



SURFACE VEHICLE STANDARD

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FTIR Gas Analyzer Performance Evaluation/Qualification for Automotive Testing

RATIONALE

Over the past several decades, manufacturers have made great strides in reducing emissions and improving fuel efficiency of the modern internal combustion engine. Integral to these improvements has been the use of various emissions monitoring equipment to test, verify, and certify the improvements. As industry compliance standards become ever more stringent, the need for new capabilities has forced automotive emissions engineers to utilize alternate technologies for monitoring gaseous emissions. The Fourier Transform Infrared (FTIR) gas analyzer is one example of a technology that shows much promise in meeting the needs of the modern emissions engineer. As use of FTIR analyzers become more widespread in both R&D and certification, there is a growing need for an SAE standard to provide recommended practices and minimum performance standards for the analyzer in various automotive related applications.

This document is to be used as a guideline for the use of Fourier Transform Infrared (FTIR) spectrometry in engine exhaust certification testing as well as engine and vehicle development activities. The goal is to create a test protocol that follows the concepts and procedures as laid out in EPA document 40 CFR Part 1065 for engine emissions testing but applied to a multi-component FTIR analyzer. This document contains instructions on how to perform and certify the various stages of the verification testing as well as what needs to be done after major maintenance and other interruptions as defined by Part 1065. By using this procedure one can verify the ability of the FTIR and sampling system to measure multiple gases in the presence of interfering constituents such as high concentration components like water and carbon dioxide. The FTIR system chosen for this application should have the necessary sensitivity and linearity across the full range of concentration values for each of the selected exhaust species that will be seen for each particular engine test encountered. The full range is defined by the specific EPA standard for the test or the end user requirements whichever is greater.

Since the FTIR can monitor multiple components simultaneously, self-validating procedures as described below are implemented and utilized to initially validate the technology, as well as provide routine tests that ensure compliance after implementation of the integrated system. Because all the infrared (IR) active exhaust components are measured at the same time without any chemical reaction any potential interference observed can normally be reduced or eliminated by modifying the analytical method resident in the software and/or by reducing the internal pressure of the gas cell. If interference is noted during the verification process, all the data collected can be reprocessed against an updated method (provided the overall system pressure and temperature remains the same) to demonstrate that the interference has been reduced or eliminated to acceptable levels.

Once the FTIR validation has been completed, there will be sufficient data to support the use of FTIR in order to meet the existing 1065 analyzer performance requirements.

Note that it is the responsibility of the user to ensure that the equipment used for certification testing is approved by the appropriate certification authorities.

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1. SCOPE

The scope of this document focuses on the tests required by EPA to validate the performance of the FTIR system following the section in the Code of Federal Regulations Part 1065 (40 CFR 1U.1065 and hereafter referred to as "EPA Part 1065") on the guidelines and performance criteria for various regulated gases. This document focuses on the use of continuous emissions sampling for both engine and vehicle testing. Future addenda will be needed to cover bag and other sampling techniques.

Gas components that do not currently have performance criteria but may soon be regulated are noted and EPA suggestions as to what should be required are applied. This will help ensure that the FTIR will be recognized as a valid and alternative tool for engine exhaust emissions testing. Components in engine exhaust that are specifically called out in this document include: carbon monoxide (CO), carbon dioxide (CO₂), oxides of nitrogen (NO, NO₂, and N₂O), ammonia (NH₃), methane (CH₄), ethane (C₂H₆), and formaldehyde (H₂CO). Others (for example, hydrocarbons for non-methane, non-ethane hydrocarbons (NM NE HC) or volatile organic compounds (VOC)) may be added as needed by following this standard operating procedure and determining the certification requirements from EPA or other governing bodies as required.

This SAE standard references the specific sections in the EPA Part 1065 certification test requirements (current as of April 23, 2014) for all of the steps required to provide proof of performance. The document provides the user a guideline for the FTIR gas analyzer as applied to engine or vehicle emissions testing. This document is applicable to a wide range of fuels including, but not limited to, diesel, gasoline, natural gas, liquid petroleum gas (LPG), blends, bio-fuel, and bio-fuel blends.

