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 wide-spread 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 (40CFR1U.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 (CO_2), oxides of nitrogen (NO, NO_2 and N_2O), ammonia (NH_3), methane (CH_4), and formaldehyde (H_2CO). Others 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

e-CFR 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 PPM or % 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 CO_2.

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 see 40CFR 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

LPM = Liters 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 1Hz 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

a. 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).

b. 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.

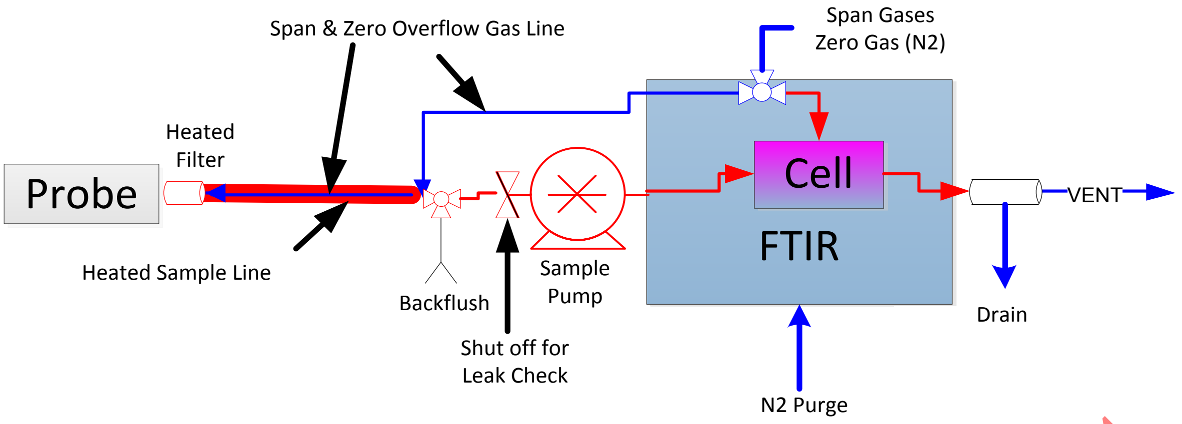


Figure 1 - Sample extraction via pushing of the gas

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 °C ± 5 °C. See Figure 2.

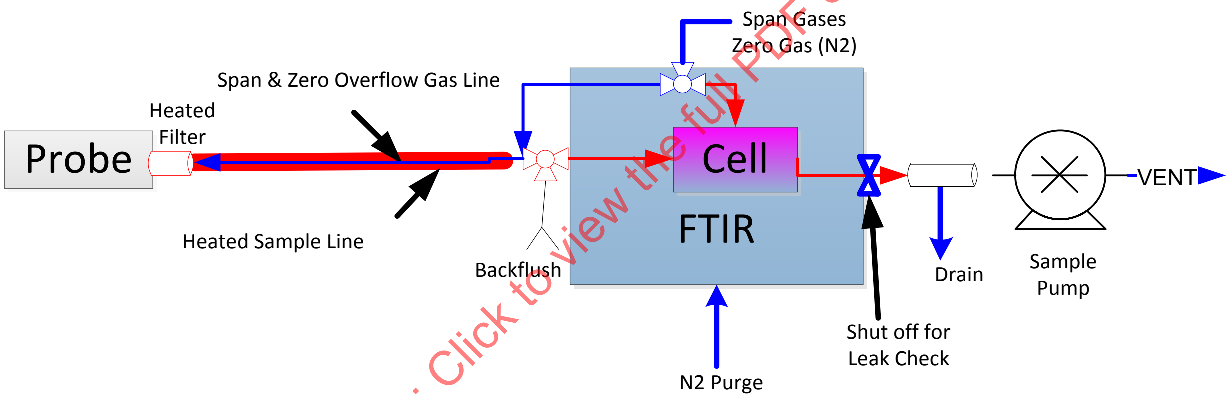


Figure 2 - Sample extraction via pulling of the gas

4.5 Experimental Considerations

4.5.1 FTIR Calibrations are pressure sensitive

a. Pushing a sample through the FTIR allows for the internal pressure to be at or near ambient pressure.

b. Pulling a sample through the FTIR lowers the overall internal pressure.

c. In order to reduce calibration errors due to pressure, the gas reaching the FTIR gas sampling cell should be within ±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°C ± 5 °C or within ± 5 °C of temperature at which the calibration was created.

4.5.3 Span Gas Line

The Span & 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 lpm while high flow systems reference flow rates > 30 lpm.

b. Avoid all cold spots prior to the gas sample entering the FTIR and maintain the gas temperature within 191 °C ± 5 °C.

c. In order to reduce analysis errors it is suggested that the gas cell temperature should be kept at 191 ± 5 °C or within ± 5 °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 ± 10% of the calibration method pressure.

Note that for gas components with very narrow lines such as CO and CO_2 the gas cell pressure might need to be held to within ± 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 less 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 1μ 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 0.1μ 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 °C ± 5 °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 °C ± 5 °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 Manufacturers 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 °C ± 5 °C.
  2. Dry N2 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 N2 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.

  - For example – a known amount of NH3 blended with ambient air can be pulled through the entire system. A cold spot exists if the known amount of NH3 does not reach the FTIR

c. Use the established T50 timing for the sampling system with respect to NH3 as the test parameter pass or fail. A reference tracer gas such as CO_2 may also be used with the NH3 to ensure delivery of gas through the full sampling system.

d. With the sample gas flowing check that the sample cell pressure is within ±10% of the method pressure (see Section 5.5.4 for Note on CO and CO_2).

5.2 Verify the System Hardware Parameters

a. See Section 6.1 above 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 ± 5 °C or within ± 5 °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 °C or lower (~11 ppm H_2O)

Table 1 – Zero air purity

Constituent	Purified synthetic air[1]	Purified N_2[1]
THC (C_1equivalent)	≤ 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_2O[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 N_2O limit applies only if the standard-setting part requires you to report N_2O. 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, CO_2, 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, CO_2, and NO

b. CH4, C3H8, CO, CO_2, 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.

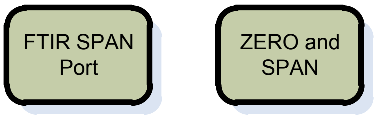
b. Gas-division system that is annually calibrated to be linear with the span gas.

Note that all test gases (Zero and Calibration) must be at 191 ± 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.

