

SUMMARY SHEET 10
Carbon Monoxide

| | | Run #1 | Run #2 | Run #3 | Avg |
|---|-----------------|----------|--------|--------|-----|
| Client/Plant Name | FDS 10 | | | | |
| Job No. | FDS 10 | | | | |
| Sampling Location | FDS 10 | | | | |
| Run ID # | FDS 10 | | | | |
| Test Date | FDS 10 | | | | |
| Run Start Time | FDS 10 | | | | |
| Run Finish Time | FDS 10 | | | | |
| Concentration of CO measured, dry, ppm | $C_{CO\ NDIR}$ | FDS 10 | | | |
| Vol. fraction of CO ₂ in sample, (%CO ₂ /100) | F_{CO_2} | FDS 3/3B | | | |
| Conc. of CO in stack, dry, ppm | $C_{CO\ stack}$ | SS 10 | | | |

$$C_{CO\ stack} = C_{CO\ NDIR} (1 - F_{CO_2})$$

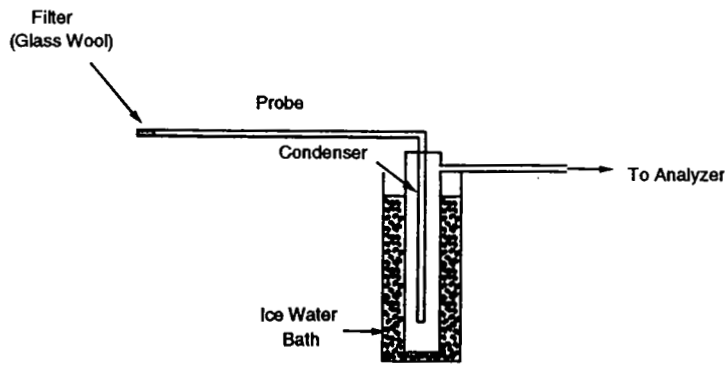


Figure F10-1. Continuous Sampling Train.

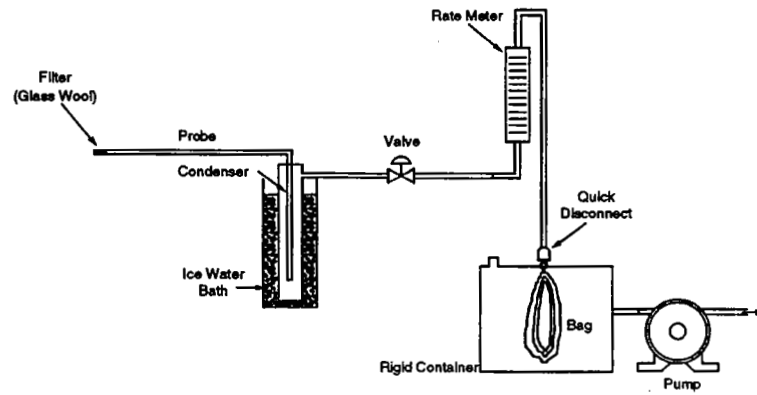


Figure F10-2. Integrated Gas-Sampling Train.

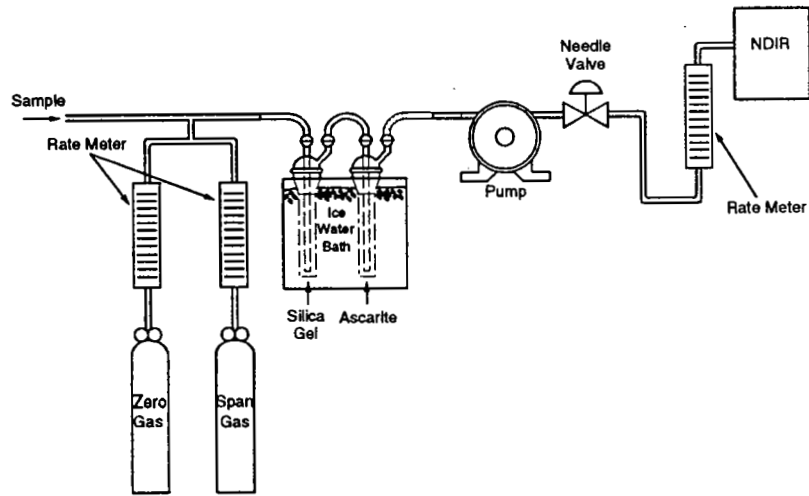


Figure F10-3. Analytical Equipment.

