

Development of an On-Board System for the Simultaneous Analysis of Tailpipe Exhaust and Ambient Air

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ABSTRACT

An on-board sampling and analysis system has been developed, capable of measuring on-road emissions at extremely low levels. Ambient air quality can also be monitored and compared to tailpipe emission levels in real time during actual roadway driving. This FTIR-based system can measure several chemical species, including HC, NO_x, and CO. Hardware and software were developed to facilitate the monitoring of target species at concentration levels below 1ppm, in the harsh environment of an on-road vehicle. Countermeasures were required to mitigate or eliminate the effects of chemical interferences and vehicle motion. Algorithms were developed to calculate results on a mass basis (g/mi), thereby allowing such data to be acquired in real-world conditions outside a laboratory environment. This system has been used in the development of near-zero-emission vehicle technology; it has allowed instant assessment of vehicle software tuning during actual roadway driving. It is currently being used to determine the true impact of various advanced-technology vehicle classes upon the environment. In this capacity the system is performing a dual role as an on-board analyzer and also as a high-sensitivity ambient air monitoring station.

要 旨

極低濃度レベルの路上エミッションを測定できる車載分析計を開発した。この装置では、大気レベルの濃度も計測でき、走行中にリアルタイムに測定された排気ガス濃度と比較する事が可能である。このFTIRを用いたシステムはHC、NO_x、並びにCOを含むいくつかの化学物質を測定する事ができる。ハードウェアとソフトウェアの両面から開発が行われ、実車という非常に厳しい使用環境の下で1ppmという非常に低濃度の計測が可能となった。化学物質同士の干渉や自動車からの振動の影響を取り除いたり、最小限に止めるために幾つかの対策が必要であった。また、濃度から排出量(g/mile)への変換のために計算アルゴリズムを作成した。これにより、実験室外の実社会の条件で排出量を求める事が可能となった。このシステムは極限エミッション車を開発するために使われてきた。このシステムにより実走行で即座に排ガス制御システムのセッティングの評価ができるようになった。現在、さまざまな環境条件で色々な先進技術車がどのようなインパクトを与えているかを測定するために使われている。このシステムは、車載の分析装置としてだけでなく、非常に高感度の大気モニタリングステーションとして活用する事が出来る。

1. Introduction

1.1. Background

Tailpipe emission rates are commonly determined by operating a vehicle on a chassis dynamometer under strictly controlled, reproducible conditions. For example, the US FTP-75 emission test cycle is used in California to assign emission categories; i.e., LEV, ULEV, etc. However, to achieve a more complete assessment of a vehicle's impact upon the environment, laboratory testing must be augmented by evaluating real-world emission

performance. This had previously been attempted by mounting bulky measurement equipment in a vehicle, or by towing such equipment behind in a trailer. The performance of the vehicle was thereby altered, and true real-world emission data could not be collected. Furthermore, instrument calibration and data reduction were complicated, due to the use of several discrete measurement devices, each with its own sample inlet, data format, sampling rate, etc.

A new concept was therefore required to address these issues. To this end, a relatively compact,

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integrated on-board sampling/analysis system has been developed. This system can measure the concentration of several chemical species from the tailpipe exhaust and ambient air simultaneously, while the vehicle is driven on the highway. In addition, the system can be easily modified to function as a stationary, highly sensitive air monitoring station.

This system has been used in the development of advanced low-emission vehicle technology, and to confirm the robustness of such technology under varying driving conditions on actual city roadways⁽¹⁾. The data from this on-board analysis system can also be used to develop better estimates of advanced-technology emission system inventories for use in air quality simulation studies.

1.2. Objectives

Advancements in emission control technology have led to the development of vehicles with progressively lower tailpipe emission levels; in some cases, the concentration of criteria pollutants approaches zero. Therefore, the target for this project was set to these “near-zero” concentration levels. These concentrations are similar in magnitude to those encountered in ambient air. The following requirements were placed upon the development of this system:

- Target components = NMHC, NO_x, and CO
- Limit of detection (LOD) near 0.1 ppm, with acceptable accuracy and precision
- Minimal weight burden on vehicle
- Robust and verifiable
- Integrated system with real-time data acquisition and display

1.3. Choice of analytic technique

Given the requirements above, it was decided to use Fourier Transform Infrared Spectroscopy (FTIR) as the basis for this on-road analyzer. Acquiring data with a single system would facilitate synchronization with engine parameter data (simultaneously recorded on a separate computer). Fieldwork logistics would be simplified because an FTIR requires no daily calibration per se. As an added benefit for future research, FTIR data can be re-processed to reveal concentration data of other components. (This is possible because the spectrum recorded by the FTIR contains concentration information for all infrared-sensitive components.) However, the use of this technique presented some special challenges:

- Spectral interference (primarily from H₂O and CO₂)
- Signal-to-noise ratio affected by external vibration and instrument orientation
- Demanding accuracy and LOD requirements
- Response time

Other single-system approaches that were considered for this project included non-dispersive IR, high-resolution mass spectrometry, and gas chromatography - mass spectrometry. These were rejected due to sensitivity, size, and response time considerations, respectively.