While this document specifically references 191 °C as the temperature used for performance validation, this may not always be the case. The temperature specification applies specifically to the raw gaseous emissions that have not been diluted. If the analysis is to be performed upon a diluted gas stream where the emission gas is at a lower temperature, then the analyzer can also be run at a lower temperature, provided that the components of interest do not condense or react prior to reaching the analyzer.

1.1 Purpose

The purpose of this document is to provide a set of guidelines and test procedures for the use of FTIR systems for engine emission testing as defined by EPA Part 1065. Each FTIR system manufacturer will need to provide a set of procedures to the end user that when performed prior to the emission testing would demonstrate and confirm that the FTIR has maintained its calibration and is functioning properly. Those procedures are not described below because they are dependent upon the instrument manufacturer.

2. REFERENCES

2.1 Applicable Documents

The following publications form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest issue of SAE publications shall apply.

2.1.1 EPA Publications

EPA Title 40: Protection of Environment, Chapter 1, Subchapter U, Part 1065: Engine Testing Procedures.

eCFR current as of December 2013 - United States Environmental Protection Agency test procedure that applies to engines and vehicles with engines as described in detail within the regulation including locomotive; heavy duty on road; non road, stationary, and marine diesel; large and small non road and stationary spark or compression ignition engines; vehicles such as automobiles and motorcycles; and others as stated in the final document.

EPA Title 40: Protection of Environment, Chapter 1, Subchapter D, Part 136 Appendix B - Definition and Procedure for the Determination of the Method Detection Limit.

Federal Register Volume 76, No. 179/Thursday, September 15, 2011/Rules and Regulations Updates for PART 1065 - Engine Testing Procedures: Pages 57437 - 57470.

3. DEFINITIONS

3.1 Terms

3.1.1 ELECTRONIC CODE OF FEDERAL REGULATIONS

eCFR is the latest revisions of the EPA methods are stored and available for public access.

3.1.2 FLOW WEIGHTED MEAN CONCENTRATION

FWMC is the concentration value that is expected during the actual engine testing. Flow-weighted mean is the mean of a quantity after it is weighted proportional to the corresponding flow rate. For example, if a gas concentration is measured continuously from the raw exhaust of an engine, its flow-weighted mean concentration is the sum of the products of each recorded concentration value times its respective exhaust flow rate, divided by the sum of the recorded exhaust flow rates. Refer to EPA Part 1065.602 for information needed to estimate as well as how to calculate flow-weighted means. While EPA Part 1065 references FWMC, this document will reference the concentration range that is specified by the specific instrument manufacturer in order to ensure that the FTIR will span the ranges that will be required for each particular engine test.

3.1.3 RANGE

Range as defined in this document is the concentration values in parts per million (ppm) or percent (%) on a volume basis, listed from low to high over which the FTIR instrument is valid. The range is depicted in form of [0 - 100] in this document. Multiple ranges can be used in the case of a widely varying process and the performance verification must be tested at each of the specified ranges if they are to be used together.

3.1.4 RISE TIME

Time it takes the FTIR response to rise/increase from 10% up to 90% of the final reading ($t_{10} - t_{90}$).

3.1.5 FALL TIME

Time it takes the FTIR response to fall/decrease from 90% down to 10% of the final reading ($t_{90} - t_{10}$).

3.1.6 SIGNAL TO NOISE RATIO

SNR is a combination of signal strength and detector noise. Since FTIRs are detector noise limited, the noise component is generally attributed to the detector which is constant for constant time, so by increasing the signal or light intensity there is then a direct increase in SNR.

3.1.7 TRACER GAS

A gas component that is used as an "internal standard" to verify that the gas sample is reaching the FTIR as well as to provide accurate dilution rates, if needed. This component is generally not present in the emissions being tested or is present in very low concentrations and it must not be retained by the sampling system. Examples include SF₆, CH₄, NO, and CO₂.

3.1.8 STANDARD

The use of the term "standard" in EPA Part 1065 refers to the particular EPA standard that provides the regulated species as well as the maximum limit for the type of engine that is being tested. For instance, refer to 40 CFR Part 86 for light duty (passenger vehicles), heavy duty engines, light duty trucks, etc. The relevant emission values are defined by the actual standard part for each category.

