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Continuous Hydrocarbon Analysis of Diesel Emissions

1. **Scope**—The method presented is the current recommendation for the use of flame ionization detectors to determine the hydrocarbon content of diesel engine exhaust, or exhaust of vehicles using diesel engines, when operating at steady-state. The requirements of the associated sampling system and a general procedure for a continuous measuring method are presented.
 - 1.1 **Purpose**—This SAE Recommended Practice provides for the continuous measurement of the hydrocarbon concentration in diesel exhaust.
2. **References**—There are no referenced publications specified herein.
3. **Definitions**
 - 3.1 **Exhaust Emission**—Any substance emitted into the atmosphere from any opening downstream from the exhaust port of the combustion chamber.
 - 3.2 **Diesel Engine**—Any compression ignition internal combustion engine.
 - 3.3 **Steady-State Condition**—An engine operating at a constant speed and load and at stabilized temperatures and pressures.
 - 3.4 **Flame Ionization Detector**—A hydrogen-air diffusion flame detector that produces a signal proportional to the mass flow rate of hydrocarbons entering the flame per unit time.
 - 3.5 **Hydrocarbons**—All organic materials including unburned fuel and combustion by-products present in the exhaust which are detected by the flame ionization detector.
 - 3.6 **Parts Per Million Carbon**—The mole fraction of hydrocarbon measured on a methane equivalence basis.
 - 3.7 **Calibration Gas**—A gas used to establish instrument response. Concentration known to $\pm 1\%$ accuracy traceable to The National Institute of Standards and Technology (NIST) Reference Materials where available.
 - 3.8 **Span Gas**—A gas used routinely to check instrument response. Concentration traceable to calibration gases.
 - 3.9 **Air Zero Gas**—Air containing less than 2 ppm hydrocarbon on a methane equivalence basis.

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3.10 Abbreviations Used

°C	—Degrees Celsius
CHA	—Continuous hydrocarbon analyzer
conc	—Concentration
cm	—Centimeters
diam	—Diameter
Exh	—Exhaust
FID	—Flame ionization detector
g	—Gram(s)
h	—Hour
H ₂	—Hydrogen
HC	—Hydrocarbon
He	—Helium
Hg	—Mercury
ID	—Internal diameter
kg	—Kilogram
L	—Liter(s)
m	—Meter(s)
max	—Maximum
min	—Minimum, minute(s)
mL	—Milliliter(s)
kW	—Kilowatt
N ₂	—Nitrogen
O ₂	—Oxygen
OD	—Outside diameter
ppm	—Parts per million
ppmc	—Parts per million carbon
s	—Second(s)
%	—Percent

4. **Section**—This report is divided into the following sections:

3. Definition of Terms and Abbreviations
5. Equipment
6. Instrument Operating Procedures
7. Engine Test Procedure
8. Information to be Recorded
9. Calculations, Data Analysis, and Report
10. Supplementary Discussion

5. **Equipment**

5.1 **Instruments**—The Continuous Hydrocarbon Analyzers (CHA) recommended for measuring unburned hydrocarbons in diesel exhaust can be of a positive pressure burner type (Figure 1) or a reduced pressure burner type (Figure 2). In both systems, a fraction of the engine exhaust stream flows through a heated sampling line and filters to the sample pump inlet. The sample stream is split with a fraction of the exhaust stream sample diverted to the burner. The sample split is made upstream of the pump inlet in the reduced pressure system and downstream of the pump outlet in the positive pressure system. The burner in both systems (Figures 1 and 2) is a flame ionization detector; the system components are described in 5.2. The system employed should be capable of measuring hydrocarbons over a range of 10 to 6000 ppmc and have the ability to follow rapid changes in hydrocarbon concentration. The response of the total sampling system to diesel exhaust should be at least 90% of full scale within 20 s for a 100% of full scale step input to the system.

