

Development Of Low-Cost Ozone and Nitrogen Dioxide Measurement Instruments Suitable For Use In An Air Quality Monitoring Network

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Abstract—We demonstrate low-cost ozone and nitrogen dioxide measurement instruments suitable for use in an air quality monitoring network. The instruments are based on the gas response of tungstic oxide at elevated temperature. We have shown that with careful attention to detail, small, robust instruments can be made, with sensitivity for O₃ and NO₂ less than 10 part-per-billion (ppb), and which track reference analysers in the atmosphere to within 10ppb over periods of months, without recalibration.

I. INTRODUCTION

Air quality concerns every individual and impacts directly on health and productivity. At local scales the temporal and spatial heterogeneity of emission patterns, local wind flow patterns and the complexity of the urban surface results in complex dispersion pathways, giving strong gradients in vertical and horizontal pollutant concentration[1]. In urban areas, people spend a substantial component of their outdoor time near busy roadways and intersections while commuting to work, employed at local shops or cafes or using the pavement space for retail or recreational activities[2]. Although individuals may not remain in this environment for more than a few hours each day, the prevalence of local pollutant hot spots often results in significant exposure. Attempts to measure O₃ and NO₂ distribution have been limited by the instrumentation available. Conventional instruments are based on spectroscopy and chemiluminescence and are bulky, expensive, require significant power, temperature-controlled enclosures and a rigorous calibration and maintenance programme. This limits their deployment in the field. Even in densely populated areas of economically advanced countries, instrument spatial densities are at most 1-5 per square kilometer, as is the case with the London Air Quality Network[3]. Monitoring sites are chosen typically to be representative of ‘average’ or background concentrations in the region and not influenced by

local sources of pollution. At this scale sparsely distributed instruments are used to provide information about pollutant gas accumulation and transportation on a regional or district scale, and data are used to inform local authorities if pollutant levels in their areas are within or are exceeding acceptable levels. However, there remains considerable debate about the intra-region and particularly intra-urban homogeneity of pollutant concentrations due to variations in source strength, meteorology, topography and location of monitoring sites[1]. A limited number of fixed monitoring stations may not provide a good indication of personal exposure to pollutants, so hindering the establishment of strong relations between pollutant concentrations and meteorology[4] or health outcomes[5,6]. Such limitations have led to attempts to deduce the pollutant burden from measurement of traffic density, emission inventories and dispersion modeling[7,8]

There is therefore a great need for the introduction of low-cost instruments which can easily be deployed in a high density network. The results from such a network will help inform the understanding of pollutant dispersion pathways and human exposure. Such data are essential to the formulation of coherent air pollution abatement strategies. The work described in this article is part of an effort directed at enabling high spatial density air quality monitoring through the development of suitable low-cost instrumentation

Gas sensors based on conductivity changes of heated semiconducting oxides[9] are widely used for industrial safety monitoring. The range of oxide materials for which significant conductivity changes induced by the presence of reactive gases in air can be observed is very large. Since the electrical conductivity of all these materials is a strong function of the temperature, devices intended for accurate measurement require accurate temperature control. General principles for understanding the way the observed conductance changes are related to the surface chemistry and microstructure of the

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device have been enunciated[9-11]. The choice of material thus comes down to understanding the signal due to the target gas and interferences in the particular application, to appropriate manipulations of the microstructure, and to compatibility with a chosen manufacturing route for the sensor.

WO₃ shows particularly large resistance-increase signals in response to traces of the oxidizing gases ozone, nitrogen dioxide and chlorine in the atmosphere, with relative magnitude O₃ >> NO₂ > Cl₂. The ozone response shows a strong temperature dependence that can be used to advantage. Very large resistance-decrease signals are also exhibited by WO₃ in response to the presence of traces of H₂S. The resistance-decrease signals in response to the presence of hydrocarbons, solvents, SO₂ and NO, and in response to variations of water vapour pressure, are very much smaller. Thus, as we show below, devices based on WO₃ will specifically measure the NO₂ concentration in the NO₂ – NO mixtures found in the urban atmosphere, and are not responsive to, nor poisoned by, the traces of SO₂ present in the atmosphere as combustion products. There will be an interference if H₂S is present, but this is not in practice a common circumstance. The presence of chlorine would also be very site-specific. Thus, the ozone signal is in practice essentially free from interference whilst for measurement of NO₂, the key is removal of the ozone interference.