3.1.9 STANDARD USAGE FLOW RATE

Refers to the sample flow rate going to the FTIR analyzer.

3.2 Abbreviations

The abbreviations used in the document have the following meanings in both capital and lower case.

AU	= Absorbance Unit
CFR	= Code of Federal Regulations
eCFR	= Electronic Code of Federal Regulations
FWMC	= Flow Weighted Mean Concentration
L/min	= Litres per Minute
NONMHC	= Non-Oxygenated Non-Methane Hydrocarbons
SEE	= Standard Error of the Estimate
SNR	= Signal to Noise Ratio

4. INSTRUMENTATION

4.1 Gas Blending System

A gas blending system consists of either: (1) a gas divider which has fixed intervals for blending with a matrix gas, or (2) a series of mass flow controllers and/or fixed orifices.

4.2 Fourier Transform Infrared Spectrometer (FTIR)

The FTIR must be capable of at least 1 Hz data acquisition. Contact the instrument manufacturer or use published data to determine what the differences are between instrument detectors, other components, as well as different instrument manufacturers to ensure the FTIR will meet the needs of the test.

4.3 Calibration Cylinders

- Certified dual component cylinders for calibration linearity testing is a single analyte in N₂ or air balance gas as described by EPA (see reference later in document).
- Certified multi-component cylinder is a mixed blend of analytes (as well as a reference tracer gas component) in N₂ or air balance as described by EPA (see reference later in document).

4.4 Extractive Techniques

In both cases shown below, it is prudent to install a heated filter at or near the probe end to reduce the amount of particulate matter or volatiles condensing in the lines or on the FTIR mirrors.

4.4.1 Pushing Gas into the Analyzer

Example schematic layout of sample extraction via pushing the gas sample through the FTIR. Blue lines indicate no heating required, while red lines require heating to 191 °C ± 5 °C. See Figure 1.

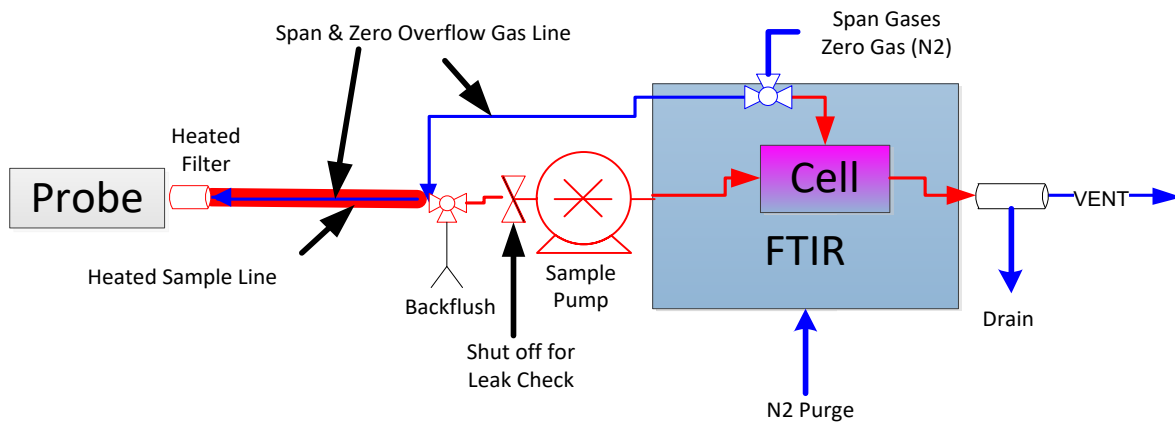


Figure 1 - Sample extraction via pushing of the gas

4.4.2 Pulling Gas into the Analyzer

Example schematic layout of sample extraction via pulling the gas sample through the FTIR. Blue lines indicate no heating required, while red lines require heating to $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. See Figure 2.