2. Experimental

2.1. System overview

The initial test platform chosen for this project was a prototype Honda ZLEV (Zero Level Emission Vehicle). The exhaust emissions from this gasoline-powered vehicle are less than 1/10 ULEV levels⁽²⁾. An overview of the sampling and measurement systems is shown in Figure 1. Intake air was sampled from the front of the vehicle, and an exhaust sample was taken via a heated line inserted into the tailpipe. The samples were drawn through a water-removal system by a dual-head pump, and then directly into the gas cells of twin FTIR spectrometers. System power was supplied through a DC-AC inverter from three nickel-metal hydride battery cells. Results were processed by two networked 400 MHz computers, and combined onto a single touch-screen flat panel display.

2.2. FTIR principles

In infrared gas analysis, IR light is passed through a sample in a gas cell. Some of the infrared light is absorbed by the sample and some is passed through. The measurement of these absorbed and transmitted wavelengths of infrared light constitute an infrared spectrum; see Figure 2. Since no two chemical species have the same infrared spectrum, the spectrum serves as a fingerprint for identifying different gas components in the sample. The intensity of the infrared absorption indicates the quantity of the component in the sample.

Unlike dispersive IR spectrometers, an FTIR instrument can acquire an entire spectrum in less than one second; i.e., it can record all frequencies simultaneously in this time. FTIR uses an interferometer with a moving mirror to modulate the light by phase, producing an interferogram. This interferogram is then Fourier transformed, thereby producing the sample infrared spectrum in frequency or wavenumbers (cm⁻¹).

The infrared spectra were analyzed using a “classical” least squares (CLS) pattern recognition algorithm⁽³⁾. This data reduction technique compares spectra of reference gases to sample spectra, determining a degree of fit of the reference gas spectra to the sample spectrum. The degree of fit is then reported as a concentration.

2.3. Gas cell

One of the key components of an FTIR system is the gas cell. The sensitivity of the instrument is directly proportional to the amount of sample that is exposed to the infrared beam. FTIR gas cells typically use the *White* optical design, in which multiple internal reflections occur. The cell is classified based upon the total “path length” of these reflections. For example, the gas cell chosen for this work was less than 50cm in length, yet had a path length of 10m.

Initially, a 2m path length gas cell was evaluated for this application, due to its favorable size and weight characteristics. It quickly became clear that the 2-meter cell would not achieve the target noise level, and therefore the required LOD could not be attained.

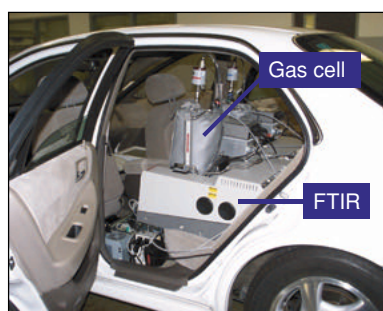
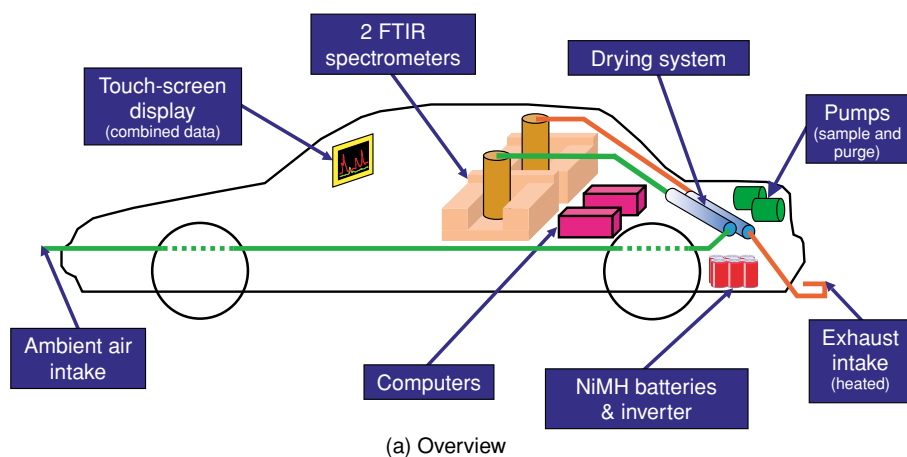