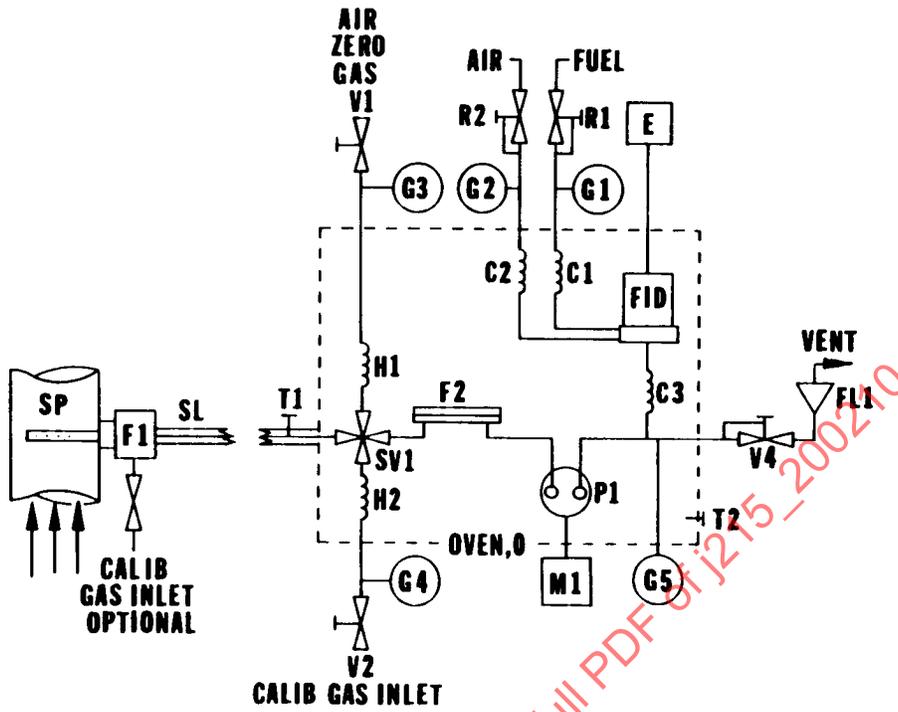


FIGURE 1—CHA POSITIVE PRESSURE BURNER

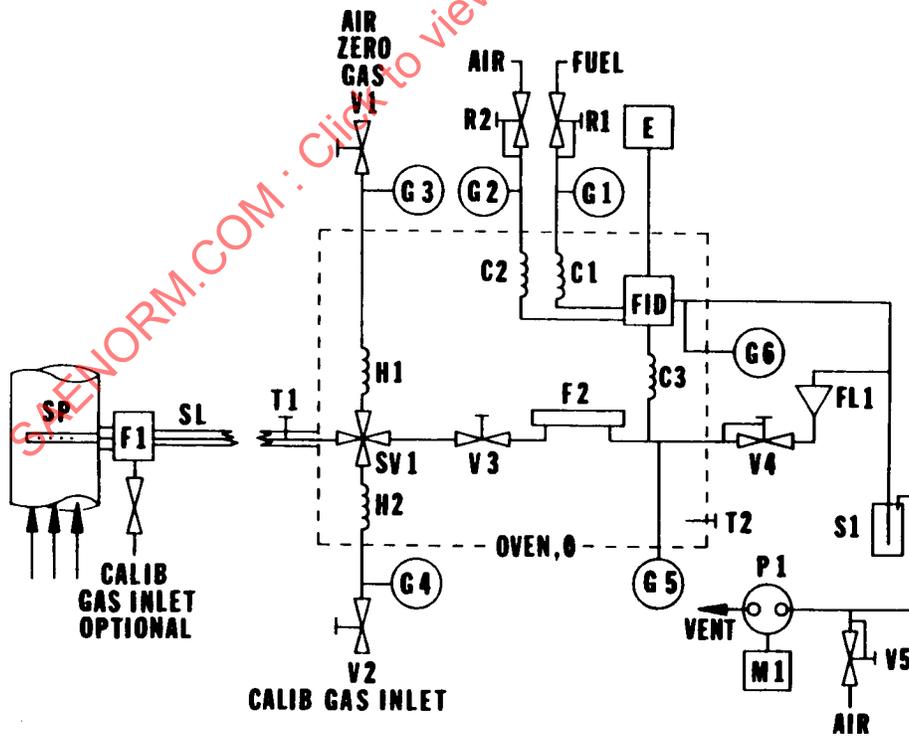


FIGURE 2—CHA REDUCED PRESSURE BURNER

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5.2 Component Description—The following components are utilized in the CHA of Figures 1 and 2. All parts are common to both systems unless otherwise stated.