While there are reports in the literature of gas-sensitive resistors being used for ambient atmospheric monitoring, there is a perception that the devices suffer from effects such as drifts of zero and calibration slope, and cross-sensitivities to other gases making them unsuitable for quantitative, long-term atmosphere measurement. This is because the technologically most-used material for gas-sensitive resistors is SnO₂. This material shows slow surface hydration-dehydration effects[12], which are particularly important over the temperature range where the gas signals are greatest. It is also slowly poisoned by traces of SO₂[13] and the signals for hydrocarbons and NO are relatively high. WO₃ does not show any of these effects. The response of an oxide-based gas-sensitive resistor is critically dependent on the microstructure; WO₃ has some particular advantages in this respect also, being easily prepared in nano-crystalline form by the thermal decomposition of tungstic acid, itself easily accessible in nano-crystalline form by a variety of simple routes[e.g.14]. The microstructure of WO₃ layers is easily manipulated by alteration of processing temperature and time (fig 1); we have used the resultant effect on the gas response signal to advantage for the control of the signal in response to ozone and NO₂.

The very large conductance response at temperatures in the range 300 – 550°C of highly porous layers of WO₃ to the introduction of part-per-billion concentrations of ozone in air has been discussed in detail[15,16]. Paradoxically, the thermally-catalysed decomposition rate of ozone on the WO₃ surface is sufficiently low that measurement at these rather high temperatures is indeed feasible. The response mechanism deduced was a reaction with surface oxygen vacancies. We note that this mechanism is different to that which has been deduced for the response of other oxides to hydrocarbons or

carbon monoxide, where the species mediating the response is given as a surface electron trap derived from chemisorbed oxygen. We speculate that the very large signals associated with ozone, NO₂ and H₂S on WO₃ are because of the direct reaction of these gases at surface oxygen vacancies, whilst the smaller signals developed by hydrocarbons, CO, etc, are due to reactions at chemisorbed oxygen. The facile formation of surface oxygen vacancies, and the dramatic effect that these species have on the electrical conductivity, is a particular feature of WO₃.

There are preliminary reports in the literature describing the use of WO₃ sensors in ozone measurement instruments[17,18]. Long-term drifts in baseline were a significant issue dogging these first attempts to develop monitoring instruments based on WO₃ sensor elements[18]. The mechanism deduced was a slow exchange of oxygen vacancies between the surface and interior of the micro-crystals making up the device, and this deduction has led to a solution to the problem. Thus, it has been shown that the conductance of the porous sensor assembly can adequately be described as a parallel equivalent circuit, with one element (the ‘interface’ element, representing the surface zone of the individual grains of the material) being dependent on the ozone concentration, and the other (the ‘bulk’ element, representing the interior of the individual grains) being independent of the ozone concentration but dependent on oxygen partial pressure. The predicted variation of conductivity, σ , with ozone concentration, P_{O_3} fitted the data well. The conductance difference in the presence and absence of ozone did not vary over several months of operation, consistent with the prediction of the assumed mechanism and equivalent circuit[16]:

$$\sigma_{(O_3=0)} - \sigma = \frac{aP_{O_3}}{bP_{O_3} + 1} \quad (1)$$

For measurement over the ozone concentration ranges found in the atmosphere, in eq 1, $bP_{O_3} \ll 1$, so, $\Delta\sigma$ denoting the difference of conductance measured at low and high flow rate:

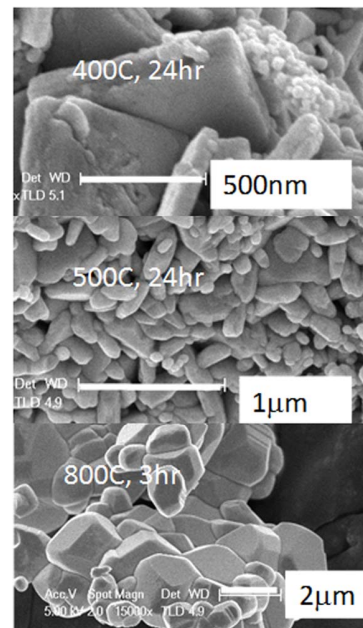


Figure 1. Microstructure of WO₃ derived from thermal decomposition of tungstic acid. The very fine-grained micro-structures promote very high gas sensitivity

$$P_{O_3} = \frac{1}{\alpha} \Delta\sigma + \frac{b}{\alpha^2} (\Delta\sigma)^2 + O(\Delta\sigma)^3 \quad (2).$$

Hence the ozone partial pressure can be estimated from the observed conductance difference with a simple quadratic, as previously noted[17]. The solution to the drift problem requires a continual measurement of the zero, which can be implemented, as described below, by implementing gas flow-rate modulation, taking advantage of the extreme reactivity of ozone to modulate the concentration of ozone in the vicinity of the sensor. This solution, together with attention to detail to avoid decomposition of ozone in the sampling system, has been the most important factor in the successful implementation of a stable and accurate atmosphere measurement instrument.

Previous literature reports have noted the high sensitivity of WO_3 elements to NO_2 [e.g.11,14]. However, the lower limit of concentration explored in these reports has been >100ppb. Measurement at low-ppb concentrations has not previously been described. We have discovered in fact that almost arbitrarily large sensitivity can be achieved by manipulation of the microstructure to yield an extremely porous device with a very large internal surface area. In view of the literature, this is not surprising. The key development has been that of a simple system to scrub ozone from the gas stream presented to the device.

II. INSTRUMENT DESIGN AND OPERATION

A. Sensor fabrication

The sensor substrate was a 2 x 2 x 0.250 mm alumina tile with a meandering platinum heater track printed on one side, and inter-digitated gold electrodes printed on the other. A thin (90 μm approx) layer of WO_3 was screen printed over the gold electrodes, creating the sensing element. The sensor was held inside a sealed plastic housing with an inlet and outlet nozzle to allow sample gas to be drawn over the sensor at a controlled rate. Precise control of the operating temperature was essential to instrument precision and stability, simply because the oxide conductivity varies strongly with temperature : the activation energy is about 0.5eV. The method employed for temperature control incorporated the platinum heater track into a Wheatstone bridge circuit whereby the out-of-balance signal across the two arms of the bridge was used to regulate the current through the sensor heater, thus controlling the heater temperature by keeping its resistance constant[17]. The sensor resistance was determined with a simple DC measurement, with the potential difference across the sensor controlled at 0.1V. Higher potential differences across the sensor caused an excessive resistance drift. The sensor housing was a cylindrical plastic enclosure with 4 bonding posts in the base from which the sensor was suspended freely in the air by fine platinum connecting wires.

B. Ozone

Because the oxide conductance response to ozone is so large, the sensor for this gas was fabricated with a relatively coarse microstructure, which also served to minimize the signal in response to NO_2 . The two key problems to resolve were to overcome the limitations imposed by the very slow

ozone response, and to find a way to determine the zero ozone signal, so that eq.2 could be applied and the zero drift compensated. The means adopted to resolve these issues have been crucial to the development of a successful operating procedure. The first problem, that of the slow response, was solved by the use of a temperature stepping regime[16]. At sufficiently high temperature, the ozone signal becomes essentially zero. The idea is that the surface comes rapidly to equilibrium with oxygen in the air, establishing a surface oxygen vacancy concentration that is repeatable, being dependent only on the temperature and the oxygen partial pressure. Thus a step to a sufficiently high temperature establishes a repeatable initial condition for a subsequent step back to a lower temperature for the measurement. The theory[15,16] shows a clear and simple relationship between the ozone concentration and the initial rate of change of conductance, inferred from the conductance measured a fixed (short) time after the step. The choice of temperature and time is a tradeoff between signal and cycle time. The upper temperature is also limited by the stability of WO_3 , which will sublime at a significant rate at high temperature, especially in the presence of a high vapour pressure of water. This sublimation changes the microstructure of the sensing layer and hence the sensitivity. Loss of WO_3 from the sensor would ultimately lead to a loss of electrical continuity in the sensing layer and hence failure of the device.