FIELD PROCEDURE 10
Carbon Monoxide

A. Pre-Test Preparation

1. Obtain a CO analyzer using nondispersive infrared spectrometry, or equivalent. Obtain from the manufacturer a certification that the analyzer meets the specifications below:

| Parameter | Specification |
|------------------------------|---|
| Range (min) | 0-1000 ppm |
| Output (min) | 0-10 mV |
| Min detectable sensitivity | 20 ppm |
| Rise time, 90% (max) | 30 sec |
| Fall time, 90% (max) | 30 sec |
| Zero drift (min) | 10% in 8 hr |
| Span drift (max) | 10% in 8 hr |
| Precision | ± 2% of full scale |
| Noise (max) | ± 1% of full scale |
| Linearity (max dev) | 2% of full scale |
| Interference rejection ratio | CO ₂ : 1000 to 1 H ₂ O: 500 to 1 |

2. Obtain CO calibration gases (CO in N₂), certified by the manufacturer to be within ± 2% of the specified concentration, as follows:
 - a. Span. ≤ 1.5 times the applicable performance standard.
 - b. High-Range. About 60% of span.
 - c. Mid-Range. About 30% of span.
 - d. Zero. Prepurified grade of N₂.

B. Continuous Sampling

1. Set up the equipment as shown in Figures F10-1 and F10-2. Ensure that all connections are leak free.
2. Prepare the CO analyzer according to the manufacturer's instructions. Allow at least 1 hr for warm-up. Calibrate the CO analyzer according to the manufacturer's procedures using N₂ and the calibration gases. Record the data on FDS 10.
3. Place the probe in the stack at a sampling point, and purge the sampling line with stack gas.

4. Connect the analyzer, and draw sample into the analyzer. Allow 5 min for the system to stabilize, then record the analyzer reading.
5. Before introducing each sample, purge analyzer with N₂.
6. After the test, check the zero and the span again.
7. Determine the CO₂ content of the gas according to Method 3 or 3B integrated sampling procedure (attach appropriate data sheets).

C. Integrated Sampling

1. Leak-test the flexible bag. Evacuate the bag with a pump followed by a dry gas meter. After evacuation, the meter should indicate zero flow.
2. Set up the equipment as shown in Figure F10-3 with the bag disconnected. Evacuate the flexible bag again, if necessary.
3. Place the probe in the stack at a sampling point, and purge the sampling line with stack gas.
4. Connect the bag. Ensure that all connections are leak free.
5. Sample at a rate proportional to the stack velocity. Use a pitot tube, if velocity is varying with time.
6. Analyze the bag sample using appropriate procedures in section B.
7. Determine the CO₂ content as in step B7.

D. Alternatives

1. The sample conditioning system described in Method 10A, sections 2.1.2 and 4.2, may be used instead of the silica gel and ascarite traps.
2. CO₂ may be determined by weighing the ascarite CO₂ removal tube and computing CO₂ concentration from the gas volume sampled and the weight gain of the tube.

SUMMARY SHEET 10A
Carbon Monoxide

| | | Run #1 | Run #2 | Run #3 | Avg |
|---|----------|---------|--------|--------|-----|
| Client/Plant Name | | FDS 10A | | | |
| Job No. | | FDS 10A | | | |
| Sampling Location | | FDS 10A | | | |
| Run ID # | | FDS 10A | | | |
| Test Date | | FDS 10A | | | |
| Run Start Time | | FDS 10A | | | |
| Run Finish Time | | FDS 10A | | | |
| Net Traverse points | | FDS 1 | | | |
| Traverse Matrix (if rectangular) | | FDS 1 | | | |
| Net Run Time, min | θ | FDS 10A | | | |
| Sampling Rate, mL/min | Q_s | FDS 10A | | | |
| CO ₂ Concentration, fraction | F | FDS 10A | | | |
| Field Temperature, °C | t_f | FDS 10A | | | |
| Field Barometric Pressure, mm Hg | P_b | FDS 10A | | | |
| Average Absorbance | A | LDS 10A | | | |
| Absorbance, Reagent Blank | A_r | LDS 10A | | | |
| Room Temperature, °C | t_r | LDS 10A | | | |
| Lab Barometric Pressure, mm Hg | P_b | LDS 10A | | | |
| Bag Moisture Content | B_w | LDS 10A | | | |
| Cal Curve CO Concentration, ppm | C_g | LDS 10A | | | |
| Bag CO Concentration, ppm dry | C_b | SS 10A | | | |
| Stack CO Concentration, ppm dry | C | SS 10A | | | |