Fig.1 On-board sampling/analysis system

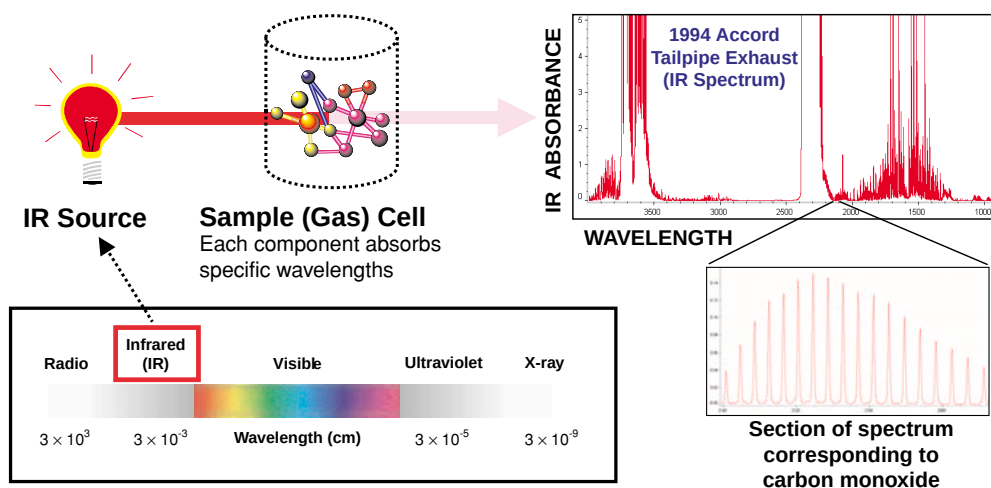


Fig.2 Principle of infrared spectroscopy

Acceptable results were obtained upon switching to a 10-meter cell; Figure 3 shows the improvement in sensitivity. The strong infrared absorption bands from (exhaust) water became a problem with the increase in path length, as discussed later in this paper. The longer sample mixing time resulting from the 10m cell's larger internal volume was also of some concern, but the final system provided adequate temporal resolution. The 10m cells can clearly be seen in Figure 1b.

2.4. Calibration

The initial calibration of an FTIR gas analyzer is performed similarly to that for a non-dispersive infrared or a chemiluminescent analyzer. In this case, spectra of single-component reference gases (in a nitrogen matrix) were acquired, and the resulting patterns used in a CLS spectral data analysis. The absorbance response of a reference gas coupled with its concentration determined the calibration. A sequence of concentration steps was

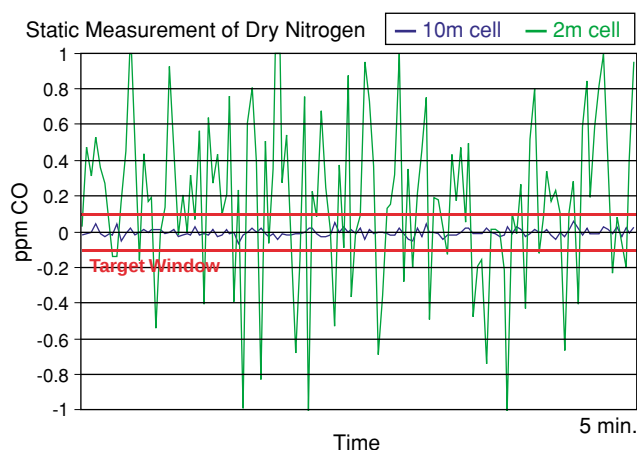


Fig.3 Effect of gas cell path length upon measurement precision

added in order to calculate a non-linear correction, when necessary.

A different approach was required for the calibration of the non-methane hydrocarbon component. Instead of using reference spectra for the individual hydrocarbon species, it was desirable to use a spectrum of a representative mixture of hydrocarbons as the reference gas in the CLS data analysis. The mixture used to calibrate the FTIR analyzer was a bag sample of engine exhaust from a prototype ULEV sampled during the first 30 seconds after a cold start. A concentration for the non-methane hydrocarbon component in the bag sample was assigned using careful bench FID and gas chromatography analyses.

2.5. Sample treatment

Exhaust gas from gasoline-powered internal combustion engines contains roughly 10 to 15 mole% CO_2 and H_2O . Both pose interference challenges for the FTIR analysis. Dilution was never considered; the resulting sensitivity loss would have been unacceptable for this project. The CO_2 problem was mitigated by the careful choice of analytic regions in the spectra, aided by the 0.5 cm^{-1} spectral resolution. However, the H_2O spectral features proved too pervasive (Figure 4); a similar countermeasure was precluded. Consequently,

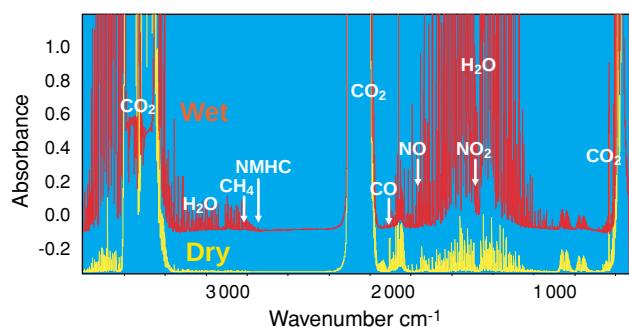


Fig.4 Effect of water upon exhaust spectrum
Note that water occludes the spectra of many target species

various techniques were tried to remove the water from both the ambient and tailpipe samples. A technique using an ice-chilled aluminum heat exchanger, water trap, and desiccants efficiently removed the water. As expected, NO_2 was unfortunately removed with equal efficiency. Studies with NO , CO , and C_3H_8 (propane) indicated no loss in this water-removal system. Since actual exhaust gas contains virtually no propane, a 15-component hydrocarbon mixture was also tested. Analysis by gas chromatography⁽⁴⁾ revealed that substantial hydrocarbon losses were occurring as early as C_6 , necessitating the search for a different drying technology. (Data are shown later.)

A drying system based on the polymer Nafion[®] was assembled for evaluation. Nafion has a Teflon[®] backbone, with periodic side chains of perfluorated ether terminating in a sulfonic acid group. These acid groups form “ionic channels” that extend through the walls of the Nafion tubing. Each sulfonic acid group can absorb up to 13 molecules of water. Assuming the partial pressure of water in the sample exceeds that external to the Nafion tube, water molecules will travel along the ionic channels and be released outside the tubing. This mechanism is selective for water; the only (potential) exhaust species that could experience some loss are alcohols, ketones, and ammonia.