7.2 Test Performed on the FTIR + Sample Train

References in this document to tests performed on the FTIR + Sample Train means that the sample must traverse the full sample train from the probe end all the way through the FTIR gas sample cell and are designated throughout the text as color coded in purple by the following convention:

7.3 Laboratory Test Requirements

Tests that are done in the Laboratory or at the Instrument Manufacturer's site are as follows:

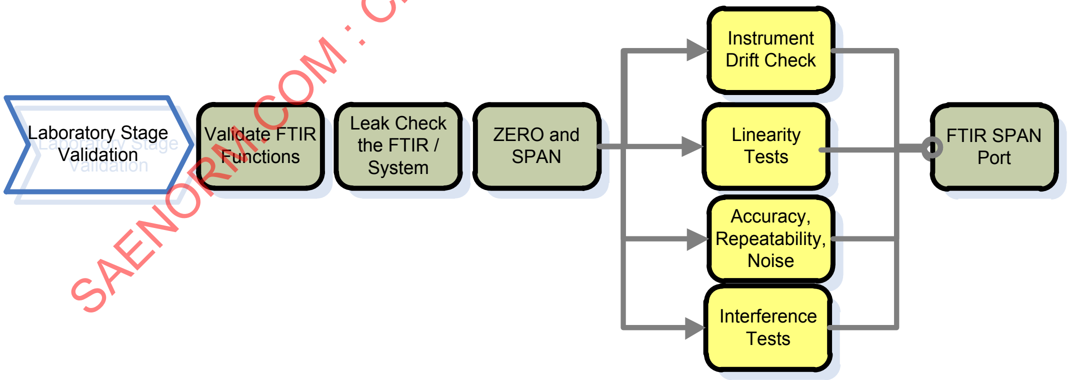


Figure 3 - Test requirements

a. Validate the FTIR Functions based upon Manufacturer's requirements (Section 6)

b. Perform a Leak Check (Part 1065.345, Section 10)

c. Zero the FTIR (Section 9.2)

d. Span the FTIR (Section 9.3)

e. Run the Instrument Drift Check (Section 14.3)

f. Perform the Linearity Verification Test (Part 1065.307, Section 9.6)

g. Accuracy, Repeatability, Noise Tests (Part 1065.305, Section 11)

h. Interference Tests (Part 1065.350, 1065.355, 1065.700, 1065.750, Section 12)

7.4 System Deployment Tests

Test Requirements for Initial System Deployment at the Certification Bench Site are as follows (See Figure 4):

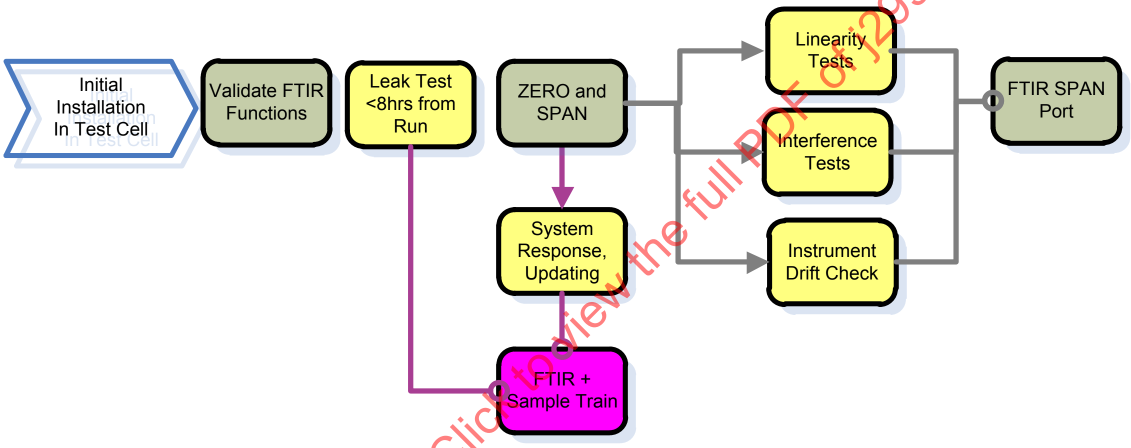


Figure 4 - Test requirements for initial deployment

a. Validate the FTIR Functions based upon Manufacturer's requirements (Section 6)

b. Some tests are performed flowing gas through the FTIR Span Port (Gray squares)

c. Linearity Verification Test (Part 1065.307, Section 9.6)

d. Interference Tests (Part 1065.350, 1065.355, 1065.700, 1065.750, Section 10)

e. Instrument Drift Check (Section 14.3)

f. Test performed flowing gas through the Full System (FTIR + Sample Train) (Purple squares)

g. Leak Test (Part 1065.345, Section 10)

h. System Response, Updating Tests (Part 1065.308, Section 13)

7.5 System Testing after Major Maintenance, System Modification or System Disruption

Tests followed as shown in Figure 5.

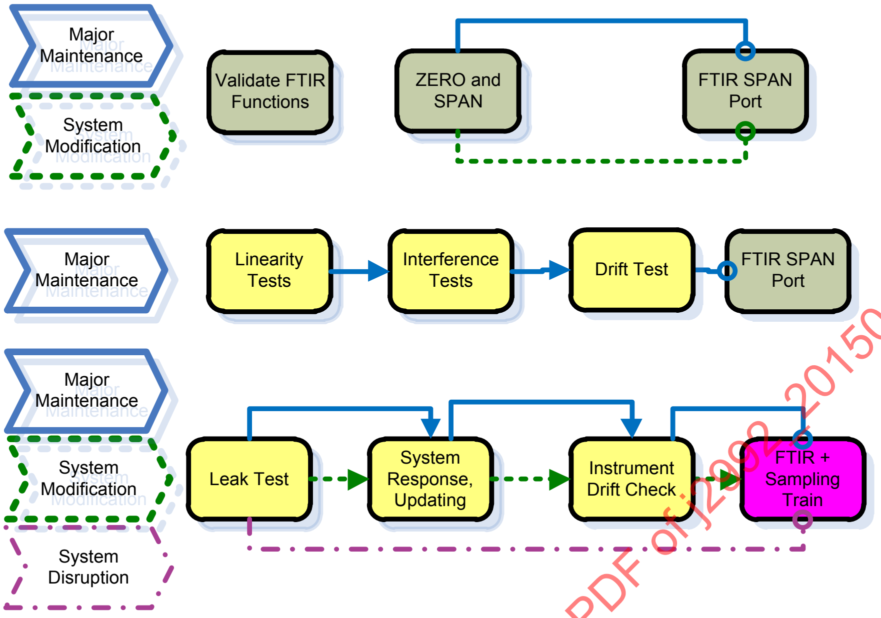


Figure 5 - Test requirements after major maintenance, system modification or system disruption

