

FTIR: an Advanced FTIR for Toxic Gas Analysis

(ISO 19702; EN 45545-2; IMO)

firetesting
technology



Toxicity analysis of fire effluents is an important aspect for developing modern materials used in aircrafts, trains and buildings to ensure public safety. Analytical techniques and performance criteria have been specified in various fire safety standards and regulatory codes. FTIR is the technique that has been chosen by the ISO, EN and IMO standardisation committees as the most suitable method for measuring toxic fire effluents. The commonly targeted toxic species are CO, CO₂, HCN, SO₂, NO_x, HCl, HBr and HF.

Fourier Transform InfraRed (FTIR) Spectroscopy

FTIR spectroscopy is a full-spectrum analytical technique that allows all IR absorbing species to be detected and measured by a single instrument.

The infrared light from the source is modulated by the interferometer. This device allows for the light to be split into two different paths and recombined, producing an interference wave known as an interferogram. The light is split via

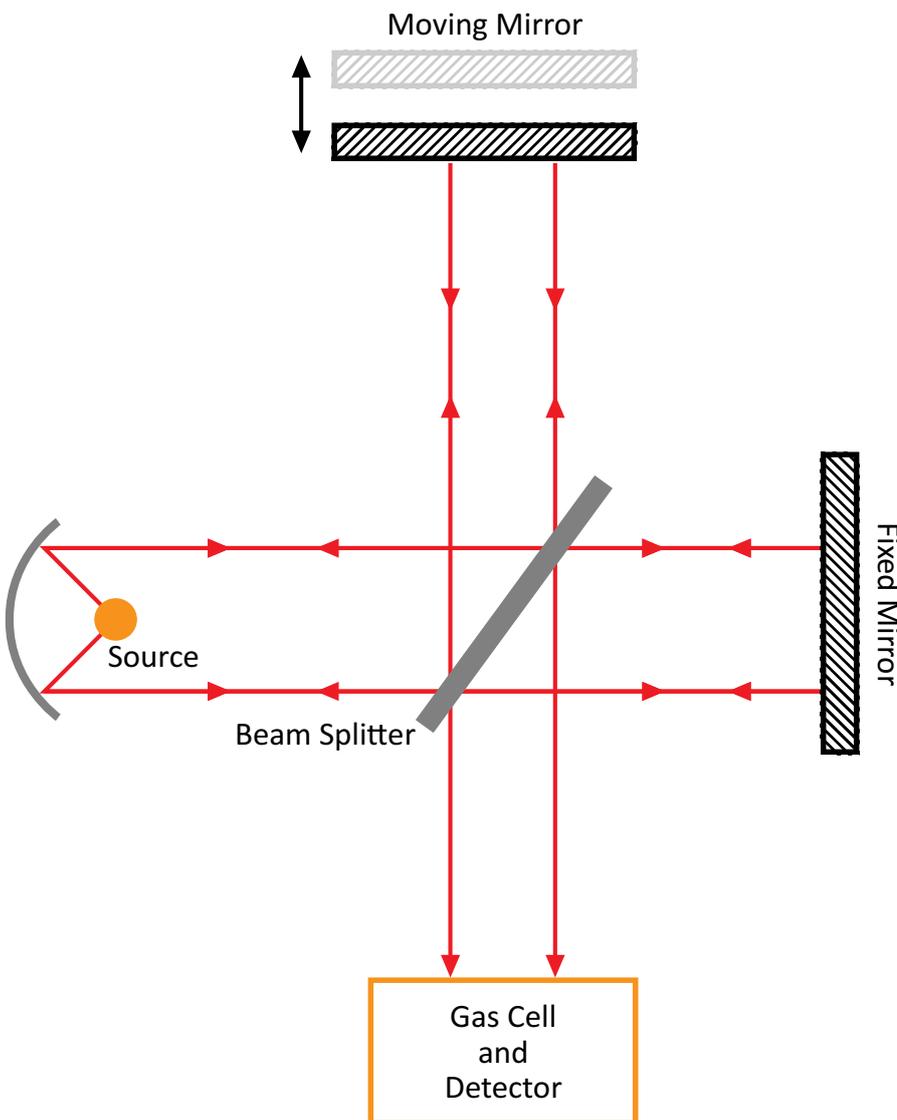
an optical device known as a beam splitter.

The use of a monochromatic or single wavelength light source, typically a laser, is used to provide a reference signal in the interferometer. Measurement of the interference pattern of the single wavelength allows the speed of interferometer's mirror movement and alignment to be controlled precisely.