$$C_b = \frac{C_g}{(1 - B_w)}$$

$$C = C_b (1 - F)$$

FIELD PROCEDURE 10A Carbon Monoxide

A. Pretest Preparation

1. **Optional:** Leak-check the bags before sampling according to FP 3.
2. Loosely pack glass wool in the tip of the probe.
3. Place 400 mL alkaline permanganate solution in the first two impingers and 250 mL in the third.
4. Evacuate the Tedlar bag completely using a vacuum pump.
5. Assemble the sampling train as shown in F10A-1. Do not connect the Tedlar bag to the system at this time.
6. Leak-check the sampling system as follows: plug the probe inlet, open the 3-way valve, and pull a vacuum of ~250 mm Hg on the system. No flow on the rate meter indicates the system is leak free.

B. Sampling

1. Insert the probe into the stack and draw sample through the system at 300 mL/min \pm 10% and purge the system for 5 min.
2. Connect the evacuated Tedlar bag to the system, and sample at a rate of 300 mL/min for 30 min, or until the Tedlar bag is nearly full.
3. Replace the scrubber solution after every fifth sample or every 50 L of stack gas when the concentration of SO₂ or NO_x is <1000 ppm and CO₂ is <15%, and more often if greater.
4. Measure the CO₂ content to the nearest 0.5% each time a CO sample is collected. A simultaneous grab sample with a Fyrite analyzer is acceptable.

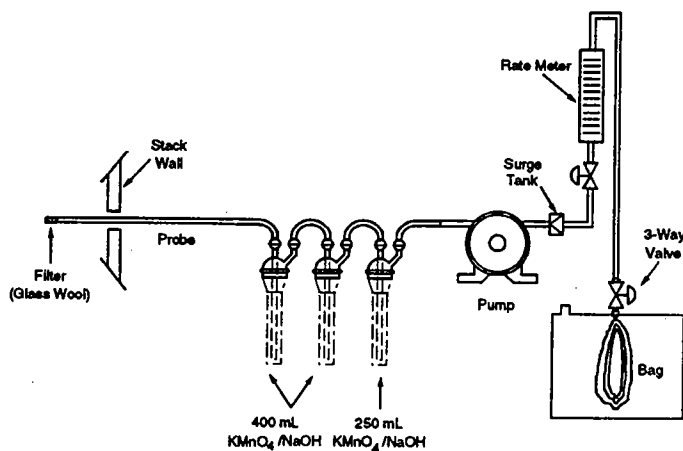


Figure F10A-1. Sampling Train.

**FIELD DATA SHEET 10A
Carbon Monoxide**

Client/Plant Name _____ Job # _____

City/State _____ Bar Press, P_b _____ mm Hg Date _____

Test Location _____ Personnel _____

| | Run #1 | Run #2 | Run #3 |
|--|--------|--------|--------|
| Optional pre-test leak check acceptable? | _____ | _____ | _____ |
| Bag evacuated until rotameter reads zero? | _____ | _____ | _____ |
| Sample line purged at 300 mL/min ± 10% for ≥ 5 min before each sample? | _____ | _____ | _____ |

Note start and end times:

| Run #1 | | | Run #2 | | | Run #3 | | |
|--------|------------------|-----------|--------|------------------|-----------|--------|------------------|-----------|
| Time | Rot Rdg (mL/min) | Temp (°C) | Time | Rot Rdg (mL/min) | Temp (°C) | Time | Rot Rdg (mL/min) | Temp (°C) |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

| | Run #1 | Run #2 | Run #3 |
|--|--------|--------|--------|
| Sampling rate 300 ± 30 mL/min? | _____ | _____ | _____ |
| Sampling time ≥ 30 min or bag almost full? | _____ | _____ | _____ |
| Fyrite CO ₂ (If Method 3 is used, attach FDS) | _____ | _____ | _____ |