5.2.1 Pressure regulators, R1 and R2, pressure gages, G1 and G2, and capillary tubes or restrictors, C1 and C2, to control air and fuel flows to CHA detector burner. C1 and C2 should be maintained at constant temperature ± 2 °C.

OPTIONAL—Flow controllers and flowmeters may be used in place of R1, R2, G1, and G2.

5.2.2 Flame ionization detector, FID, capable of operating in the range of 175 to 200 °C.

5.2.3 Electrometer, E, coupled to a recorder, meter, or other comparable readout device may be used.

5.2.4 Constant temperature oven, O, for detector and sampling system components, capable of operating in the range of 175 to 200 °C and holding temperature ± 2 °C.

5.2.5 Stainless steel probe, SP, to obtain sample from the exhaust system. A closed end, multihole static probe extending at least 80% across the exhaust pipe is recommended. The total area of the holes should be equal to or less than the cross-sectional area of the probe. A typical hole ID used is 0.15875 cm. The probe location should be governed by the purpose of the test. For routine measurements, the probe should be located in the exhaust line at a distance of 1 to 3 m from the exhaust manifold outlet flange or the outlet of the turbocharger. Longer distances can be used if the results are equivalent to those obtained at the specified 1 to 3 m distance.

5.2.6 Heat prefilter, F1, in line to remove soot and reduce acoustical effects in sample flow (optional). Spun Pyrex¹ glass is one example of a suitable filter material.

5.2.7 Sampling line, SL, must be heated to maintain the gas temperature to ± 10 °C of the oven compartment temperature and never less than 175 °C during sampling. Construction of the line should be of stainless steel and/or Teflon.¹

A 0.635 cm OD or 0.9525 cm OD line is recommended. A response time of 20 s or less is desired. To test response, inject span gas sample at the stack inlet (F1) and measure the time elapsed before a deflection of 90% of stabilized response is observed on the readout meter or recorder. The gases used to check response should give a deflection of at least 50% of full scale.

5.2.8 Temperature readout, T1, of sample stream entering oven compartment (minimum temperature 175 °C).

5.2.9 Suitable valving, SV1, for selecting sample, span gas, or air zero gas flow to the system. The valve(s) should be in the oven compartment or heated.

OPTIONAL—Use external pump to supply sufficient heated clean air to flush the entire system when not calibrating or sampling.

5.2.10 Valves, V1 and V2, and gages, G3 and G4, to regulate calibration gas and air zero gas.

5.2.11 Coils, H1 and H2, to preheat calibration gas and air zero gas.

OPTIONAL—Introduce gases into sampling line near probe inlet.

5.2.12 Filter, F2, to remove particulates. A 7 cm diam glass fiber type filter disc or equivalent is suitable. Filter should be readily accessible and changed daily or more frequently as needed.

1. Registered trademark.

- 5.2.13 Regulating valve, V3, (reduced pressure system only) to control pressure in sample line.
- 5.2.14 Sample pump, P1, and motor, M1. In the positive pressure system, the pump head must be heated to oven temperature. In the reduced pressure system, the pump and motor are outside the oven. The pump used in the positive pressure system should not affect the emission concentration or composition.
- 5.2.15 Capillary tubing or restrictor, C3, to control sample flow to the detector.
- 5.2.16 Pressure gage, G5, to measure in sample line. Pressure tap should be located near the capillary line (C3) takeoff and designed to minimize velocity effects.
- 5.2.17 Pressure regulator valve, V4, to control pressure in sample line and flow to detector. This valve could be connected across the detector in the vacuum system. The valve should be maintained at a temperature above the dew point.
- OPTIONAL—Use a fixed restrictor and vary the pump speed to control the pressure.
- 5.2.18 Vacuum gage, G6, (reduced pressure system only) to measure pressure in detector or cannister.
- OPTIONAL—Combine with G5 and use one gage to measure pressure drop across detector
- 5.2.19 Surge tank, S1, (reduced pressure system only) to stabilize flow tank, if required, should be sized for system. A 1 to 2 L tank is suggested for 10 L/min sample flow.
- 5.2.20 Flowmeter, FL1, (reduced pressure system) to measure sample bypass flow. Maintain above dew point. Flowmeter is optional on positive pressure system.
- 5.2.21 Vacuum regulator, V5, (reduced pressure system only) to control vacuum in detector.
- 5.2.22 Oven temperature readout, T2, thermocouple or equivalent.