7.6 Tests performed after System Modification

a. Test on the FTIR or Sampling system (Green Lines):

b. Validate the FTIR Functions based upon Manufacturer's requirements (Section 6)

c. Zero the FTIR (Section 9.2)

d. Span the FTIR (Section 9.3)

e. Leak Test (Part 1065.345, Section 10)

f. System Response, Updating Tests (Part 1065.308, Section 13)

g. Run the Instrument Drift Check (Section 14.3)

7.7 Tests performed in order after Major Maintenance

a. Performed on the FTIR or Sampling System (Blue Lines)

b. Validate the FTIR Functions based upon Manufacturer’s requirements (Section 6)

c. Zero the FTIR (Section 9.2)

d. Span the FTIR (Section 9.3)

e. Linearity Verification Test (Part 1065.307, Section 9.6)

f. Interference Tests (Part 1065.350, 1065.355, 1065.700, 1065.750, Section 12)

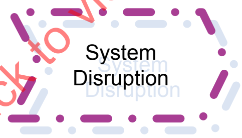
g. Leak Test (Part 1065.345, Section 10)

h. System Response, Updating Tests (Part 1065.308, Section 13)

i. Run the Instrument Drift Check (Section 14.3)

7.8 Test performed after a System Disruption

a. Test performed on the sampling system and FTIR after a System Disruption (Purple Lines)



b. Leak Test (Part 1065.345, Section 8)

7.9 Test performed prior to and after Emissions Cycle Testing

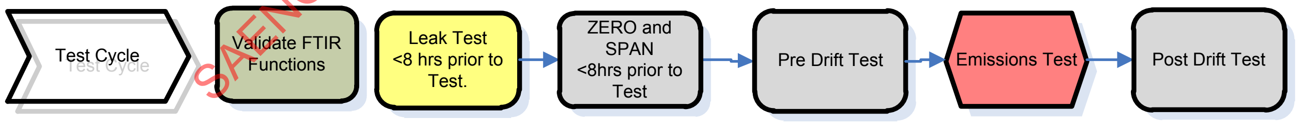


Figure 6 - Tests before and after emissions cycle testing

a. Validate the FTIR Functions based upon Manufacturer's requirements (Section 6)

b. Leak Test (Part 1065.345, Section 10)

c. Zero the FTIR (Section 9.2)

d. Span the FTIR (Section 9.3)

e. Pre Drift Test (Part 1065.550, Section 14.3)

f. Post Drift Test (Part 1065.550, Section 14.3)

g. The results from the Emission Test need to be corrected following 1065.672 of 40 CFR 1065 and are not discussed in this document.

8. LINEARITY VERIFICATION TEST

This section is to verify that the FTIR response is proportional over the full range in which the instrument is intended to be used. The linearity performance criterion values are from ***Table 1 Section 1065.307*** and the required calibration and verification rates are from ***Table 1 Section 1065.303 of 40 CFR 1065.*** The general overview of this Section Tests is:

a. Ensure that the all gaseous samples are at 191 ± 5 °C or that the gas sample is at ± 5 °C of the calibration temperature prior to entering the FTIR gas cell.

b. Measure at least 10 separate test points (approximately evenly spaced from the maximum range value down to zero) on the FTIR then compare them to the nominal reference value as reported by the gas blending system (gas divider or MFC based).

c. Perform a least squares fit of the data and use the resulting values and fit parameters for compliance evaluation.

Note that the system pressure and temperature must be the same as what is to be used during the cycle tests.

8.1 Frequency of Test

a. Upon initial installation

b. Within 370 days before testing

c. After major maintenance

8.2 Zero the FTIR

Send the Zero Gas (in this case it must be dry N2) to the FTIR through the sample gas cell. Run the Zero Gas until the value for each component to be certified comes to steady-state, then ZERO the instrument.

8.3 Span the FTIR

Send the Span gas to the FTIR through the sample gas cell. Run the Span Gas until the value comes to steady-state, then Span the instrument using a certified gas using the nominal value. This concentration value should be up to 5% below the maximum range value for the FTIR gas component as specified by the instrument manufacturer or 5% above the maximum concentration expected during testing provided this value is not about the FTIR range.

Flow the Span gas until the value of the component to be certified comes to steady-state, then SPAN the instrument for the analyte. If using a Gas Mixture then flow the Span gas mixture until the value for each component to be certified comes to steady-state, then SPAN the instrument response for each of the analytes.

Span each analyte and range using an EPA approved gas mixture and / or separate Span gas cylinders. If more than one cylinder is used repeat this step and make sure that all gaseous samples are at 191 ± 5 °C or that the gas sample is at ± 5 °C of the calibration temperature prior to entering the FTIR gas cell before spanning.

8.4 Recheck the Zero

Flow Zero Gas through the gas cell as described above in Section 8.2 then recheck the Zero values. The System passes the Zero check if the absolute error of the Zero FTIR value for each analyte of interest reads ≤1% of the analyte Span gas concentration. If this criterion is not met then rezero and respan the FTIR.

If more than one rezero is required to be compliant then go back and check all of the FTIR parameters to make sure they are still within the manufacture's parameter set. If the parameters are not within the manufactures specifications then repair and retune the FTIR and start the test again.

8.5 Create the Calibrated Test Data Points

Set the Data Acquisition to the scan rate to be used during the certification testing. Use Gas Divider or Gas Blending system to create a total of 11 calibrated data points as follows:

a. The certified concentration value of the gas cylinder (no dilution of the cylinder gas) must be at the specified FTIR range maximum.

b. Create a "zero" calibration point using dry N2 gas or zero air, based upon the balance of the span gas being used.

c. Create 9 other calibration points in between and preferably evenly spaced between the maximum FTIR range value and the zero value.

Collect data on the FTIR and save at least 2- 3 min for each calibrated data point / cut point once the value has reached steady-state (or use good engineering judgment to determine integration time). Switch the Gas Blending system to collect the next calibrated data point.

a. Repeat this step for each Analyte as well as Range that is to be validated.

b. Zero and Span the analyte first before repeating this step using the maximum value of the FTIR Range as the Span value.