For this application, multi-tube Nafion dryers manufactured by Perma Pure Incorporated were selected for use, after comparison to a single-tube configuration. This dryer consisted of a bundle of 0.76mm (OD) Nafion tubes contained within an aluminum shell. Fittings near each end of the shell permitted a purge gas to flow countercurrent to the sample; Figure 5. Two dryers in series were used for both of the sampling systems.

The exhaust sample was heated (70°C) from the tailpipe through half of the first dryer. There were two reasons for this: (1) the interaction of liquid water with Nafion is endothermic, and leads to a cascade failure reaction. Therefore the exhaust gas must be kept above the dew point temperature until most of the water has been removed. (2) The initial water absorption and transport by Nafion proceed as a First Order Kinetic reaction, in which absorption becomes more rapid with increasing temperature⁽⁵⁾.

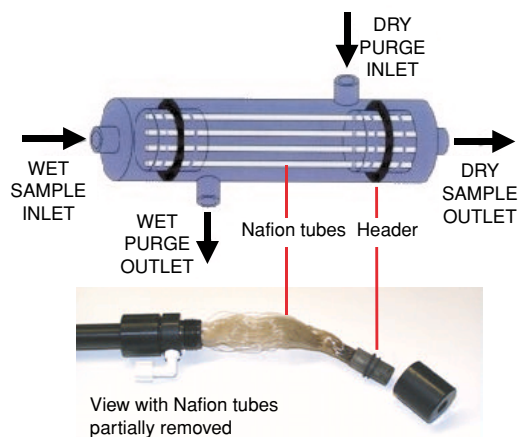


Fig.5 Concept of Nafion-based sample drying

The second dryer for each sample stream was blanketed with ice water. The purpose here was to coax more of the retained water from the sulfonic acid groups, since this residual water becomes the limiting factor after the initial First Order reaction. Leckrone, et al, have recently quantified this effect⁽⁶⁾.

The ultimate performance of the Nafion dryers is dependent upon the partial pressure of water in the purge gas. Dry nitrogen was considered for this application, but the weight and safety ramifications of carrying a compressed gas on board a passenger vehicle precluded this choice. Ambient air would normally have an insufficient dew point for this application. However, good results were obtained by pulling ambient air through a specially constructed dehydrating system with a vacuum pump. The resulting negative pressure in the dryer shell lowered the partial pressure of the water in the treated ambient purge gas to an acceptable level. The water content of the exhaust gas sample was

decreased to below 100ppm using this technique, equivalent to a 99.9% reduction.

A comparison of the Nafion-based dryer performance with that of previous dryer versions can be seen in Figure 6. The 15-component hydrocarbon mixture was passed through a given drying system at 7 L/min, and collected into Tedlar® bags. These bags were subsequently analyzed on a gas chromatograph, and the resulting concentrations compared to the label values. The Nafion system produced acceptable results for all components, with only a slight (4%) loss for a C₉ aromatic.

A more severe test of the drying system was performed⁽⁷⁾, in which CO₂ (11.5%) and H₂O vapor (10%) were injected into the reference gas streams. As previously noted, these compounds are a potential source of interference for FTIR, and are present in exhaust gas at approximately these concentration levels. In addition to the 15-component hydrocarbon mixture, CO and NO reference gasses were used for this study. The gas chromatography results shown in Figure 7a indicate good recovery for the individual hydrocarbons in the mixture; even when the sample matrix includes CO₂ and H₂O, there was no preferential loss of hydrocarbon species. (The data have been corrected for the effect of dilution, and propane was omitted due to contamination.) Figure 7b indicates the same good recovery performance for CO and NO. It is important to note in this figure that the FTIR system was used to measure the recovery. Therefore, these results also demonstrate the accuracy of the on-board FTIR system, including the ability to measure the full range of hydrocarbon species encountered in vehicle exhaust. This level of accuracy was attained despite the presence of CO₂ and H₂O in the sample gases, indicating that interference from these compounds had successfully been avoided.

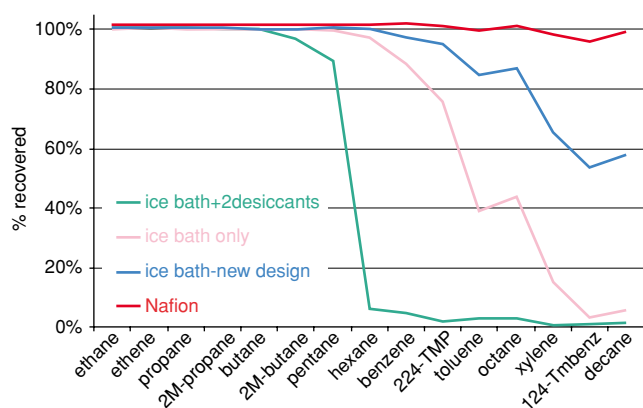


Fig.6 Comparison of drying system performance, in terms of hydrocarbon recovery. (Gas chromatography data)