The solution to the second problem, that of continually determining the zero ozone conductance, came from measurement of the dependence of the signal on the air flow rate over the sensor. The signal was independent of the air flow rate if this was sufficiently large but fell to zero if the air

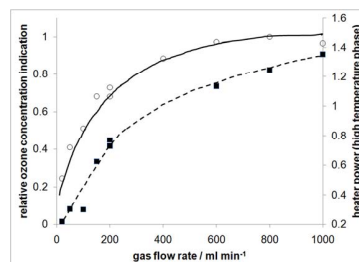


Figure 2. Effect of gas flow rate on module indication and heater power. (O)

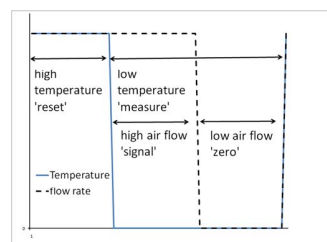


Figure 3. Ozone instrument control cycle – sensor temperature and air flow rate

flow rate was decreased[9]: fig.2. The explanation is that ozone is rapidly decomposed on plastic surfaces, especially if these are warm. Ozone is also rapidly decomposed in air if the temperature is sufficiently high.

There is a thermal boundary layer just outside the sensing layer, and the power dissipated by the sensor warms the plastic housing. Both effects mean that the sensor signal is strongly dependent on the air flow rate over the sensor. Thus, a periodic zero measurement can be obtained by dropping the flow rate across the sensor to zero. Implementing a periodic zero flow condition, during which the sensor temperature was cycled between the high and low temperature states, provided a

continual measurement of the sensor baseline resistance: fig.3. In the final implementation in the instrument, the high and low sensor temperatures were 600 and 520°C respectively, which gave a good compromise of stability of micro-structure of the sensing layer, sensitivity, and required cycle time. An entire measurement cycle lasted one minute and comprised a zero flow condition at high and low temperatures and a high- flow condition at high and low temperatures. The low temperature conductance difference between high and low air flow states, measured a fixed time after the temperature step, followed eq 2. Cycling of the air flow rate was obtained by drawing air across the base of the sensor housing, which had a hole drilled through it, centred on the sensor and with diameter just

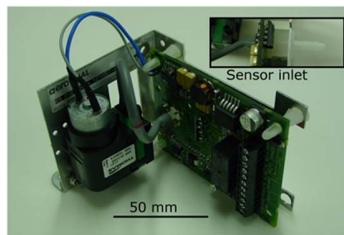


Figure 4. Sensor and instrument module. This unit controls the sensor operation, measures the sensor conductivity, applies the calibration and gives a digital output of gas concentration

greater than the sensor diagonal. The entry face of the sensor housing was fitted with a tapered nozzle that directed the air flow onto the sensor. Because ozone is such a reactive gas, readily decomposed on surfaces, particularly if traces of organic matter are present, it is well-known to take care in the choice of materials for housings and pipework, and also

C. Nitrogen dioxide

Obtaining a good signal for NO₂ in the concentration range appropriate for urban atmosphere measurement (0-200ppb) required fabrication of a device with a very fine-grained microstructure and very large internal porosity. It was operated at constant temperature. The signal increased with decreasing operating temperature, but also became slower to respond to changes in gas concentration, so the design compromise weighted response time against signal magnitude. This microstructure of course gave an extremely large signal in response to traces of ozone. Hence, the key problem to solve was to achieve a robust, reliable and extremely efficient method for