The light is passed through a sample compartment which is often referred to as the sample cell or gas cell. Sample cells can be of various designs in order to achieve the most suitable pathlength i.e. total length that the IR light passes through the absorbing medium. For long pathlengths (of the order of meters) this usually involves the use of mirror arrangements to bounce the light through the sample medium. As the sample cell contains the extracted sample medium, care has to be taken that the sample cell is constructed of suitable materials and operates at the required temperature and pressure.

An infrared detector, e.g. deuterated triglycine sulfate (DTGS) detector, and associated electronics are required to make single point measurements of the infrared signal as the interferometer scans.

An FTIR analyser does not directly produce a spectrum for analysis; an interferogram is produced. This is time-domain measurement of IR signal and contains the modulated wave of the entire broad band source. To extract the IR spectrum a mathematical manipulation called a



Classic Michelson Interferometer

Fourier Transform must be applied to the interferogram. The mathematics of this are all handled in software in real-time.

The resulting single-beam or intensity spectrum is then compared against a zero or background spectrum to produce an absorbance spectra. This absorbance spectrum is what we need to run a spectral analysis, applying Beer's Law.

Beer's Law describes the linear relationship between IR absorbance and concentration when variables such as temperature, pressure and path length are kept the same. With absorbance spectra collected and saved, chemometric techniques can be applied to extract concentration information. FTIR spectroscopy is considered the most suitable analytical technique for measuring toxic gas species in fire effluents because:

- a variety of gases across wide concentration ranges can be determined by a single method;
- monitoring of species development throughout the fire is possible with time resolved measurements;
- toxicants can be identified or reanalysed retrospectively in the stored spectra from previous experiments.

FTT FTIR System

FTT has been at the forefront of supplying a turnkey solution of FTIR system in analysing toxic gases in fire effluents. The makeup of this turnkey solution comprises of an advanced FTIR analyser, heated sampling system including all the pneumatics, control/processing



FTT FTIR (left) and FTT Smoke Density Chamber (right)

electronics and an industrial PC, which are mounted in a 19" cabinet for easy accessibility and service.

FTT FTIR is an advanced FTIR gas analyser used for continuous gas monitoring in conjunction with FTT's Cone Calorimeter, Smoke Density Chamber and Single Burning Item (SBI) for online measurements of combustion gases in fire tests.

Spectroscopic data are often complex, containing large numbers of features which often overlap. The analysis of gases in fire effluents is especially challenging due to the great number of different organic and inorganic chemicals which representative atmospheres can contain.

FTT FTIR software uses chemometrics to resolve data into

meaningful and accurate information. It offers users the ability to perform chemometrics analysis on data sets. This software is designed so that untrained users can simply run preloaded models, but will also allow more advanced users to build and develop models.

FTT's application specialists have experience of developing and implementing chemometric techniques on various projects. We can provide in depth training courses on chemometric techniques and data analysis of spectroscopic measurements, enabling users to fully benefit from this powerful software.

As any chemometric technique will only ever be as good as the calibration data it is based on, FTT FTIR is calibrated in a purpose built

calibration lab using certified traceable standards.

FTT FTIR is fully configurable to meet the requirements of EN 45545-2, ISO 19702 and IMO standards. In addition, various process monitoring applications are also possible. Measured components and calibration ranges can be selected according to application.

FTIR Gas Analyser

The FTIR gas analyser is an integral part of the system which allows simultaneous measurement of multiple gas compounds.

Typically concentrations of H₂O, CO₂, CO, SO₂, NO, NO₂, N₂O, HCl, HF, NH₃, etc. are continuously measured.

The analyser has a multi-pass gas cell which is heated to 180°C. The gas cell mirror is gold plated with protective MgF₂ coating which ensures high performance even in high water vapour concentrations or corrosive gases.

The analyser also has an internal solenoid valve to allow zero gas (usually 99.999% N₂) to pass to the gas cell for cell evacuation and zero background measurements. This

can be set as a Normally Open (NO) valve which provides a failsafe in case of power failure to ensure the gas cell is purged and gas does not condense on the optics.

Pressure transducer is installed to monitor the pressure inside the gas cell. Fluctuations in the cell pressure will be corrected for in real-time by software.