6. Instrument Operating Procedures—Follow the instrument manufacturer's startup and operating procedure for the particular type CHA. In addition, the following minimum calibration and instrument checks should be included.

6.1 Calibration and Instrument Checks

6.1.1 INITIAL—The following instrument checks may have been determined by the manufacturer or user. If not, they should be evaluated prior to instrument use.

6.1.1.1 Optimize Detector Response

- a. Set burner fuel and air settings as prescribed by the manufacturer. Ignite the burner. Set sample flows recommended by the manufacturer.
- b. Set the oven temperature at 175 to 200 °C. Allow system to reach equilibrium. This usually takes at least 1/2 to 2 h.
- c. Determine the optimum burner fuel flow for maximum response. Introduce a constant continuous concentration of propane in N₂. Use about 500 ppmc hydrocarbon concentration or a concentration that would be a mid-point in the normal operating range. Vary burner fuel flow and determine peak response. Select an operating flow that gives maximum response and the least variation in response with minor flow variations. Normally, there is a plateau in the region of peak response Figure 3. Use best judgment in selecting optimum fuel flow.

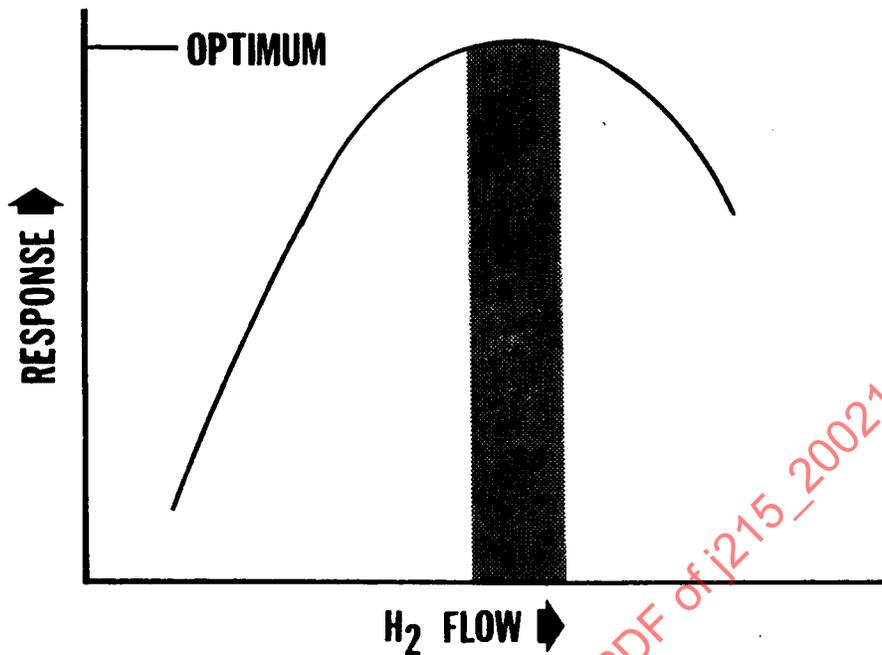


FIGURE 3—EFFECT OF HYDROGEN FLOW

- d. Determine optimum air flow. Set burner fuel flow as determined in 6.1.1.1(c) and vary air flow. Although less critical than burner fuel flow, nonoptimized conditions may reduce quantitative accuracy. If air flow is too low, response is low. High air flow may result in increased noise. A typical curve is shown on Figure 4. Select desired air flow and, if it is significantly different than that used in 6.1.1.1(c), repeat step (c).
- e. Measure optimum flows accurately and record.