8.6 Determine the Linearity for Each Analyte and Range

Calculate the mean for of each of the Analyte calibrated concentration reference values created above. Record the average value for each calibrated reference value point once it reaches steady-state using a 30 s average. Use all of the 11 calibrated reference values to calculate the regression line and regression parameters which are used in the Linearity Check below. Check the results against the specifications from Table 1 of section 1065.307 (shown in the Table 2 below. Repeat this step for each Analyte and FTIR Range that is to be validated.

Table 2 - Linearity requirements

Measurement system	Quantity	Minimum verification frequency	Linearity criteria			
			$\lvert x_{min}(a_1-1)+a_0 \rvert$	a_1	*SEE*	r^2
FTIR for Laboratory Testing	x	Within 370 days before testing	$\leq 0.5\% \cdot x_{max}$	0.99–1.01	$\leq 1\% \cdot x_{max}$	≥ 0.998
FTIR for field testing	x	Within 370 days before testing	$\leq 1\% \cdot x_{max}$	0.99–1.01	$\leq 1\% \cdot x_{max}$	≥ 0.998

a. **Xmax** is the maximum calibrated reference value used during the linearity test.

   1. This value is the FTIR Range Maximum that is being tested.
   2. If the FTIR manufacturer specifies that the analyte within the method is valid across multiple ranges then there will be a different Xmax value for each of the FTIR Ranges that are to be verified.
   3. The selected value should be at or near the full scale value of the FTIR Range being tested and this value must be above the maximum value that would be seen during the Test Cycle.
   4. Use good engineering judgment to determine the maximum value expected for the particular engine test.

b. **Xmin** refers to the minimum calibrated reference value used during the linearity test that is closest to zero (0) but not zero.

   1. If the FTIR manufacturer specifies that the analyte within the method is valid across multiple ranges then there will be a different Xmin value for each of the FTIR Ranges that are to be verified.
   2. The selected value should be the point nearest zero (0).
   3. Use good engineering judgment to determine the lowest value closest to zero (0).

c. **SEE** refers to the ***Standard Error of Estimate*** and is calculated in Equation 1 (see **40CFR Part 1065.602** for definitions and examples):

$$SEE_y = \sqrt{\frac{\sum_{i-1}^{N}[y_i - a_0 - (a_1 * y_{ref\,i})]^2}{N-2}} \qquad \text{(Eq. 1)}$$

N	Total number of measurements made (minimum of 11 including zero)
y_i	FTIR Measurement Value at point (i)
$\bar{y}_i$	Average FTIR Measurement Value at point (i)
y_{refi}	Reference Concentration Value (calculated from the gas dilution system) at point (i)
$\bar{y}_{ref}$	Average Reference Value which is the average of all the Cutpoints generated during the validation test including zero (0).
a$_0$	Intercept of linear regression curve using the calibrated test points
a$_1$	Slope of linear regression curve using the calibrated test points

d. **a**$_0$ refers to the intercept calculated by:

$$a_0 = \bar{y} - (a1 * \bar{y}_{ref}) \qquad \text{(Eq. 2)}$$

e. **a**$_1$ refers to the slope calculated by:

(Eq. 3)

9. SYSTEM LEAK TESTS

9.1 General Overview

This section follows section 1065.345 of 40 CFR 1065 to verify that the vacuum side of the sampling system (shown in GREEN in the figures below) does not have any significant leaks. During the Leak Check the sample inlet located at or near the probe must be blocked off.

For those systems with a pump on the upstream side of the analyzer (see Figure 7), it may be prudent to check the full sampling system for leaks, even though the System Leak check only requires testing for vacuum side leaks, as this type of leak will affect the amount of analyte being seen at the analyzer.

Verification of the presence of a leak is demonstrated by one of the following:

a. Measuring small amounts of flow when there should be none.

b. Measuring the pressure rise within an evacuated sampling system which is above the acceptable limit.

  - A pressure readout gauge or monitor must be located within the evacuated section

c. Measuring excessive dilution of a known tracer gas flowing through the vacuum side of a sampling system.