Sampling System

The hot extractive sampling system consists of a heated sample probe, heated filter, heated sample lines and heated pump unit. The whole system is kept at 180°C to avoid condensation and subsequent washing of soluble fire gases out of the sample. Two stage particle filtration is used in order to remove particles from the sample gas. The sample pump unit includes gas connections for the FTIR gas analyser. All sample lines have a PTFE core sample line of 6mm OD, 4mm ID, together with a secondary line for calibration/span gas. End fixtures are stainless steel which is robust and provides long lifetimes.

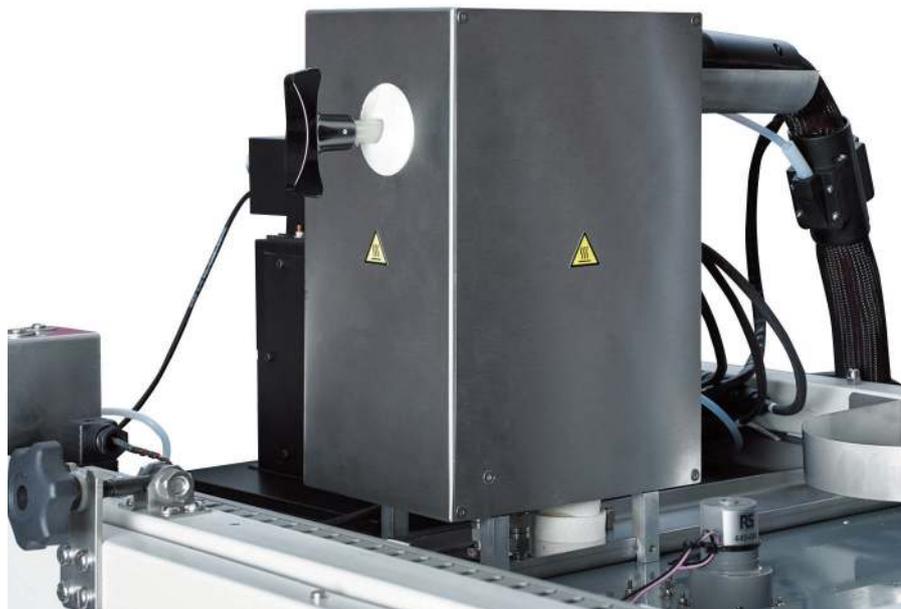
Panel PC

The touchscreen panel PC is required to operate the analyser, to control the sampling system, to translate measured and analysed concentrations and send alarms to higher level automation and control systems. It is also used for processing and storing the sample spectra.

FTT FTIR is supplied with a suite of three analytics software.



FTT FTIR System Interior (front door opened)



Heated filter/valve unit, PTFE filter can be easily replaced from the front

valuable tool for the analyst working with spectra, e.g. adding, subtracting, multiplying and dividing spectra, peak position locator, baseline correction, etc.

PAS – From Spectrum to Results

The advanced, easy to use PAS software provides outstanding analytical performance. It analyses the sample spectrum using sophisticated chemometrics. It is capable of simultaneous detection, identification and quantification of multiple gas species.

Cross-interference effects are compensated for and analysis accuracy is maintained even when analysing complex gas mixtures where there is a possibility of spectral overlapping. Resolution is carefully optimised to meet requirements in fire tests. This allows the collection of several measurements every minute whilst retaining high sensitivity. PAS also allows for model switching based on constants or other variables. For example, two chemometric models can be built over different ranges, one 0-100ppm, and one 100-

i. PAS-Pro

Analysed Software for Process (installed on built in touchscreen PC) PAS-Pro is a very simple to use but comprehensive user interface with setup menus for running the FTIR and control of all system parameters. It displays real time analytical measurements for the selected gases with an optional “Pass” and “Fail” result quality indicator next to each measurement. There is an alarm window which indicates any faults with the system and an event log updates with each task the system has carried out.