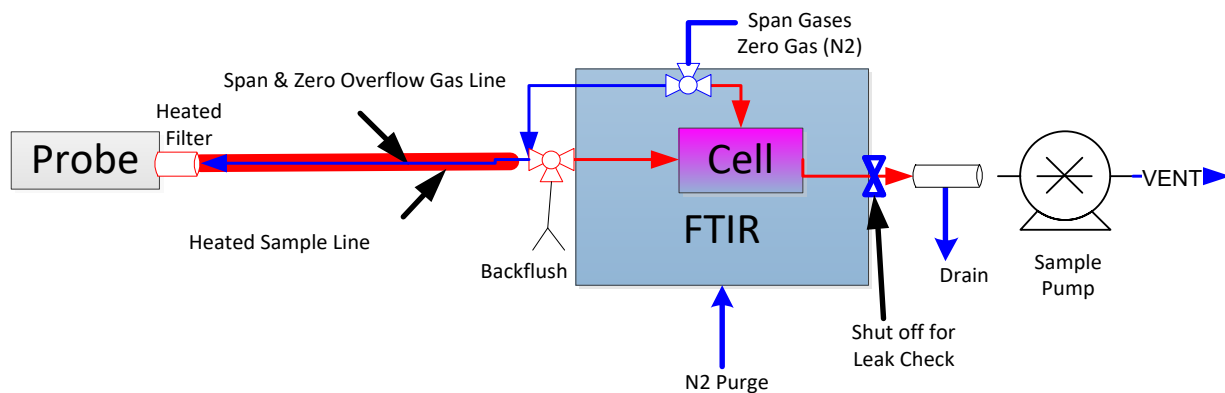


Figure 2 - Sample extraction via pulling of the gas

4.5 Experimental Considerations

4.5.1 FTIR Calibrations are Pressure Sensitive

- Pushing a sample through the FTIR allows for the internal pressure to be at or near ambient pressure.
- Pulling a sample through the FTIR lowers the overall internal pressure.
- In order to reduce calibration errors due to pressure, the gas reaching the FTIR gas sampling cell should be within $\pm 10\%$ of the pressure at which the calibration method was created.

4.5.2 FTIR Calibrations are Temperature Sensitive

In order to reduce calibration errors due to temperature, the gas reaching the gas cell should be at $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, or within $\pm 5\text{ }^{\circ}\text{C}$ of temperature at which the calibration was created.

4.5.3 Span Gas Line

The span and zero overflow gas line is shown above as a dual core heated line but the lines can be completely separate.

4.5.4 Standard Sampling Component Considerations

All of the mechanical components required to deliver a hot/wet sample to the FTIR without loss of any engine exhaust emissions components as well as components required for verification testing, such as heated lines, filter, pump, probe, by-pass, flushing, etc.

- a. Low flow systems as referenced herein are for flow rates ≤ 30 L/min while high flow systems reference flow rates > 30 L/min.
- b. Avoid all cold spots prior to the gas sample entering the FTIR and maintain the gas temperature within $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.
- c. In order to reduce analysis errors, it is suggested that the gas cell temperature should be kept at $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ or within $\pm 5\text{ }^{\circ}\text{C}$ of the calibration temperature.
- d. In order to reduce analysis errors, it is suggested that the gas cell internal pressure should be kept within $\pm 10\%$ of the calibration method pressure.

Note that for gas components with very narrow lines such as CO and CO₂, the gas cell pressure might need to be held to within $\pm 5\%$ of the calibration method in order to reduce calibration errors.

4.5.4.1 Particulates in the Gas Stream

- a. The particulate filter location can be either inside or outside of the analyzer, but it must be heated.
- b. To avoid more frequent gas cell cleanings, place a second heated filter at or near entrance to the FTIR.

4.5.4.2 Filter Considerations

- a. Filter must be used to avoid particulate matter or volatiles collecting on the optics of the gas cell.
- b. Must be heated.
- c. Must not retain any components that are to be certified by the FTIR.
 1. Filter and filter material should be tested to ensure minimum adsorption of the component of interest by running the sampling system and measuring the system response time.
 2. Sample system validation for component retention must be performed as part of the system performance tests as listed below in this document.
 3. Must allow for maximum throughput to avoid sample retention.
 4. Borosilicate glass or quartz is generally a good filter material to use, especially when working with NH₃ analysis.
- d. Must be changed on a regular basis using good engineering practice.
 1. For example, monitoring the pressure or flow rate across the sample train on a daily basis prior to use is a good way to determine if the filters are plugged.
 2. If the pressure or flow rate drops by more than 10% of the initial set-up value, then replace the filters.
- e. Coarse filter placed at or near the probe.
 1. Greatly reduces particulate matter deposition within the lines.
 2. Need to monitor flow rate or pressure closely to determine clogging of filter.
 3. Should be at least 1 μ pore size or smaller.

