also modified to correct for the sample dilution from the preservative.

4.0 APPARATUS

- 4.1 Ordinary laboratory glassware.
- 4.2 130 ml HDPE bottles. These bottles are pre-charged with preservative. For the sour water stripper feed inlet the bottle contains 5 ml of zinc acetate and 10 ml of sodium hydroxide.

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5.0 REAGENTS

- 5.1 Hydrochloric acid, HCl, 6 N
- 5.2 Standard iodine solution, 0.1000 N: Dissolved 20 to 25 g KI in a little water in a liter volumetric and add 12.8 g iodine. Allow to dissolve. Dilute to 1 liter and standardize against 0.1000 N sodium thiosulfate using a starch indicator.
- 5.3 Sodium thiosulfate 0.1000N: Dissolve 24.82 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in water. Add 1 ml of chloroform and dilute to 1000 ml.
- 5.4 Starch indicator: Dissolved 10 g soluble starch and 10 mg Hg_2I in hot water and dilute to 4 liters.
- 5.5 Standardize the sodium thiosulfate against KIO_3 . Adjust the concentration to 0.1000 N. Use this sodium thiosulfate to standardize the iodine solution.
- 5.6 Zinc acetate solution, 2N: Dissolve 220 g $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to one liter.
- 5.7 Sodium hydroxide, 8N: Dissolve 240 grams of sodium hydroxide in 800 ml of water. Dilute to one liter. Caution: much heat will be liberated.

6.0 SAMPLING

- 6.1 The sample bottles (4.2) are pre-charged with zinc acetate and sodium hydroxide preservative and labeled. The sample bottle sample contains 5 ml of preservative and 10 of sodium hydroxide.
- 6.2 The sample is obtained by carefully filling the appropriate bottle. Fill the bottle slowly to prevent the sample from splashing the preservative out or overflowing the bottle. The bottle should be completely filled with no headspace air. If necessary, the sides of the bottle can be squeezed while screwing on the lid to exclude the remaining air.
- 6.3 Experience shows that the pH of these samples, taken and preserved as described, are above 9. No further pH adjustment is required.

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7.0 PROCEDURE

- 7.1 Shake the container to suspend all solids and remove the sample. Measure the volume of sample. This is used to correct the results for the dilution due to the preservative. Check the pH of the sample using pH test paper to confirm that it is 9 or higher.
- 7.2 Place 20 ml of standard iodine solution (5.2) into a 500 ml iodine titration flask.
- 7.3 Add 15 ml of 8N HCl (5.1).
- 7.4 Thoroughly mix the sample and quickly take a 25 ml aliquot and place it in the flask.
- 7.5 If the iodine color disappears, add more iodine until the color remains. Record the total number of milliliters of standard iodine used steps 7.2 and 7.5.
- 7.6 Titrate with the reducing solution (0.1 N sodium thiosulfate) to a pale straw color. Add the starch indicator and titrate until the blue color disappears. Record the volume used.

8.0 CALCULATIONS

8.1 Sulfide as H₂S, mg/l = $\frac{(A - B) \times 17.01 \times 1000}{\text{sample aliquot, ml} \times K}$

Where: A = Volume of Iodine, ml * Normality of Iodine
B = Volume of Thiosulfate, ml * Normality of Thiosulfate
K = $\frac{\text{ml of sample} - \text{ml of preservative}}{\text{ml of sample}}$

This is a correction for the preservative volume. The volume of sample is the total volume in the sample container including the preservative. The volume of preservative is the volume added to the container before the sample was obtained.

9.0 REFERENCES

- 9.1 Standard Methods for the Examination of Water and Wastewater, 19th Edition, p 4-127, Method 4500-S₂ F, (1995)
EPA Method 376.1

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METHOD #6B
PRECISION AND ACCURACY METHOD FOR THE SOUR
WATER STRIPPER FLOW METER

1.0 SCOPE AND APPLICATION

This method is applicable to any typical orifice type flow meter that is installed consistent with ASME code procedures found under ASME MFC-3M-1989 (Measurement of Fluid Flow in Pipes Using Orifice, Nozzle, and Venturi). Such a designed and installed flow meter will, if maintained consistent with these procedures, have an accuracy of 2 percent of the upper range.

2.0 SUMMARY OF METHOD

The calibration is to ensure installation parameters are maintained and the flow measurement components maintain their integrity during the period of operation so that the output can be relied upon as representative of a 2 percent accurate flow.

3.0 PROCEDURE FOR ANNUAL CALIBRATION

3.1 Meter Information

Obtain the sour water feed meter installation information for the meter to the Sour Water Stripper. This information should be verified as consistent with the designed system and as installed in the field.