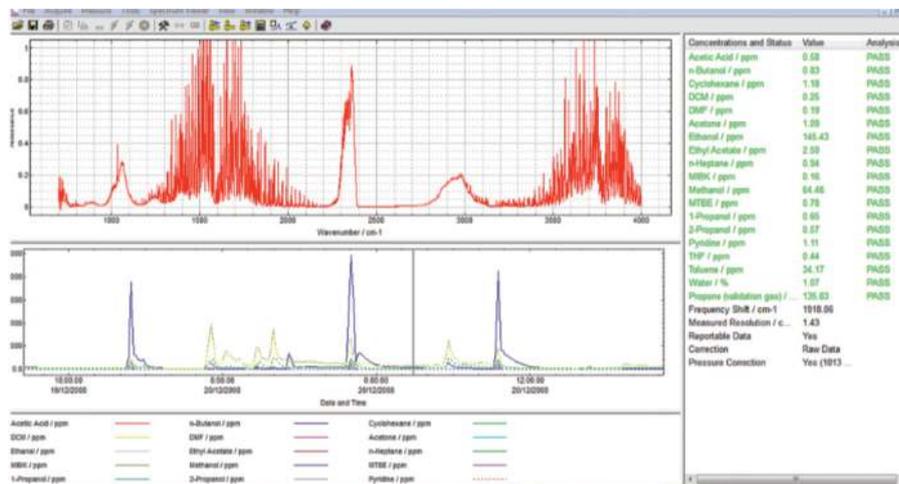
acquisition parameters and allows complex analytical models to be built. Models can be span corrected to match reference gas cylinders and linearity checks can be made for compliance with performance standards.

iii. Spectrum Viewer (installed on standalone PC)

It is a standalone application for manipulating spectra and identifying species that are present. It contains no analytical or collection routines, but is a

ii. PAS Analyser Software (installed on standalone PC)

Collected FTIR data (results and spectra) can be downloaded from the test station and transferred to another PC for further analysis using PAS which allows the analysis to be checked and interfering species to be identified. It gives the user complete flexibility over the FTIR



1000ppm. PAS will automatically switch and use the most suitable model for the current concentration mix.

PAS software is designed for easy and efficient processing of the results. Advanced modelling methods of PLS (Partial Least Squares) are used for accurate and robust analytical predictions, even in the presence of unknown interfering gases. PLS models are built on a component specific basis

and are factor based, constructing a model with the optimum number of factors that models the specified gas in the full matrix.

There is no limit to the number of gases that can be measured at one time with PAS software. Sample spectra are stored as separate files on the computer, they can be easily reanalysed with different analysis settings for previously unknown interfering gases. PLS models are built on a component specific basis

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Key Features and Benefits

- Complete turnkey solution for reliable and accurate analysis of toxic gas species in fire effluents
- Time resolved results enabling continuous monitoring of multiple gas species development
- Hot extractive sampling; no sample loss or change of composition
- Fully automated measurement system with comprehensive safety functions
- Fully modular system for maximum flexibility
- Fully configurable to meet requirements of ISO 19702, EN 45545-2 and IMO standards.
- Capable of individual analysis of airborne concentrations of CO, CO₂, NO, NO₂, SO₂, HCl, HF, Phenol, Acrolein, water vapour, etc.
- Powerful PAS software suite for different operational and analysis requirements
- Span and linearity correct models
- Pressure correction, dilution correction, dry/O₂ correction of data
- Spectra saved with date and time stamped name
- Specially configured file contains all chemometric models and analysis information
- Final results selection allows best model to be selected for given range
- Report concentration results as ppm, mg/m³ or %Vol

TEST PARAMETERS**General Parameters**

Measuring Principle	FTIR (Fourier Transform Infrared) Spectroscopy
Performance	Unlimited simultaneous analysis of multiple gases, preloaded analysis for 21 gas species
Operating Temperature	+5°C to +35°C, non-condensing, dust free ambient air
Storage Temperature	20 - +60°C
Response Time (T ₉₀)	Depending on the gas flow and measurement time
Resolution	4 cm ⁻¹ standard. 1 cm ⁻¹ optional
Wavenumber Range	399.718 – 5000.088 cm ⁻¹
Gas Cell	Ni-plated aluminium gas cell of volume 0.3 litres Ambient pressure during normal operation
Gas Cell Temperature	180°C (variable)
Gas Cell Volume	0.3 litres
Gas Cell Path Length	4.2 m (standard, but changeable)
Optics	Non-hygroscopic Zinc Selenide beam splitter Barium Fluoride gas cell windows (changeable dependent on application) Diamond turned aluminium gas cell mirrors with protected gold coating
Reference Laser	Solid state laser
Source	MidIR source, ceramic Globar With advanced electronic stabilisation and temperature measurement
Detector	DTGS
Sample Gas	Non-condensing, particle free
Flow Rate	Approximately 4 l/min (variable via external flow orifice)
Sample Gas Pressure	Ambient
Dimensions	600 mm (L) × 600 mm (W) × 1400 mm (H) (not incl. castors or plinth)
Net Weight	Approx. 125 kg