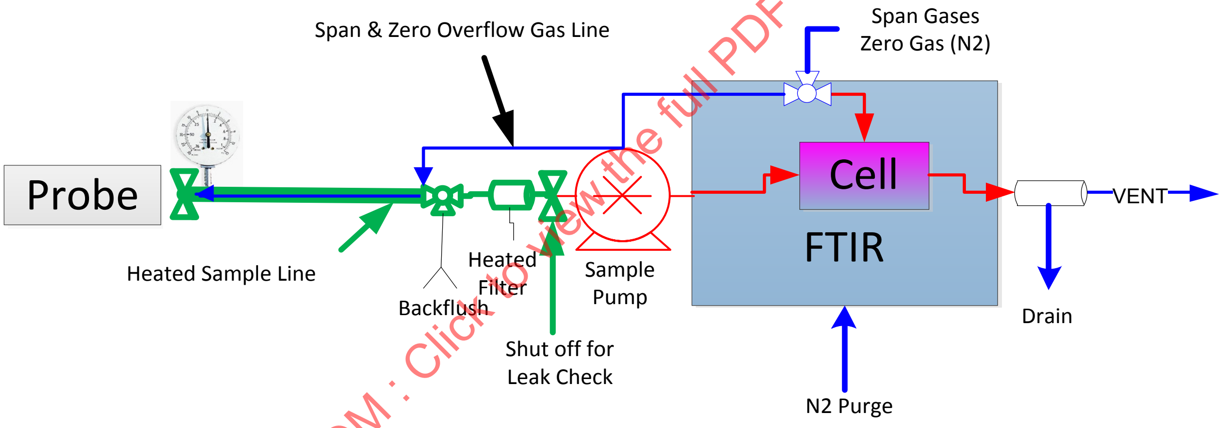


Figure 7 - Leak test with pump upstream of the analyzer

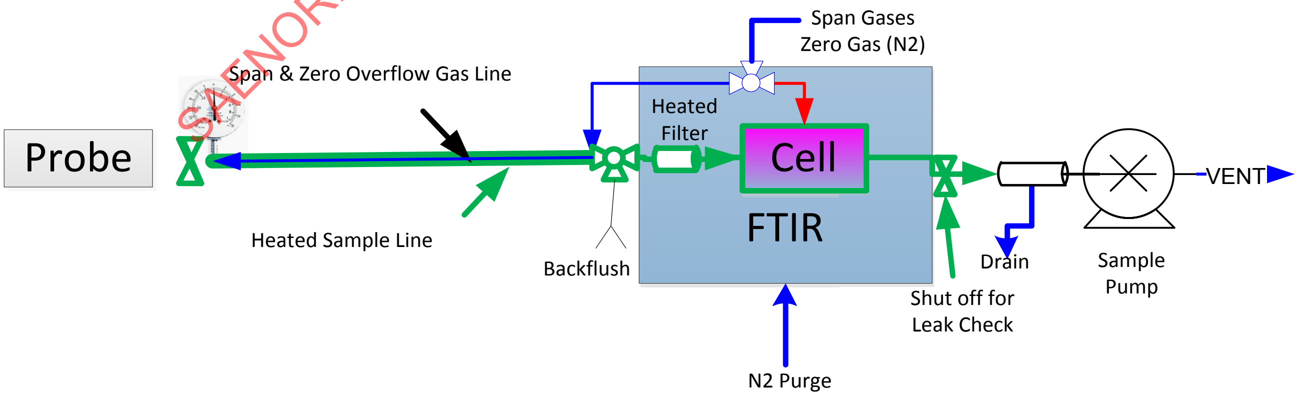


Figure 8 - Leak test with pump downstream of the analyzer

9.2 Frequency of Test

a. Laboratory Testing / Sampling System Manufacturing Stage

- Upon initial sampling system installation

b. Field Testing Stage

1. After each installation of the sampling system into the engine test site prior to the start of the field test.
2. 8 hours prior to the first test interval of a duty cycle test

c. After any system modification that would affect response such as:

1. Changing the sample transfer line, materials or fittings on the sample train
2. Replacing, adding or removing a filter

9.3 System Leak Test #1: Low-Flow Leak Test

Note that for this Leak Check procedure a flow measuring device must be installed on the vacuum side of the sampling system.

a. Seal the inlet side near the probe end of the sampling system within 92cm from the probe:

- Cap or plug the end of the sample probe or close a valve located in the sample line and after the probe but within 92cm of the probe.

b. Pull a vacuum across the vacuum side of the sampling system.

1. Turn on all sampling pumps within the system
2. Let flow reading come to a stable value

c. System passes Low-Flow leak test if:

1. Flow rate measured is ≤ 0.5% of the Standard Usage Flow Rate
2. If a bypass system, where the sample flow rate through the vacuum side of the sample system is not the same as the FTIR cell flow rate, is used, then it is the higher flow rate (bypass plus analyzer/FTIR flow(s)) that is used as the Standard Usage Flow Rate.
    - For example, if the total standard usage flow rate used during the Cycle test is 2 lpm then Leak Test #1 passes if the flow measuring device reads a value of 0.01 lpm or less.

9.4 System Leak Test #2: Vacuum-Decay Leak Test

a. Requires the following:

1. Vacuum decay leak rate is based on the equations from Section 1065.644.

2. Must use accurate measurement equipment as defined in Subpart C and Subpart D of Part 1065

3. Vacuum side volume must be known to within ± 10% of its true volume

b. Seal the probe end of the sampling system

- Cap or plug the end of the sample probe or close a valve located in the sample line and after the probe but within 92 cm of the probe.

c. Pull a vacuum across the vacuum side of the sampling system

   1. Turn on all sampling pumps within the system – use good engineering practice to make sure that any pressure spike will not damage the FTIR components.
   2. Let the pressure come to a stable value
   3. Close off the sampling pump using a shut off value on the vacuum side.
   4. **Turn off the pump** as quickly as possible once the shut off valve is closed

d. Monitor the vacuum / pressure and temperature of the system

   1. Wait long enough for any transient pressure spike to settle.
   2. Once the pressure has reached at least 10x the resolution of the Pressure Transducer record the pressure ~ 2 – 5 min

Note that any sample and / or vacuum pumps used to create the vacuum must all be turned off at this point during the leak test.

e. Calculate the Vacuum Decay Rate (moles/s)

$$DecayRate = \frac{V_{vac}\left(\frac{P_2}{T_2} - \frac{P_1}{T_1}\right)}{R(t_2 - t_1)} \quad \text{(Eq. 4)}$$

where:

V_{vac}= geometric volume of the vacuum side of the sampling system (m^3)

R = molar gas constant = 8.314472 J/(mol·K)

P_1 = vacuum side absolute pressure (Pa) at time t_1

P_2 = vacuum side absolute pressure (Pa) at time t_2

T_1 = vacuum side absolute temperature (K) at time t_1

T_2 = vacuum side absolute temperature (K) at time t_2

t_1 = time (s) at start of the vacuum-decay leak verification test

t_2 = time (s) at end of the vacuum-decay leak verification test

f. The sample system passes the leak test if the vacuum decay leak flow rate is ≤ 0.5% Standard Usage Flow Rate.

   1. If a bypass system, where the sample flow rate through the vacuum side of the sample system is not the same as the FTIR cell flow rate, is used, then it is the higher flow rate (bypass plus analyzer/FTIR flow(s)) that is used as the Standard Usage Flow Rate.
   2. For example, if the total standard usage flow rate used during the Cycle test is 2 lpm then Leak Test #1 passes if the flow measuring device reads a value of 0.01 lpm or less.

9.5 System Leak Tests #3: Dilution of Span Gas Test

In this section you can either use a Span Gas or N_2 as the overflow of gas at the probe. If using N_2 gas for leak verification the Sampling System must have an O_2 sensor in the sample train within the leak check region. The test requires that the O_2 sensor must be able to discern a change of <0.5% in the O_2 concentration or if using a certified span gas then that span value concentration must be within 0.5% of the certified cylinder value.

9.5.1 Overflow the Span (or N_2) Gas at the Probe

a. Operate the analyzer and sample train as you would for emissions testing.

b. Allow the system's flows and temperatures to stabilize.

c. Zero the instrument according to the practice described earlier.

d. If using a Span Gas for this test then Span the instrument according to the practice described earlier.