____ Rotameter Calibration Data Sheet attached?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____
Personnel (Signature/Date)

Team Leader (Signature/Date)

LABORATORY PROCEDURE 10A
Carbon Monoxide

A. Reagents

1. Alkaline Permanganate, 0.25 M KMnO_4 /1.5 M NaOH. Dissolve 40 g KMnO_4 and 60 g NaOH in water, and dilute to 1 L.
2. Sodium Hydroxide, 1 M. Dissolve 40 g NaOH in ~900 mL of water, cool, and dilute to 1 L.
3. Silver Nitrate, 0.1 M. Dissolve 8.5 g AgNO_3 in water, and dilute to 500 mL.
4. Para-Sulfaminobenzoic Acid (p-SABA), 0.1 M. Dissolve 10.0 g p-SABA in 0.1 M NaOH, and dilute to 500 mL with 0.1 M NaOH.
5. Colorimetric Solution. Add 100 mL of p-SABA solution and 100 mL of AgNO_3 solution into a flask. Mix, and add 50 mL of 1 M NaOH with shaking (should be clear and colorless). Do not use after 2 days.
6. Standard Gas Mixtures. Use at least two CO concentrations (in N_2) between 50 and 1000 ppm (NIST-traceable) to span each calibration range.

B. Equipment Preparation and Analysis

1. Calibrate the reaction bulbs as follows (Use CDS 10A).
 - a. Weigh the empty bulb to ± 0.1 g.
 - b. Fill the bulb to the stopcock with water, and weigh to ± 0.1 g.
 - c. Measure room temperature of water. Calculate the volume to ± 0.001 L using the density of water at the measurement temperature.
 2. Collect the standards according to FP 10A to span 0-400 ppm or 400-1000 ppm, or both if samples occur in these ranges.
 3. Assemble the system shown in L10A-1. Pipet 10.0 mL of the colorimetric reagent into each gas reaction bulb, and attach the bulbs to the system.
 4. Evacuate the reaction bulbs and leak-check the system as follows:
 - a. Open the stopcocks to the reaction bulbs, but leave the valve to the Tedlar bag closed.
 - b. Turn on the pump, fully open the coarse-adjust flow valve, and slowly open the fine adjust valve until the pressure is reduced to at least 40 mm Hg.
 - c. Close the coarse adjust valve, and observe the manometer after ≥ 2 min.
 5. Flush the manifold completely at least twice as follows:
 - a. Open the Tedlar bag valve, and allow the system to come to atmospheric pressure.
 - b. Close the bag valve, open the pump coarse adjust valve, and evacuate the system again.
 6. Transfer the standards and field samples from each bag into the reaction bulbs as follows (Analysis of each standard and sample requires a set of three bulbs):
 - a. Close the pump coarse adjust valve, open the Tedlar bag valve, and let the system fill to atmospheric pressure.
 - b. Open the stopcocks to the reaction bulbs, and let the entire system come to atmospheric pressure.
 - c. Close the bulb stopcocks, remove the bulbs, record the room temperature and barometric pressure to nearest mm Hg.
 - d. Place the bulbs on the shaker table with their main axis either parallel to or perpendicular to the plane of the table top.
 - e. Purge the bulb-filling system with ambient air for several minutes between samples.
 7. Prepare a set of three bulbs containing colorimetric reagent but no CO as a reagent blank.
 8. Shake the samples for exactly 2 hr.
 9. Immediately after shaking or as quickly as possible, measure the absorbance of each bulb sample at 425 nm if CO is ≤ 400 ppm or at 600 nm if CO is > 400 ppm.
 - a. Use a small portion of the sample to rinse a spectrophotometer cell several times before taking an aliquot for analysis.
 - b. If one cell is used to analyze multiple samples, rinse the cell several times between samples with water.
 - c. Use water as the reference. Reject the analysis if the blank absorbance is > 0.1 .
- A pressure increase of ≥ 1 mm Hg indicates a leak.
- d. Measure the vacuum pressure to ± 1 mm Hg, and close the reaction bulb stopcocks.