- f. Fine filter placed at or near the FTIR or pump (if on the inlet of the FTIR).
 - 1. Greatly reduces particulate matter deposition on the FTIR gas cell mirrors and windows.
 - 2. Requires more frequent sample line backflush or cleaning.
 - 3. Need to monitor retention of any of the components as particulate matter builds up.
 - 4. Should be at least 0.1 μ pore size or smaller to reduce the deposition of particulate matter on the gas cell windows.
- g. Use good engineering judgment with respect to sizing the particulate filter.
 - 1. Contact the instrument manufacturer for input on system requirements. Too coarse of a filter will require more frequent cleaning of the analyzer gas cell.

4.5.4.3 Sample Line Considerations

- a. Maintain gas sample temperature within $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ at all times to avoid condensation of sample gas component.
- b. Line material must minimize component retention as well as avoid reactions or diffusion.
 - 1. Stainless steel (SS) electropolished, SS corrugated, SilcoNert™ coated and Per-Fluro Alkoxy (PFA) are the preferred line material when working with components such as ammonia that have a tendency to adhere to the walls of the sampling lines.
 - 2. Lines should be cleaned on a regular basis to avoid particulate matter build up which can result in reaction with the gas molecules that are being monitored.
- c. Flow rates should be high enough to ensure that the analyte does not adhere to the sample line wall.
- d. Should be as short as possible to reduce sample delay time.

4.5.4.4 Sample Pump Considerations

- a. Use oil-free pumps and make sure to recondition the pump following the pump manufacturers recommendations for maximum run time.
- b. Wetted material should be stainless steel, Teflon, Teflon coated, or ceramic to avoid component retention.
- c. If sample pump is placed upstream of FTIR:
 - 1. The pump head must be heated to $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.
 - 2. A fine filter can be used at the inlet side of the pump.
 - 3. Heated line must connect the outlet of pump to FTIR.
- d. If sample pump is placed downstream of FTIR:
 - 1. If the pump head is not heated, then a coalescing filter or some other configuration is needed to make sure all condensing liquids are removed before the gas stream enters the pump.
 - 2. If the pump is an air jet type, then a coalescing filter is only required if additional dry analyzers are included in the stream or if a flow measurement is made just upstream of the air jet exhaust.

5. INSTRUMENT FUNCTION VALIDATION PERFORMANCE

This section is based upon the instrument manufacturer's suggested time scale, but at a minimum would need to be performed and documented upon initial installation of the equipment and after any maintenance performed which would affect the sample train or the FTIR light path.

5.1 Validate the System Hardware Parameters

- a. The FTIR and sampling system must be powered up and running properly.
 1. The detector must be cooled to the manufacturer's specified running temperature, the FTIR sample cell and the sampling system and lines must be heated to $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.
 2. Dry N_2 or zero air must be flowing through the gas cell using a span port if one is available or a diverter valve placed at the outlet of the sample pump or gas cell inlet.
 3. Use the manufacturer's specified flow rate or best engineering practice.
 4. Dry N_2 or zero air should be flowing through the optical purge compartments or a vacuum must be pulled on these components to remove the ambient air interferents.
- b. The full sampling system should be checked for cold spots. Use good engineering practices to determine if cold spots exist.
 1. For example: A known amount of NH_3 blended with ambient air can be pulled through the entire system. A cold spot exists if the known amount of NH_3 does not reach the FTIR.
- c. Use the established T50 timing for the sampling system with respect to NH_3 as the test parameter pass or fail. A reference tracer gas such as CO_2 may also be used with the NH_3 to ensure delivery of gas through the full sampling system.
- d. With the sample gas flowing, check that the sample cell pressure is within $\pm 10\%$ of the method pressure (see 5.5.4 for note on CO and CO_2).