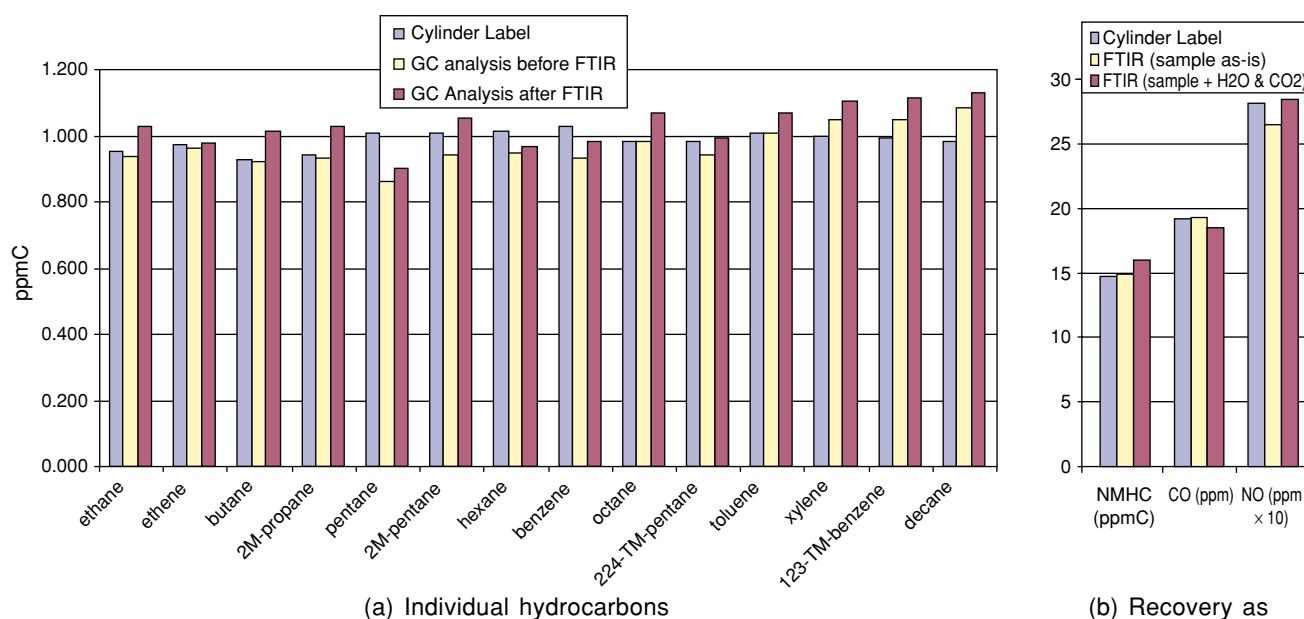


Fig.7 Component recovery through final version of drying system
Sample injected with CO₂ and H₂O.

2.6. System environment

FTIR instrumentation is basically designed for use on a vibration-free laboratory bench in a controlled environment. Relocating such instrumentation to the interior of a passenger vehicle is challenging, given the unpredictable dynamics resulting from road surface irregularities and vehicle motion. One of the initial concerns was the effect of the vehicle acceleration force vector upon the interferometer (moving mirror) component of the FTIR. Nicolet suggested that this force should be perpendicular to the motion of the mirror; (Figure 8). This configuration did in fact prove to be best, although the mirror was still subjected to coaxial forces during vehicle turning maneuvers.

The main area of concern was the unpredictable mechanical vibration and shock transmitted to the FTIR as the vehicle encountered imperfections in the road surface. An attempt was made to characterize these forces through the use of an accelerometer, but the test results only confirmed our prediction that the vibration frequencies varied widely. Several studies were performed involving dampening systems, changes in FTIR location, etc. The final conclusion was that the

FTIRs were best isolated by simply placing them on the rear seat of the vehicle. As an added benefit, placing the FTIRs side-by-side on the rear seat exposed them to an identical environment, and the instrument electronics could be kept within the specified temperature range.

Finally, each FTIR received some internal modifications; refer to Figure 8. For example, the laser and IR source were secured more firmly to prevent movement.

3. Results and Discussion

3.1. System specifications

The final system specifications were as follows. Information is shown for a single sampling/analysis system.

Analytic system

FTIR base unit : Nicolet model Protégé™ 460

Detector : MCT, LN₂ cooled

Spectral resolution : 0.5 cm⁻¹

Gas cell : 10m path

Sample conditions : 70°C, 93kPa (absolute)

Sample flow rate : 12 L/min (max)