10. Calculate the average absorbance for each set of standards (two sets of three required for each range). Plot a calibration curve absorbance vs concentration. Draw a smooth curve through the points. The curve should be linear over the two concentration ranges.
11. Reject the standard set if any of the individual bulb absorbances differ from the set mean by more than 10%.
12. Determine the CO concentration of each bag sample using the calibration curve for the appropriate concentration range.

C. Post-Test Leak-Check

Mandatory: Leak-check the bag according to FP 3b.

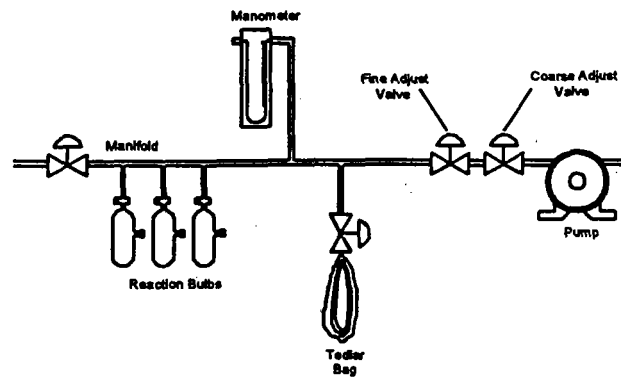


Figure L10A-1. Sample Bulb Filling System.

**LABORATORY DATA SHEET 10A
Carbon Monoxide**

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Spectrophotometer ID # _____ Date Last Calibration _____

Room Temp _____ °C Bar Press, P_b _____ mm Hg Analyst _____

Note: Analyze samples with CO <400 ppm at 425nm, samples with CO >400 ppm at 600nm.

| Sample No. | Sample ID # | Bulb No. | Bulb Vol., V _b (L) | Rgt Vol. in Bulb, V _r (L) | Bulb Vac Press, P _v (mm Hg) | Shaking Time (min) | Abs. vs water | Avg Abs, A | A _s See eqn below | C _g (ppm) |
|------------|-------------|----------|-------------------------------|--------------------------------------|--|--------------------|---------------|----------------|------------------------------|----------------------|
| | Blank | | | | | | | A _r | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | Std #1 | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | Std #2 | | | | | | | | | |
| | | | | | | | | | | |
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| | | | | | | | | | | |
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Moisture Content in Bag: If condensate is visible in bag, use room temperature and barometric pressure (LDS 10A) to calculate B_w; if not, use field temperature and barometric pressure (FDS 10A) to calculate B_w.

$$B_w = \frac{P_w}{P_b}$$

Run #1 Run #2 Run #3

B_w _____ _____ _____

| Rm Temp (°C) | V.P. of H ₂ O (mm Hg) | Rm Temp (°C) | V.P. of H ₂ O (mm Hg) |
|--------------|----------------------------------|--------------|----------------------------------|
| 4 | 6.1 | 18 | 15.5 |
| 6 | 7.0 | 20 | 17.5 |
| 8 | 8.0 | 22 | 19.8 |
| 10 | 9.2 | 24 | 22.4 |
| 12 | 10.5 | 26 | 25.2 |
| 14 | 12.0 | 28 | 28.3 |
| 16 | 13.6 | 30 | 31.8 |

$$A_s = \frac{(A - A_r) P_b}{(V_b - V_r) (P_b - P_v)}$$

- _____ Plot of calibration curve attached?
- _____ Each std abs ≤ ± 10% of the set mean?
- _____ Blank analyzed at the same wave length as samples and has absorbance of ≤ 0.1?
- _____ Post-test bag leak-check okay?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____
Analyst (Signature/Date)

Team Leader (Signature/Date)