5.2 Verify the System Hardware Parameters

- a. See 6.1 for relevant parameters.
- b. Verify FTIR and sampling system temperature settings.
- c. Verify FTIR and sampling system pressure settings.

5.3 Validate the System Signal

- a. Validate the system signal alignment is within the manufacturer's parameters.
- b. Validate the infrared signal parameters are within the manufacturer's parameters.

6. FTIR 1065 PERFORMANCE VALIDATION

This section assumes that the sampling frequency in this section is the same as that which will be used during testing. The FTIR must be running and have passed all of the manufacturer's prescribed system checks listed in Section 6. The gas cell temperature should be kept at $191\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ or within $\pm 5\text{ }^{\circ}\text{C}$ of the calibration temperature.

6.1 The gases used are defined as:

- a. Zero air (from Table 1 of 1065.750 of 40 CFR 1065); see Table 1.
- b. The zero air must also have a moisture dewpoint of at least $-60\text{ }^{\circ}\text{C}$ or lower ($\sim 11\text{ ppm H}_2\text{O}$)



Table 1 - Zero air purity

Constituent	Purified Synthetic Air ¹	Purified N ₂ ¹
THC (C ₁ equivalent)	≤0.05 μmol/mol (ppm)	≤0.05 μmol/mol (ppm)
CO	≤1 μmol/mol (ppm)	≤1 μmol/mol (ppm)
CO ₂	≤10 μmol/mol (ppm)	≤10 μmol/mol (ppm)
O ₂	0.205 to 0.215 mol/mol or 20.5 to 21.5%	≤2 μmol/mol (ppm)
NO _x	≤0.02 μmol/mol (ppm)	≤0.02 μmol/mol (ppm)
N ₂ O ²	≤0.02 μmol/mol (ppm)	≤0.02 μmol/mol (ppm)

¹ EPA does not require these levels of impurities to be NIST-traceable.

² The N₂O limit applies only if the standard-setting part requires you to report N₂O. Value is under consideration to move from 50 ppb down to 20 ppb as of 10/3/2013.

6.2 Gas Standards (from Section 1065.750 of 40 CFR 1065)

6.2.1 Gases must be NIST traceable ±1% accuracy standards.

6.2.2 Accepted Single Gas Components (from Section 1065.750 of 40 CFR 1065)

- a. CH₄, C₂H₆, C₃H₈, CO, CO₂, NO, N₂O can be single components in balance of N₂.
- b. NO₂ must be in balance of air.

6.2.3 Accepted Gas Mixtures in N₂ Balance (from Section 1065.750 of 40 CFR 1065)

- a. C₃H₈, CO, CO₂, and NO.
- b. CH₄, C₃H₈, CO, CO₂, and NO.

6.2.4 For creating calibration reference and SPAN gases, you can use:

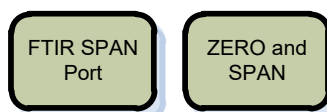
- a. Series of gas cylinders of known gas concentration still valid within the manufacturer's certification expiration date.
- b. Gas-division system that is annually calibrated to be linear with the span gas.

Note that all test gases (zero and calibration) must be at 191 °C ± 5 °C or within ±5 °C of the calibration temperature prior to entering the FTIR sample gas cell, as measured by the instrument manufacturers process.

7. CFR 1065 TESTING OVERVIEW USING FTIR

7.1 Tests Performed on the FTIR Span Port or Zero and Span

References in this document to tests performed on the FTIR span port or zero and span means the gas is not required to pass through the full sample train from the probe to the FTIR. These tests are performed by supplying the gas directly to the zero or span port at the inlet to the FTIR gas cell provided they are at the required temperature when they reach the gas cell. This reference is designated throughout the text as color coded in gray by the following two conventions:



In all cases, the span value referenced below refers to the maximum range value that is specified by the FTIR instrument manufacturer. If two or more ranges are specified for the same analyte, then there will be two or more span values corresponding to the specified ranges that must be validated.