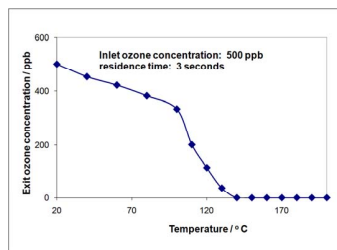


Figure 5. Thermal decomposition of ozone in a heated stainless steel tube

scavenging ppb-levels of ozone from the gas stream without perturbing the NO₂ concentration, either by oxidation to NO or by reduction of this gas, which is generally at significantly higher concentration. Because ozone decomposes extremely rapidly at elevated temperature, both by gas-phase reaction and by catalysed decomposition on metal surfaces, we have

used a simple thermal scrubber. Fig.5 illustrates the decomposition of trace ozone in air passed through a heated stainless steel tube. With a residence time of 3s, at tube temperatures above 180°C ozone was completely absent from the exit gas.

III. RESULTS

A. Ozone

Fig.5 shows a 24hr comparison of ozone value derived from the semiconductor instrument with that from a spectrometric analyser, with measurements made every minute on air sampled directly from the atmosphere. Ozone was injected periodically into the gas sampling system in order to extend the concentration range explored. There was a slope and offset error of the initial calibration of the semiconductor instrument, understandable because of the need to fit a non-linear calibration function using a small number of calibration points (just two ozone concentrations within the atmospheric range – 50 and 100ppb). Following eq 2 and considering only the leading error term, that due to estimation of the parameter, *a*, the estimate of the resulting error in the indicated ozone concentration would be:

$$\delta P_{O_3} \approx \frac{\partial P}{\partial a} \delta a \approx -\frac{\delta a}{a} P_{O_3} \quad (3)$$

The expected linear error dependence is shown in fig.6, and can be used to refine the instrument calibration. The refined calibration tracks exactly the ozone concentration indicated by the reference analyser: fig.7. A long-term assessment of the performance of several instruments operating in the atmosphere alongside a spectrometric reference instrument showed that the semiconductor-based instruments delivered results accurate to better than ±10ppb over periods of months, without recalibration: fig.8. Thus, errors usually associated with semiconductor-based devices, particularly the effects of variation of water vapour pressure or

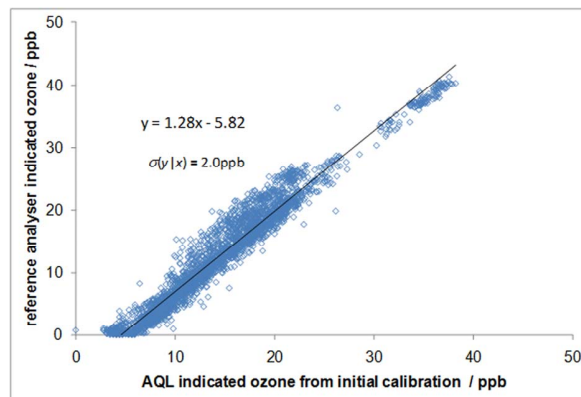


Figure 6. Correlation of semiconductor instrument and reference analyser, operating in the atmosphere for 24hr. Measurements made concurrently on the two instruments, every minute

ambient temperature, have been effectively nullified by the measurement protocol and sensor control that we have applied. One instrument of the ten studied showed a drift. We demonstrated that failures of this sort could be detected early using other measurements available from the instrument.