Measuring Parameters

Zero Point Calibration	24 hours, calibration with Nitrogen (5.0 or higher N ₂ recommended)
Zero Point Drift	< 2% of measuring range per zero point calibration interval
Sensitivity Drift	< 2% of measuring range over 24 hours
Linearity Deviation	< 2% of measuring range
Temperature Drifts	< 2% of measuring range per 10 K temperature change
Detectable Limits	Gas dependent, but all <2% measurement range
Pressure Influence	1% per 1% change in sample pressure. Pressure measured and compensated for in gas cell

Heated Line	
Tube Size	4 mm ID/ 6 mm OD
Core Material	PTFE core
Operating Pressure	Max. 400 kPa (4 bar)
Temperature	180°C
Fittings	6mm Swagelok
Power Supply	230 VAC or 115 VAC
Power Density	90 Watts/metre
Length	Varies for different application requirement. Lengths from 3 metre to 50 metre can be supplied

Electrical Connections	
Main Supply	115V or 230V 50/60Hz
Power Consumption	The full system including the FTIR Gas Analyser, Touchscreen PC, Heated Filter/Valve Sampling Unit, Sampling Probe and Heated Sampling Lines approximately 2 kW

Gas Species						
• H ₂ O	• CO ₂	• CO	• NO	• NO ₂	• N ₂ O	• SO ₂
• HCl	• HCN	• HBr	• HF	• NH ₃	• CH ₄	• C ₂ H ₆
• C ₃ H ₈	• C ₂ H ₄	• C ₆ H ₁₄	• HCHO	• C ₆ H ₅ OH	• C ₃ H ₄ O	• COF ₂
GAS	UNIT	RANGES				
H ₂ O	%Vol	0-30				
CO ₂	%Vol	0-2		0-5		
CO	ppm	0-3000		0-10000		
NO	ppm	0-500				
NO ₂	ppm	0-500				
N ₂ O	ppm	0-500				
SO ₂	ppm	0-1000				
HCl	ppm	0-100		0-5000		
HCN	ppm	0-500				
HBr	ppm	0-100		0-1000		
HF	ppm	0-100		0-1000		
NH ₃	ppm	0-500				
CH ₄ (Methane)	ppm	0-1000				
C ₂ H ₆ (Ethane)	ppm	0-100				
C ₃ H ₈ (Propane)	ppm	0-100				
C ₂ H ₄ (Ethene)	ppm	0-100				
C ₆ H ₁₄ (nHexane)	ppm	0-100				
HCHO (Formaldehyde)	ppm	0-20				
C ₆ H ₅ OH (Phenol)	ppm	0-200				
C ₃ H ₄ O (Acrolein)	ppm	0-300				
COF ₂ (Carbonyl Fluoride)	ppm	0-50				

Due to the continuous development policy of FTT technical changes could be made without prior notice.

SERVICES

Power Supply	230 VAC – 50/60 Hz – 13 A
Gas Supplies	Purge gas: dry, filtered, oil-free compressed air at 1.0 bar, flow rate approx. 1 ℓ/min, with pressure regulator Zero gas: Nitrogen 5.0 at 1.0 bar, flow rate approx. 3 ℓ/min, with pressure regulator Check gas: typically 200 ppm Sulphur Dioxide + 90 ppm Ethylene + balance of Nitrogen at 1.0 bar, flow rate approx. 3 ℓ/min, with pressure regulator
Extraction	Exhaust from the analyser flowing at 4 ℓ/min through a 6 mm tube must be vented safely to atmosphere
Operating environment	15°C-25°C, non-condensing atmosphere

Unrivalled Experience in Design and Manufacturing

FTT's site in East Grinstead, is home to the largest group of fire scientists and instrumentation design engineers working on fire testing instrumentation, and is at the heart of our design and manufacturing. For almost 30 years

FTT has provided the highest quality instruments and service for fire testing and research professionals worldwide, directly and through its extensive global sales and support network.



Quality

- World-class manufacturing in accordance with multiple international and national standards, including: EN, ISO & ASTM
- ISO 14001, ISO 9001 certified

Integrity

- A dedicated team passionate about fire testing instrumentation and continuous product improvement
- Delivering reliable, robust and easy-to-use instruments for the past 30 years

Excellence

- A world-class team made up of qualified fire scientists, mechanical, electrical and electronic fire instrument design engineers and production, installation and maintenance engineers

Global

- World-wide distribution network for global sales, installations, training, maintenance and technical support
- Leading global supplier of the Cone Calorimeter, Large Scale Calorimeter, NBS Smoke Chamber and Oxygen Index