SUMMARY SHEET 10B
Carbon Monoxide

| | | Run # 1 | Run #2 | Run #3 | Avg |
|---|----------|---------|--------|--------|---------|
| Client/Plant Name | | | | | FDS 10A |
| Job No. | | | | | FDS 10A |
| Sampling Location | | | | | FDS 10A |
| Run ID # | | | | | FDS 10A |
| Test Date | | | | | FDS 10A |
| Run Start Time | | | | | FDS 10A |
| Run Finish Time | | | | | FDS 10A |
| Net Traverse points | | | | | FDS 1 |
| Traverse Matrix (if rectangular) | | | | | FDS 1 |
| Net Run Time, min | θ | | | | FDS 10A |
| Sampling Rate, mL/min | Q_s | | | | FDS 10A |
| CO ₂ Concentration, fraction | F | | | | FDS 10A |
| Field Temperature, °C | t_f | | | | FDS 10A |
| Field Barometric Pressure, mm Hg | P_b | | | | FDS 10A |
| Average Injection Area | A | | | | LDS 10B |
| Average Response Factor | R | | | | LDS 10B |
| Room Temperature, °C | t_r | | | | LDS 10B |
| Lab Barometric Pressure, mm Hg | P_b | | | | LDS 10B |
| Bag Moisture Content | B_w | | | | LDS 10B |
| Cal Curve CO Concentration, ppm | C_g | | | | LDS 10B |
| Bag CO Concentration, ppm dry | C_b | | | | LDS 10B |
| Stack CO Concentration, ppm dry | C | | | | SS 10B |

$$C_b = \frac{A}{R(1 - B_w)}$$

$$C = C_b(1 - F)$$

LABORATORY PROCEDURE 10B
Carbon Monoxide**A. Equipment Preparation and Checks**

1. Obtain three standard gases with nominal CO of 20-, 200-, and 1,000-ppm CO in N₂ and standard CH₄ gas of 1,000 ppm in air.
2. Establish an appropriate carrier flow rate and detector temperature for the specific instrument used.
3. Calibrate the analyzer as follows:
 - a. Inject in triplicate each of the standard CO gases in step A1.
 - b. Calculate the average response factor (area/ppm) for each gas and the overall mean of the response factor values.
4. Analyze each new tank of carrier gas with the GC analyzer in triplicate to check for contamination.

5. Check the reduction catalyst efficiency as follows:

- a. Bypass the heated reduction catalyst, and analyze in triplicate the 1,000 ppm CH₄ gas to calibrate the analyzer.
- b. Repeat the procedure using 1,000-ppm CO with the catalyst in operation.
- c. Calculate the reduction catalyst efficiency.

B. Analysis

1. Purge the sample loop with sample, and then inject the sample.
2. Analyze each sample in triplicate, and calculate the average sample area (A).
3. Determine the bag CO concentration.

LABORATORY DATA SHEET 10B
Carbon Monoxide

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Gas Chromatograph ID # _____ Analyst _____

Room Temperature, °C _____ Barometric Pressure, P_b _____ mm Hg

Chromatograph Operation

| Parameter | Setting | (✓) | Parameter | Setting | (✓) |
|------------------------------------|---------|-----|--------------------------|---------|-----|
| N ₂ cylinder pressure | psig | | H ₂ flow rate | cc/min | |
| N ₂ flow rate setting | cc/min | | Oven temperature | °C | |
| N ₂ backflush flow rate | cc/min | | Injection port | °C | |
| Burner air supply | psig | | Detector | °C | |
| Burner air flow rate | cc/min | | FID stabilized? | | |
| H ₂ cylinder pressure | psig | | | | |

Calibration

| Sample ID# | Injection 1 Area | Injection 2 Area | Injection 3 Area | Average Area, A | Response Factor, R _i |
|--|------------------|------------------|------------------|-----------------|---------------------------------|
| Carrier Gas Blank Check | | | | | |
| Cylinder ID# _____ | | | | | |
| _____ CO concentration in the cylinder < 5 ppm? | | | | | |
| Reduction Catalyst Efficiency Check | | | | | |
| 1,000 ppm CH ₄ Certified value _____ | | | | | |
| 1,000 ppm CO Certified value _____ | | | | | |
| _____ CO response within ±5% of the certified gas value? | | | | | |
| Linearity Check | | | | | |
| 20 ppm CO Certified value _____ | | | | | |
| 200 ppm CO Certified value _____ | | | | | |
| 1,000 ppm CO Certified value _____ | | | | | |
| Average Response Factor (R) = | | | | | |

_____ Average response factor of each cal gas within ±2.5% of average response factor (R)?

_____ Relative standard deviation for each set of triplicate injection < ±2%?