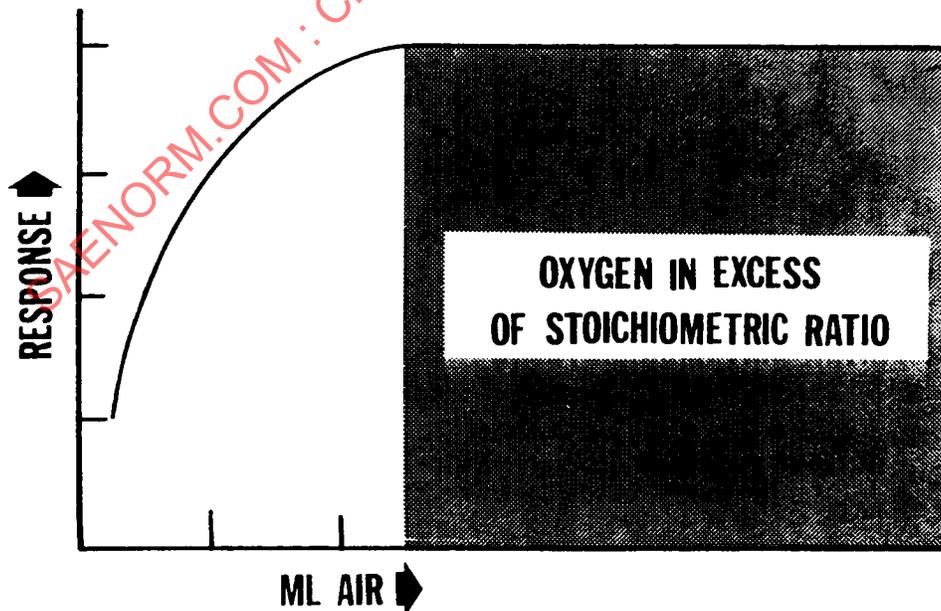


FIGURE 4—EFFECT OF AIR FLOW

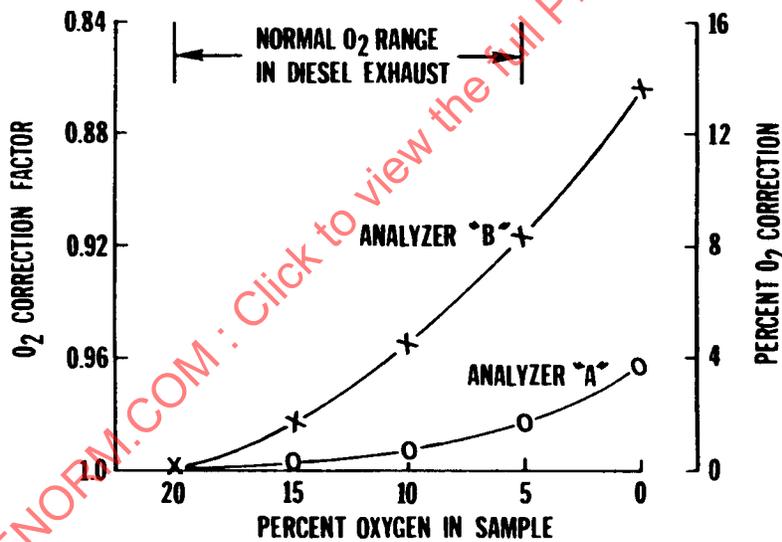
6.1.1.2 Determine Oxygen Response Curve of CHA—Variation in oxygen concentration in diesel exhaust (excess air) causes variation in detector response. The magnitude of this oxygen effect has to be determined and minimized.

- a. Ignite burner and set flows as determined in 6.1.1.1. Set oven temperature as in 6.1.1.1 and allow at least 1/2 h after heatup for system to reach equilibrium.
- b. Introduce air zero gas and zero the analyzer and span the analyzer with propane in air.
- c. Determine oxygen response by introducing propane calibration gases in the following carrier gases: 100% N₂, 95% N₂/5% O₂, 90% N₂/10% O₂, air.

The concentration level of the propane should equal the expected upper HC level, or a nominal concentration of 350 ppm ± 50 ppm. The HC concentration should be known within ±1% of the true value. (See 10.1.) The oxygen values of the mixtures should be 5% ± 1%, 10% ± 1%, and air.

Recheck zero and gain after each calibration gas is used. If either has changed more than 1% of full scale deflection rezero and/or adjust gain and repeat the test.