3.2 Field Verification

Verify the field orifice meter actually installed is that which was installed originally by checking the orifice plate tab at the meter between the orifice flanges. Verify the transmitter differential pressure range is the same as required in the design and originally set.

3.3 Transmitter Span Check and Zero Check

3.3.1 Span Check

Block transmitter at the three valve manifold. As a precaution during this procedure, the technicians should position themselves upwind due to the potential of elevated H₂S concentrations in this stream. Connect the multimeter to the transmitter and observe and note the transmitter output. Unblock the high pressure side manifold valve, vent the low pressure side of the transmitter making sure the vent is pointing away from the technician. The transmitter output should go up scale to 20 + milliamps direct current (madc).

3.3.2 Tap/Lead Line Plugging Check

Close low pressure vent. Open low pressure manifold valve and block in high pressure manifold valve making sure the vent is

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pointed away from the technician. The transmitter output should go down scale to less than 4.0 mdc. Open equalizer valves on manifold. This will allow blowing of the low side lead line to check for any plugging or plugged tap. If it blows satisfactorily, close the low pressure side transmitter vent and manifold equalizing valve. Next open the transmitter high pressure side vent and then the high pressure manifold valve to allow blowing the high pressure lead line to check for any plugging or plugged tap.

3.3.3 Zero Check

Close transmitter high pressure side vent, leave high pressure manifold valve open and open manifold equalizer valve. The transmitter output should go to zero. If no adjustment is required, proceed to place the instrument back into service. If outside a 2% accuracy, follow the steps in Section 4.0 of these procedures for additional accuracy checks.

4.0 FORTY-EIGHT MONTH ACCURACY CHECK

4.1 Scope and Application

This part of the procedure will be conducted at least three months prior to a FCC/CO Boiler turnaround but not more than a 48 months interval, to ensure the accuracy of this meter prior to a known period when the sour water stripper overheads will be incinerated in the main crude heater for an extended period of up to 40 days.

4.2 Meter Information and Field Verification

Follow steps 3.1 and 3.2 to ensure the meter installation has the designed components in place.

4.3 Transmitter Zero and Span Check

Block the transmitter at the root valves and at the three valve manifold. As a precaution during this procedure, the technicians should position themselves upwind due to the potential of elevated H₂S concentrations in this stream. Bleed pressure with the bleeder pointed away from the technician. Remove wiring and tubing, inspect flex and tubing and replace as needed. Remove mounting and equalizer bolts and remove transmitter. Take transmitter to the refinery instrument shop for Bench Check calibration.

Remove body flanges and clean all parts and check for corrosion and other problems that could affect transmitter performance. Repair or replace as needed to ensure an accurate flow measurement. Apply proper pressure to define and calibrate at zero and full scale for the span. Adjust as necessary and check repeatability.

4.4 Field Inspection of Meter Installation

After the meter transmitter is removed, remove the orifice plate and measure its diameter to confirm no change has occurred. Establish if a new plate is required due to corrosion or erosion and obtain for installation. In either case, a new meter factor must be developed consistent with ASME MFC-3M-1989 or equivalent.

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Once the orifice plate is removed, visually inspect the process piping. Clean as needed and measure the inside pipe diameter of the up stream and downstream piping to confirm no deterioration has occurred. If more than a 2% increase in orifice or pipe diameter has occurred, a new meter factor must be developed consistent with ASME MFC-3M-1989 or equivalent.

4.5 Field Zero and Span Check to Confirm Proper Installation

Reinstall all parts using new bolts and gaskets. Open root valves, bypass on the equalizer and the low side tap of the equalizer. Close the bleeder. This will give a zero reading. Verify zero reading on the transmitter. When zero checking is complete, check the range utilizing a handheld digital pneumatic meter by pumping up pressure to match the design and installed maximum pressure reading. Verify the maximum span on the transmitter. If there is more than a 2% difference, make necessary adjustments. If within the 2% accuracy, proceed to put the instrument back in service. If transmitter will not operate properly while conducting the zero and span checks, remove from service and take to the refinery instrument shop to conduct another full Bench Check on the transmitter. If unable to achieve a 2% accuracy, repair or replace and follow the above steps on the repaired or replaced instrument until a 2% accuracy is obtained on the field installed instrument. Proceed to recommission as appropriate and in a timely manner check to be sure operations is satisfactory.

5.0 TRACK CHANGES

If any changes to the original equipment, i.e. orifice plate, piping changes, differential pressure or meter factor, place new data into the refinery meter tracking information system and use for future meter calibrations and accuracy checks.

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