Scans/sample : 2 complete

Drying system

Nafion dryer : 0.76mm (OD) tube bundle

Purge gas : dehydrated ambient air under negative pressure

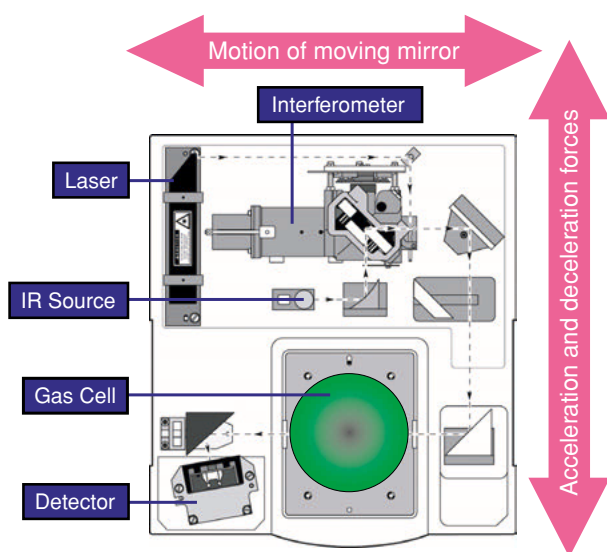


Fig.8 Overhead view of FTIR interior

3.2. System validation

In addition to the previously mentioned accuracy confirmation, numerous other validation studies were performed. A typical example can be seen in Figure 9. In this case, sensitive bench instruments in use as ambient air analyzers were used for reference. Actual exhaust gas samples were analyzed in addition to reference gases. In the case of the reference gases, the on-board FTIR results were also compared to the gas cylinder label concentrations. To prepare the exhaust samples, a ZLEV-class vehicle was started under cold conditions, while a slipstream of undiluted exhaust gas was pumped into a large Tedlar bag. As can be seen in Figure 9, the FTIR system compared very favorably with the bench analyzers for the exhaust samples and gas mixtures.

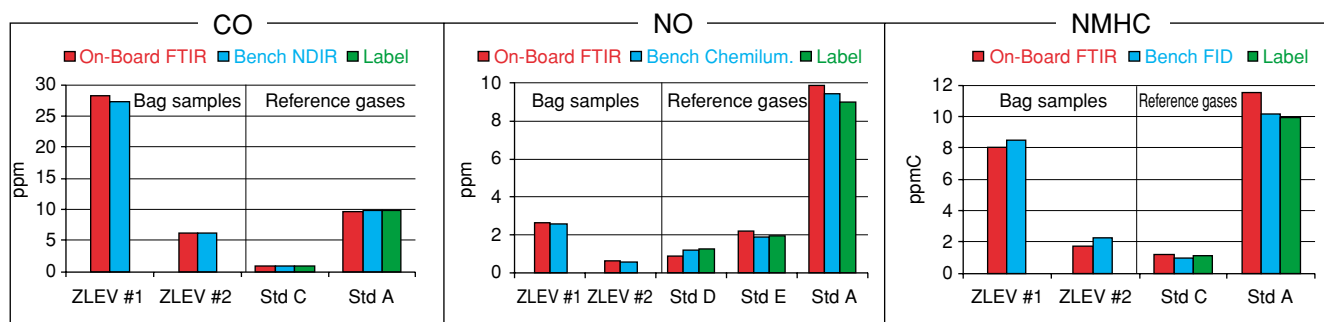


Fig.9 Comparison of on-board analyzer and bench instrumentation

3.3. On-road results

Following an on-road test program in Los Angeles, the system was sent to Japan to evaluate its performance in the Tokyo area. A representative example from the Tokyo testing is shown in Figure 10. Roadway (ambient) pollutant concentrations were found to be roughly similar to those measured in the Los Angeles area. Traffic conditions were typically stop-and-go, alternating with cruise periods.

During the course of testing, severe conditions were sometimes encountered in which the roadway pollutant concentrations of CO and NMHC were an order of magnitude greater than those shown in Figure 10. Long tunnels in the Tokyo area and highly congested traffic near Detroit are examples of these conditions. As can be seen in Figure 11, the ZLEV test platform was unaffected by this severe ambient air quality.

The data shown in Figures 10 and 11 are not smoothed, other than two-scan averaging performed by the FTIRs. This produced an approximate two-second sampling rate for this data set. The detection limit for all target compounds was approximately 0.1 ppm for such on-road testing. For this road test, NO₂ remained below the detection limit for both sample streams; the observed NO_x concentrations resulted from NO alone. In the rare cases where NO_x concentrations over 5 ppm were measured in the ambient sample during road tests, NO₂ was found to contribute approximately 20% to the total NO_x value. This ratio is similar to that expected in diesel vehicle exhaust⁽⁸⁾.

3.4. Off-road measurement of ambient air

As part of a cooperative research program with the University of Riverside, a project was initiated which required the measurement of ambient air quality at various sites in the Los Angeles area. The on-board FTIR system was recruited for this work. In this case, the system was optimized for stationary, ambient-only measurement, and was installed into a Honda Odyssey van. The Odyssey's GPS-based navigation system was used to record the location of each test site. The ambient measurements were made in areas away from the roadway, where pollutant concentrations had become equilibrated and/or mixed. This permitted the collected FTIR spectra to be averaged over a longer period; in this

case 15-second averages were performed before the data point was recorded. These longer sampling intervals in a non-moving platform allowed for excellent signal-to-noise ratios, with a corresponding decrease in the limit of detection. Figure 12 indicates that even challenging species such as NO_x (NO and NO₂) could be clearly measured in sub-ppm concentrations. (The variance in NO observed in this figure is due to actual changes in ambient concentrations.) Subsequent calibration work performed with reference gases demonstrated that NO could be measured well below 0.1 ppm under these conditions.