- Select a Span Gas that does not stick to the sample line material such as CO, CO_2, SF_6, CH_4 or NO, but do not use a reactive gas like NH_3.

e. Flow the test gas through the Span Gas Overflow port (or Tee) located at or near the Sample Probe

1. The Span Gas Overflow port must be within 92cm of the sample probe.
2. The test gas flow rate should be a minimum of 10% above the sample flow rate and the Span Gas concentration at that level should be easily detectable by the FTIR.

f. Collect the Data

1. Allow the Span Gas reading (or the O2 reading if using N2 for your leak test) to reach a steady-state value - use good engineering practice to determine what that value is.
2. Collect at least 30 s of data at steady-state.

9.5.2 Quantify the Span Gas Data at the Probe

a. **[y_{span}]** Certified Span Gas or O2 Calibration Value

b. **[y_{ave}]** The average 30 s of data at steady-state.

- **[y_{ave}]** for a 1Hz acquisition rate contains 30 data points while for 5Hz it will contain 150 data points

c. Pass / Fail Criterion

1. If you are using a Span Gas, the average of the 30 s FTIR reported values [yave] must read within 0.5% of the certified Span Gas concentration:

$$[y_{ave}] \leq [y_{span}] \pm 0.5\%$$

2. If you are using N2 as the overflow gas then the average of the 30 s O2 sensor reported reading values [yave] must read within 0.5% of the certified O2 content in the N2 gas stream:

$$[y_{ave}] \leq [y_{span}] \pm 0.5\%$$

10. DETERMINE THE ACCURACY, REPEATABILITY AND NOISE OF THE FTIR

This section is used to insure that the FTIR (not the system which is described in a later section) meets the performance specifications as defined in ***Table 1 Section 1065.205.*** This section follows the guidelines as set out by ***Section 1065.305 of 40 CFR 1065*** and are not required by by EPA but they are strongly recommended after the initial installation.

10.1 Frequency of Test

Upon initial installation of the full system in the field.

10.2 Collect Data for Accuracy, Repeatability and Noise Tests

a. Zero the instrument

b. Span the instrument using a gas cylinder value at or up to 5% below the maximum range value of the FTIR for each Analyte

c. Use the same data collection rate that will be used during the emissions test.

d. Run a gas sample of the Analyte Gas that has a concentration value based upon the greater of the two:

   1. Half of the maximum range value of the FTIR which was verified during the linearity check

   2. Half the expected value in the emissions to be tested

e. Run the gas for 2-3 min to reach measurement steady-state or use good engineering practice to determine when the system is at steady-state.

10.3 Repeat the Data Collection Process

a. Repeat the Zero, Span, Collect Data section 10 times for the current analyte and FTIR range (for those analyzers that use multiple ranges) being tested.

   1. Use the same concentration value each time.

   2. If a blend gas is used then more than one analyte can be done at this time otherwise use single cylinders and test each one separately.

b. Repeat this test for each analyte and FTIR range that is to be validated on this system.

10.4 Record the following Data on Each of the 10 Runs

If you are running the analyzer using a 1Hz acquisition rate then you will have 30 data points in the calculations below while for a 5Hz acquisition rate it will contain 150 data points. This section below uses the following nomenclature:

where

$[y_{ref}]$	Reference Gas Certified Calibration value
[yi]	Calculate the average of each 30 s data acquisition period for each of the 10 repeats.
$[y_{ave}]$	Calculate the average of the 10 average **$[y_i]$** values.
$[\sigma_i]$	Calculate the Standard Deviation of the **$[y_i]$** values for each of the 10 repeat values at each reference point.
$[\varepsilon_i]$	Calculate the error **$[y_{ref}]$ - $[y_i]$** for each of the 10 repeats.
$[\sigma\varepsilon]$	Calculate the Standard Deviation of the 10 error data values $[\varepsilon i]$ for each of the 10 repeats.

10.5 Quantify the Values

While regulatory bodies may not require the analyzer to pass Accuracy, Repeatability and Noise tests, it is strongly recommended that these tests be completed and the analyzer pass all of them in order to ensure the instrument is operating properly.

This section assumes that you have spanned the FTIR to the reference gas for each analyte that is to be validated.

10.5.1 Rise and Fall Times

The rise and fall times of the values being tested on the “FTIR only” and not the full system must be ≤5 s:

$$|t_{90} - t_{10}| \leq 5s$$

$$|t_{10} - t_{90}| \leq 5s$$

10.5.2 Accuracy

Calculate the accuracy using the absolute value of the reference value **[y_{ref}]** minus the average of the 10 FTIR reported average values **[y_{ave}]**. The analyzer passes the Accuracy Test if the result is ≤ 2% of the mean value of the expected reference point:

$$| [y_{ref}] - [y_{ave}] | \leq 0.02^* [y_{ref}]$$

10.5.3 Repeatability

Calculate the repeatability as 2 times the standard deviation of the 10 error values **[σ_ε]**. The analyzer passes the Repeatability Test if the result is ≤ 1% of the mean value of the expected reference point:

$$[2 * \sigma_\varepsilon] \leq 0.01^*[y_{ref}]$$

10.5.4 Noise

Calculate the noise as 2 times the root mean squared error of the 10 standard deviations of each 30 s measurements **[σ_i]**. The analyzer passes the Noise Test if the result is ≤ 1% of the peak value expected based upon the gas (or gas blender / divider) certification value that is being tested:

$$2^*\text{rms}\sigma = 2^*\sqrt{\frac{1}{N}\sum_{i=1}^{N}\sigma_i^2} \leq 0.01^*[\text{yref}] \qquad \text{(Eq. 5)}$$

11. INTERFERENCE TESTS

This section is to ensure that the analysis method used to determine the concentrations of the various components are not significantly affected by interferents. The following sections from ***EPA 40 CFR 1065*** were used to develop the interference testing and were applied to the FTIR:

a. For CO_2 - Section 1065.350 of 40 CFR 1065.

b. For CO - Section 1065.355 of 40 CFR 1065.

c. For NOx - Section 1065.370 of 40 CFR 1065.

d. For N2O - Section 1065.375 of 40 CFR 1065.

All other analytes that are not currently regulated are listed in the Table below with EPA comments for what might be recommended - however they are not currently mandated nor are the values agreed upon.

11.1 Choice of Interferences

Use good engineering practice when determining the list of potential interferences for each analyte. This should be based upon the estimated concentrations of the interferences that would be present as well as the fuel source used. The FTIR MFG can also provide feedback on other potential interferents that are not specifically listed here but might be interferents due the analysis regions or peaks that were chosen. The current list of suggested interferents and concentration levels for each analyte to be tested are presented in Appendix B as a function of fuel source.