- d. Using propane in air as the baseline for O₂ correction, plot a curve of oxygen correction versus the percent of oxygen in the sample Figure 5.



Analyzer A — Oxygen correction not needed
 Analyzer B — Oxygen correction needed*

$$* \text{Correction factor} = 1 + \frac{\text{Calib. bottle conc.} - \text{conc. measured}}{\text{Calib. bottle conc.}}$$

FIGURE 5—FID OXYGEN INTERFERENCE

If correction is less than ±2% over the normal operating range of the diesel, no O₂ correction need be applied to the observed HC concentrations.

If the correction is greater than ±2%, apply an O₂ correction to all measured values as follows:

$$\text{Corrected ppmc} = (\text{observed ppmc}) \times \text{O}_2 \text{ correction} \quad (\text{Eq. 1})$$

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- e. Check the effect of O₂ by using a propane concentration typical of the expected lower HC level that will be encountered during engine tests, or about 100 ppmc, whichever is smaller. If the measured results are significantly different from the known values, apply the O₂ correction factor determined in 6.1.1.2(d).
- f. If the oxygen response correction in 6.1.1.2(d) and (e) is greater than 4% over the normal O₂ range encountered in diesel exhaust (Figure 5), see 10.2.

6.1.1.3 Determine Linearity of CHA

- a. Ignite burner. Set flows as determined in 6.1.1.1 and 6.1.1.2.
- b. Set temperatures as in 6.1.1.1 and allow at least 1/2 h after heatup for system to reach equilibrium and set instrument gain with propane in air.
- c. Using propane in air, vary the HC concentration over the operating range using HC concentrations of about 1/3, 2/3, and full scale as a minimum. If more than one attenuation range is used, establish linearity by checking at least two points other than zero for each additional range used. If the response is not linear within $\pm 2\%$ of full scale, check calibrations and/or analyzer.

6.1.1.4 System Operating Temperature

- a. The initial operating temperature recommended is 175 to 200 °C.
- b. The optimum system operating temperature should be checked as discussed in 10.3.

6.1.2 AND 6.1.3 SAMPLE PRESSURE CONTROL SYSTEM CHECKS—The accuracy of the sample back pressure regulator and control system should be checked by connecting a typical span gas and adequate pressure gage to the sample inlet connection on the instrument module or in place of the sample probe. Vary the span gas supply pressure over the range anticipated at the exhaust stack probe location. If the response varies by more than 2%, adjust or replace pressure regulator valve V4 to maintain constant line pressure (indicated on G5).

6.1.2 MONTHLY—The following checks are to be made monthly or more frequently if there is any doubt regarding accuracy of HC values.

- 6.1.2.1 Ignite burner. Set air, fuel, and sample rates as determined in 6.1.1.1 and 6.1.1.2.
- 6.1.2.2 Set system temperatures as determined in 6.1.1.4. Allow at least 1/2 h after heat-up for the system to come to equilibrium.
- 6.1.2.3 Introduce air zero gas and zero the analyzer.
- 6.1.2.4 Check oxygen effect on response by introducing calibration gases of propane in air, propane in nitrogen, and propane in 90% N₂/10% \pm 1% O₂. The hydrocarbon concentration should be known to within $\pm 1\%$ of true value.
- 6.1.2.5 Recheck zero after measuring each calibrating gas. If zero varies by more than 1% of full scale, rezero and repeat step 6.1.2.4.
- 6.1.2.6 Compare oxygen response values with previous curves. Any significant ($\pm 10\%$) change in response reflects a change in the burner operating characteristics; that is, air, fuel, or sample flow rates. Check for leaks or plugged capillaries and remeasure flows. If change in response cannot be resolved, establish a new oxygen response curve as per 6.1.1.2.
- 6.1.2.7 Check calibration curve or response data as per 6.1.1.3(c).

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6.1.3 DAILY—Prior to daily testing, carry out the following:

6.1.3.1 Ignite burner, set air, fuel, and sample rates as determined in 6.1.1.1 and 6.1.1.2.

6.1.3.2 Insert clean filters.

6.1.3.3 Set system temperatures as determined in 6.1.1.4. Allow at least 1/2 h after heat-up for the system to come to equilibrium.