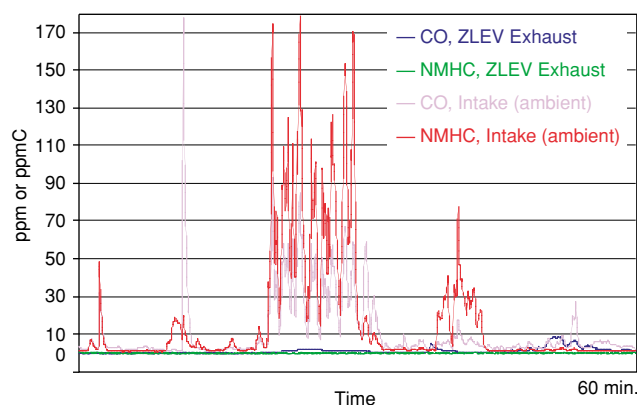


Fig.11 Detroit on-road test results, severe traffic congestion ZLEV with on-board analyzer

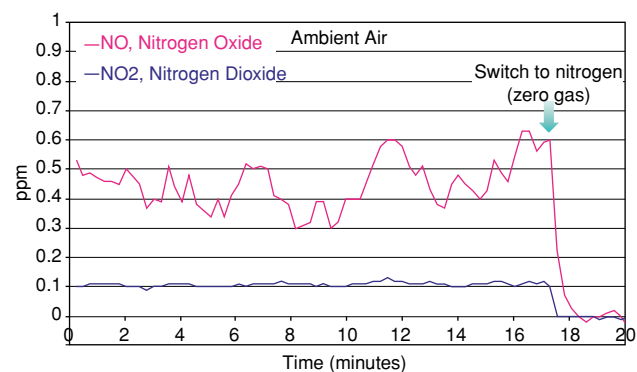


Fig.12 Real-time ambient air measurement Shopping mall parking area, Azusa, California

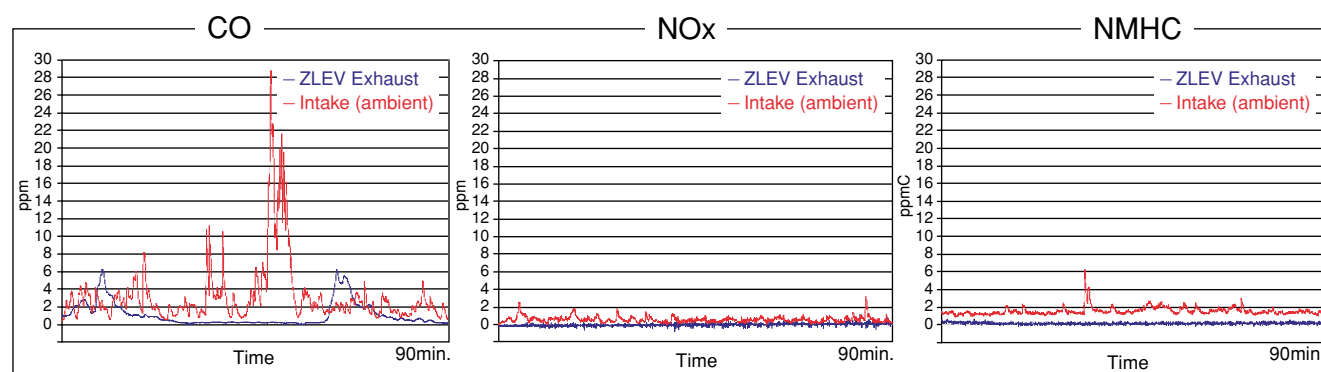


Fig.10 Tokyo on-road test results. ZLEV with on-board analyzer

3.5. Calculation of mass-based results

All data discussed to this point have been in terms of concentration, (e.g. ppm). If the tailpipe flow rate were known, the concentration data could be converted to mass results, (e.g., grams), by the following equation:

$$M = D \int C_t \times V_t dt$$

Where, for each component:

M = cumulative mass

D = density

C_t = concentration at time t

V_t = volumetric flow rate at time t

In reality, the conversion to mass-based results is somewhat more complex. In addition to the flow rate, sample delay time and instrument response characteristics must be considered. These steps are outlined below.

Flow rate determination. Measuring the tailpipe flow rate on a moving vehicle is a challenge. The concept of add-on flow measurement devices was discarded for this work, due to accuracy and size considerations. Instead, an estimate of the flow rate was determined by using data from the vehicle's ECU (engine control unit) to calculate the fuel usage rate. Examples of data extracted from the ECU for this purpose included fuel injector pulse duration, engine speed, and air/fuel ratio. Given that vehicle constants (such as exhaust system dimensions) and fuel composition were known, a sufficiently accurate estimate of the tailpipe flow rate could be calculated. Comparison with external methods of flow measurement, such as the CO_2 tracer technique, confirmed the accuracy of this method.

Sample delay time. The next step was to align this flow measurement with the FTIR's concentration measurement. The delay time between the sampling point and the instrument was determined experimentally with pulsed injection of reference gases. The previous equation was modified to include this delay time, designated δ :

$$M = D \int C_{t+\delta} \times V_t dt$$

Instrument response. The FTIR gas cell has a volume of 2L, and the sample flow rate is about 6 to 12 L/min with this system. Therefore, the concentration data resulting from fast changes in pollutant concentrations will appear somewhat smoothed, due to this relatively slow response time. To address this response characteristic, the data were processed using a "well-mixed flow cell" model; see Figure 13. The final equation shown in this figure was utilized to estimate the actual inlet concentration of the FTIR gas cell prior to mixing. The effect of the delay time and instrument response correction can be seen in Figure 14, in which a very good comparison has been achieved with dynamometer output during an FTP cycle.