11.2 Interferent Verification

The interference tests are performed by running a gas containing the interferent concentration at the highest value that could be seen during testing. If unknown then use the values referenced in ***Appendix B*** for the specific fuel that will be used during testing as a starting point.

- It is strongly suggested that each interferent should be run by itself so that any method issues that might arise can be easily isolated and fixed.

11.2.1 Interference Acceptance Criterion

While the actual EPA Part 1065 document calls for the verification to be based upon a percentage of the **Flow-weighted mean concentration (FWMC)** for the emission standard being tested, the instrumentation Manufactures feel the value should be based upon the FTIR Range in order to make sure that the analyzer would be appropriate for each test that is to be performed.

a. The interference acceptance value is based upon one of the two:
   1. A fixed value as stipulated by EPA in the regulation.
   2. Or a percentage of the maximum range as stipulated by the FTIR manufacturer and verified for each Analyte (as well as multiple FTIR Ranges if applicable).

b. Refer to Appendix B for specific interferents that must be tested for the listed fuel sources but in the case where there is not a fixed value use 1% of the FTIR Range of the analyte.

11.2.2 Frequency of Test

a. After the initial analyzer installation

b. After major analyzer maintenance

c. This test can be performed at the FTIR (avoid going through the full sampling system) provided the SPAN port lines are heated and are able to keep the H_2O or other components from condensing.

11.3 Set up the FTIR to Collect Interference Data

a. Zero the instrument

b. Span the instrument using a gas cylinder value up to 95% of the maximum range of the FTIR for each Analyte.

c. Set the Data Acquisition to the Sample Rate to be used in the Emission Tests.

d. Negative values cannot be set to zero for this test, therefore both negative and positive values must be reported.

11.4 Collect the Interferent Gas Responses

a. Run each interferent gas separately using the maximum expected concentration of the interferent gas in a dry N_2 gas stream (or for NO_2 use dry air).

   1. You can combine the interferents for this test but best practice is to run them separately so that if there is a failure on this test you can determine where the major interferences are occurring.
   2. Pass dry N_2 through a heated bubbler of distilled H_2O or other such apparatus to provide the maximum H_2O level that will be seen.
   3. The concentration value of the interferent gas does not need to be at steady state but must be on average within 10% of the required maximum concentration listed in the tables in ***Appendix B***.

b. Send the Interferent test gas stream directly to the FTIR or through the full sampling system.

   - If the FTIR system uses a dryer (or other gas conditioning system) then you must send the gas stream through the dryer first before the gas enters the FTIR.

c. Make sure the gas stream is at the required temperature of 191 ± 5 °C.

d. Make sure the gas stream pressure is within ± 10% of the pressure that will be seen during the Test Cycle runs.

11.5 Calculate the Amount of Interference on the Analyte

a. Once the analyzer response has come to steady-state collect data for 30 s.

b. Calculate the 30 s Average for each of the Analyte responses that are to be used for certification testing.

c. If running the interferent gases separately, repeat the test with the next interferent.

   - Interferents change depending upon Fuel used therefore some suggested components and their associated concentration values are listed in the tables in Appendix B.

11.6 Calculate the Maximum Error / Bias for each Analyte

a. If the FTIR method uses separate ranges for reporting High and Low concentrations (or switches across multiple peaks or ranges reporting across a broad concentration range) for the analyte of interest, then the biases must be validated for each analyte range, using the maximum interferent concentration that would be seen during the test.

b. Calculate the error from the FTIR response for each analyte of interest when only the interferents are present using the method below.

   - For this section negative values cannot be set to zero, therefore both negative and positive values must be reported.

c. If running separate or multiple interferents, add the positive and negative values together in one equation.

$$Total_{BIAS}(j) = \sum_{i=1}^{N} FTIR(j)_{Interferent(i)} \qquad \text{(Eq. 6)}$$

Where

(j) is the index for the analyte being validated (eg. N_2O, CO, NH_3, etc.)

(i) is the index for the interferent being tested (eg. CO_2, H_2O, CH_4 etc.)

FTIR(j)

d. Example

1. ***j*** is set to be N2O which is the analyte being validated
2. Test the potential biases on the FTIR response of N2O when only CO_2 and H_2O are present:
3. ***i*** (1) = 15% CO_2
4. ***i*** (2) = 15% H_2O

$$Total_{BIAS}(N_2O) = FTIR(N_2O)_{CO2} + FTIR(N_2O)_{H2O}$$

5. The Final Bias value ($Total_{BIAS}(N_2O)$) for the Analyte is then the sum of all of the positive and negative biases as reported by the FTIR for CO_2 and H_2O in this example.

e. Repeat for each analyte ***(j)*** that is to be validated.

11.7 Determine Pass or Fail for Each Analyte *(j)*

a. Use Table 3 (below) to determine each analyte ***(j)*** Pass / Fail Criterion

Table 3 - Pass / fail criteria

Component	**EPA Part 1065 Section**	**Criterion for 1065 ($Final_{BIAS}$(j)<Criterion)**
CO (Carbon Monoxide)	1065.355	≤1% of the FTIR Range value for CO
CO_2 (Carbon Dioxide)	1065.350	≤0.2 mmol/mol of CO_2
NOx (NO_2 + NO)	1065.370	≤1% of the FTIR Range value for NOx
N_2O (Nitrous Oxide)	1065.375	≤0.5 umol/mol of N_2O
CH_4 (Methane)	**Currently not regulated but may be required to be measured for informational purposes for GHG Reporting**	At a maximum it would need to meet ≤1% of the FTIR Range value for CH_4
NH_3 (Ammonia)	**Currently not required to be measured**	At a maximum it would need to meet ≤1% of the FTIR Range value for NH_3
HCHO (Formaldehyde)	**Currently not required to be measured**	At a maximum it would need to meet ≤1% of the FTIR Range value for HCHO

b. If the calculated error is too high:

1. The method for that particular analyte (j) needs to be modified in order to meet the specification
2. The Linearity verification for that method will need to be repeated at a minimum if changes are made to any analysis method
3. If modification is not possible then the FTIR System will not be qualified to be used for certification purposes for that particular analyte (j) that failed.