6.1.3.4 Introduce air zero gas and zero the analyzer.

6.1.3.5 Introduce HC span gas (propane in air) appropriate to anticipated operating range. (See 10.4). Check agreement with calibration curve. (NOTE—Sample flow for air zero gas and calibration gas should be the same as exhaust sample flow.) If this value does not agree with calibration curve, adjust instrument gain so that response agrees with calibration curve. If sufficient time has been allowed to stabilize the instrument and the change in gain or response is more than 5% of full scale, then recheck oxygen interference and linearity per 6.1.1.2 and 6.1.1.3(c).

6.1.3.6 *Conduct analyses*—Recheck zero after each analysis. If zero changes by more than 2% or more of measured value, rezero and repeat test.

CAUTION—Do not mistake HC hangup for zero change. Check for contamination in line and probe at normal instrument operating conditions by drawing in either clean air or air zero gas. A reading of 20 ppmc or more indicates that the probe and/or line should be cleaned or replaced.

6.1.3.7 At the conclusion of the test, backflush the system to clean out the sampling line preferably with hydrocarbon-free air or nitrogen.

7. Engine Test Procedure—The following test procedure is recommended for emission measurements at steady-state operating conditions. The engine operating cycle is not dictated by this procedure and the engine break-in, pretest conditioning, and measurement procedure may be modified depending on the purpose of the test; that is, emission certification or routine laboratory development test.

7.1 Engine Break-In Procedure—The engine shall be run-in according to the manufacturer's recommendation.

7.2 Emission Measurement Procedure

7.2.1 Connect sample line to instrument and check for leaks by blocking off end of sample line or probe. A leakage rate greater than 1% of the system flow recommended by the manufacturer shall be corrected before proceeding. Install probe in exhaust system and connect to sample line if not done so, to check for leaks. Check for contamination in line at normal instrument operating conditions (see 6.1.3.6).

7.2.2 Start the engine and warm it up. Complete warm-up at rated speed and full load for 10 min or until all temperatures and pressures have reached equilibrium.

7.2.3 Operate for at least 20 min in each mode for emission stabilization, allowing last 5 min for emission measurement.

7.2.4 Measure hydrocarbon emission as follows:

7.2.4.1 Follow daily instrument procedure (see 6.1.3.)

7.2.4.2 Analyze exhaust for at least 5 min during each mode.

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7.2.4.3 Check and reset zero and span after each mode; if either changed more than 2% of measured response, repeat the mode.

7.2.5 CHART READING—HC determination as follows:

7.2.5.1 Locate last 3 min of each test and average the chart reading over this 3 min period.

7.2.5.2 Determine the concentration of hydrocarbons as ppmc at each point by Equation 2 or the calibration curve from 5.1.1.1(c).

$$\text{HC conc.} = \frac{\text{Measured response}}{\text{Span gas response/ppmc in span gas}} \quad (\text{Eq. 2})$$

7.2.5.3 Correct concentration obtained in 7.2.5.2 for oxygen effect as determined in 6.1.1.2.

7.2.5.4 Additional mass calculations may be required, depending on the purpose of the test.

8. **Information to be Recorded**—The following information should be included as part of the recorded data for each test performed.

8.1 Test number

8.2 Engine or vehicle tested:

- a. Identification number.
- b. Brief description including type precombustion or direct inject (PC or DI), naturally aspirated or turbocharged (NA or TC), 2- or 4-cycle, bore, and stroke.

8.3 Date.

8.4 Instrument operator and test engineer or vehicle operator.

8.5 Starting and finishing time.

8.6 Analyzer identification.

8.7 Ambient temperature, start, and finish of testing.

8.8 Number of engine conditions tested.

8.9 Atmospheric pressure, start, and finish of testing.

8.10 Relative humidity, start, and finish of testing.

8.11 Fuel used, identification number, and type (No. 1 or No. 2 diesel, etc.).

8.12 Lube oil used, identification number, and type.

8.13 Oven temperature.

8.14 Sample line temperature.

8.15 Burner fuel and flow rate and/or pressure.

8.16 Air flow rate and/or pressure.