Comparison with dynamometer CVS results. As a final validation of the techniques described above, testing was performed at UC-Riverside in which the FTIR

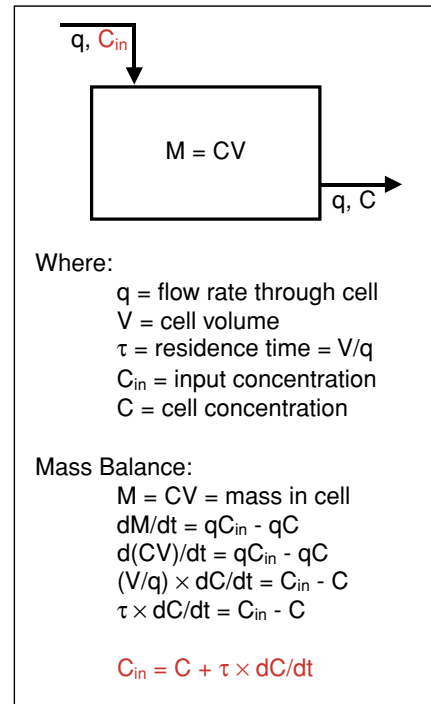


Fig.13 FTIR gas cell mixing model; Determination of input concentration

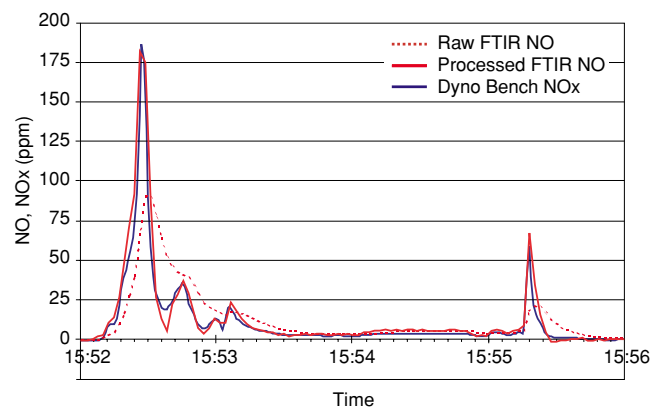


Fig.14 Comparison of on-board FTIR system with dynamometer bench analyzer during FTP
Vehicle = ZLEV

system was run concurrent with standard dynamometer methods over various cycles. To ensure that sufficient pollutant concentration were produced to facilitate this comparison, the FTIR system was moved from the ZLEV platform into an LEV Accord. Figure 15 shows the results from the third phase of a Unified test cycle, (similar to an FTP cycle). These data represent the average of three separate runs. The grams/mile of CO , NO_x , and NMHC were in good agreement between the FTIR-based system and the UCR CVS system. Results from CO_2 and CH_4 (not shown) were also good. This was generally the case for all of the test cycles and phases, although NMHC results were occasionally in relatively poor agreement. The FTIR calibration was

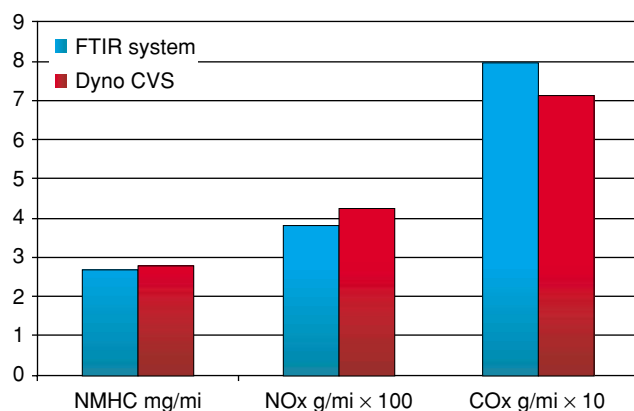


Fig.15 Mass-based comparison of on-board FTIR system with dynamometer CVS. LEV vehicle, 3rd phase of Unified Cycle.

subsequently confirmed over the range of concentrations encountered during these tests. Therefore, the deviation was likely attributable to another source, such as CVS contamination.

4. Conclusion

An on-board analyzer system has been developed, capable of measuring exhaust species from advanced-technology vehicles in real time. Accuracy was validated through laboratory studies, and overall performance was confirmed through on-road data collection.

Therefore, it is now possible to measure exhaust and ambient species in units of grams/mile during actual highway driving, with accuracy equivalent to that of dynamometer CVS data. Several chemical species can be measured simultaneously, in addition to the standard criteria pollutants. Some examples of new technologies and techniques developed during this project :

- Various techniques associated with the installation of a complex sampling/analysis system into a passenger vehicle, including electricity management and vibration mitigation.
- Analytic methods that attenuate FTIR spectral interference, including a unique calibration method for NMHC.
- A new sample treatment technique. The water content of exhaust gas can be reduced by 99.9%, with no loss of criteria pollutants.
- Custom software for real-time, simultaneous display of tailpipe and ambient data.
- Algorithms allowing the calculation of mass-based results.

This on-board analyzer is now in use as a development tool for advanced emission-control systems. It has also proven effective as a mobile air quality monitoring station.

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