12. SYSTEM RESPONSE AND UPDATING-RECORDING VERIFICATION TEST

a. Follow Section 1065.308 of 40 CFR 1065

- FTIRs will follow Section 1065.308 and not that of Section 1065.309.

b. This section is for verification of the system response of the gas analyzer and the sampling system together.

c. Assures that the system will provide a response to a rapid concentration change without loss of information.

d. For those components that have longer retention times (such as NH3) use best engineering judgment when establishing stabilization at all flow rates used.

1. Running a Hot Wet analysis for NH3 is one way to get faster response
2. Using glass coated sample lines also reduces NH3 retention
3. Shorter sample lines greatly reduce component retention
4. Test all filter material to ensure that the analyte does not get entrained in the material prior to use in the sampling system

e. In the case where the sample flow rate is >30 SLPM ambient air can be used as a make-up gas provided the final analyte concentration that is tested is within 80 – 100% of maximum concentration for the FTIR Range that is being tested for that particular analyte (repeat if multiple ranges are used in the FTIR analysis Method).

12.1 Frequency of Test

a. System Response Test is performed upon the whole sample train including the FTIR analyzer and during the following:

1. Upon initial installation
2. After system modification that would affect the response such as:
    - Increasing the sample transfer line length
    - Adding, changing material, changing pore size of a filter
    - Reduction in the frequency of the updating or sampling rate

12.2 System Requirements

a. The mean Rise (Fall) time is ≤ 10 s

b. The system must also meet one of the two requirements listed below:

1. The product of the mean Rise time and the sample frequency must be 5 or greater and the product of the mean Fall time and the sample frequency must be 5 or greater.

   OR

2. The sample frequency is 5 Hz or faster

12.3 System Setup

a. Setup the acquisition parameters to the final configuration that will be used for data acquisition

b. It is best to test the system when it is not connected to the exhaust.

c. If the FTIR shares a sample probe which is drawn upon by other instruments:

  1. All of the instruments must be turned on and the sample flow rates adjusted to the final configuration used during testing.
  2. Multiple instruments can be tested at the same time provided the gas blends are acceptable.
  3. You can use gas blends as well as gas blending devices to create the Span Gas concentrations used for testing.

12.4 Sample Probe Overflow

a. Generally the system tests are done on Dry Gas except in the case where the flow rate is >30 LPM. In this case ambient air can be used as make up gas to test the response time of the target emission component provided the conditions listed below are met.

b. For lower overall sample flow rates (≤30 LPM):

  1. Overflow the calibration gas at the probe using a flow rate at least 10% more than the gas sample flow rate.
  2. Make sure there is no air leak contributing to the final value
  3. The final analyte concentration that is tested must be within 80 – 100% of maximum concentration for the FTIR range being tested.

c. For higher overall sample flow rates (>30 LPM), send a sample of gas that contains the analyte in N2 (or Air if testing NO2) to the probe using ambient air to make up the rest of the gas requirements.

  1. Use a mass flow controller that has at least 1% accuracy at 110% of the sample flow rate that will be used.
  2. Leave the probe end open to ambient air in order to balance out the total flow required by the system.
  3. Before adding the Analyte to the ambient air flow, collect data on the ambient air and determine the analyte concentration as a starting baseline.
  4. Set the flow rate for the analyte gas such that when blended with the make-up gas / ambient air the concentration of the analyte of interest will be at 80 -100% of the maximum SPAN value that will be used for this component on the FTIR.
  5. Ambient Air can only be used if the amount of the analyte in the air is less than 0.1% of the range at which you are validating.

d. Higher Flow Rate Calculation Example:

    1. Sample System total flow rate = 50 LPM
        - 10% of the sample system flow rate (50 LPM x 10%) = 5LPM
        - 5 LPM is the flow rate assigned to the MFC on the Nitric Oxide (NO) SPAN gas
    2. NO Cylinder Concentration needed:
        - Nitric Oxide (NO) concentration at the expected standard is 200 ppm
        - In order to reach 200 ppm when only using 10% of the flow with and overall flow rate of 50 LPM the required concentration of NO in N2 would need to be 2000 ppm = 200 ppm NO *50LPM/5LPM.
    3. If the analyte is present in ambient air (such as CO_2 or CO) then make sure the final value that is used to determine the response times takes those into account.

12.5 Running the Test

a. Flow Zero Gas until the system response is stabilized.

- For the High Flow system, pull in ambient air to the FTIR and wait until the response is stabilized.

b. Start collecting data at the same rate to be used in engine test cycles.

- Do not interpolate or filter the data.

c. Switch the flow to the SPAN gas sample.

1. For the High Flow system spike in the required flow rate of gas for the analyte of interest (example above used 10% of the total flow).
2. Collect enough data to adequately capture the rise times:
    - Run the SPAN gas long enough to reach steady-state.
    - Use good engineering judgment to determine this.

d. Switch the flow to the Zero gas sample.

1. For the High Flow system, stop the tracer gas flow pulling in ambient air only to the FTIR.
2. Collect enough data to adequately capture the fall times:
    - Run the Zero gas long enough to reach steady-state “0” value.
    - Use good engineering judgment to determine this.

e. Repeat these steps 7 more full cycles ending in the Zero Gas.

f. Stop Recording when done.

12.6 Analyzing the Data

a. Determine the Rise and Fall times of the FTIR

1. Rise Time = **=** $|t_{10} - t_{90}|$**,**
    - t_{10} is the time at which the sample concentration appears at the FTIR and is 10% of the Steady-state value on the up slope side.
    - t_{90} is the time at which the sample concentration appears at the FTIR and is 90% of the Steady-state value on the up slope side.
2. Fall Time = $|t_{90} - t_{10}|$**,**
    - t_{90} is the time at which the sample concentration appears at the FTIR and is 90% of the Steady-state value on the down slope side.
    - t_{10} is the time at which the sample concentration appears at the FTIR and is 10% of the Steady-state value on the down slope side.
3. Points can be extrapolated to determine t_{90} and t_{10}

b. Use the data from the 7 data runs to determine an average Rise and average Fall time for each analyte.

$$\text{Rise Time}_{AVE} = |t_{10} - t_{90}| \leq 10\text{s}$$

$$\text{Fall Time}_{AVE} = |t_{90} - t_{10}| \leq 10\text{s}$$

c. If the data acquisition rate was 5Hz or faster you are finished – skip to next section.