There are three potentially important causes of errors in

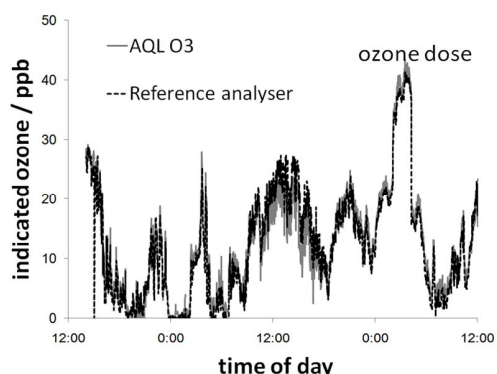


Figure 7. Semiconductor-based ozone instrument tracking spectrometric analyser: measurements made concurrently every minute for 48hr. Ozone was periodically dosed into the air sampling system to increase the measurement range

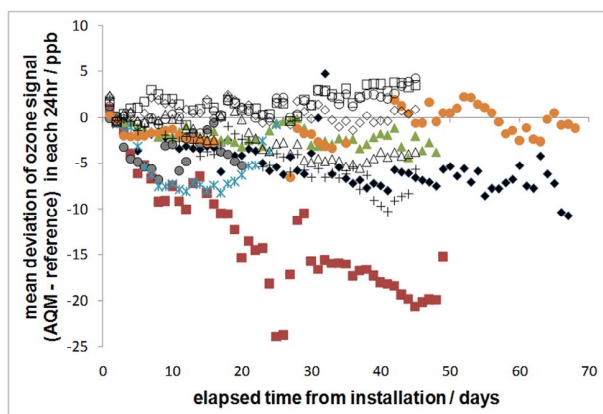


Figure 8. Long-term performance of semiconductor-based ozone instruments (each symbol a different instrument): mean deviation over 24hr from the reference analyser indication of measurements made every minute. Ozone was dosed into the air sampling system for 2 hr every day to increase the measurement range (see fig. 7).

the ozone indication: pump failure or other causes of diminution of air flow rate; dirt accumulation on the filters (dirt is a potent decomposition catalyst for ozone); and sensor microstructure change caused by WO_3 sublimation in a humid atmosphere. The first of these was easily detected by measurement of the heater power required to maintain the control temperature. The heater functioned as a hot-wire anemometer and the power consumption was a reliable indicator of the air flow rate:fig.2. The second failure mode, dirt, was detected by inspection or by loss of air flow. The third failure mode, microstructure change, could be detected by tracking the zero-ozone (low-flow) conductance: this conductance decreased rapidly if WO_3 sublimation occurs and so its variation could be used to indicate the need for sensor replacement. The instance of instrument drift shown in fig.8 was due to this cause and could indeed be tracked by observation of the low-flow conductance. The semiconductor-based instrument thus has the great advantage that known failure modes can be detected remotely, and reliable operation thereby confirmed.

B. Nitrogen dioxide

Fig. 9 shows that the NO_2 measurement system effectively rejected interference from NO and ozone. It also illustrates that the sensor resistance response to change of NO_2 concentration at the level required for atmosphere monitoring was more than adequate, but that the response transient was slow. This is a current limitation of the device. Fig.10 shows that the sensor resistance varied linearly with atmospheric NO_2 concentration. The standard error of estimate was ± 2 ppb: the main cause of deviations between reference (chemiluminescence) and semiconductor sensor instruments was the slow sensor response, which meant that rapid changes in the atmosphere were not contemporaneously tracked by the two instruments. Fig.11 shows a long time series comparing

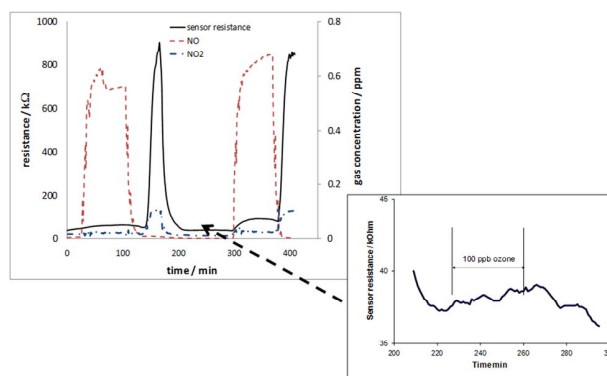


Figure 9. NO_2 module performance: rejection of NO and O_3 interference. The apparent NO signal in the second pulse was due to traces of NO_2 , as shown by the analyser signal.

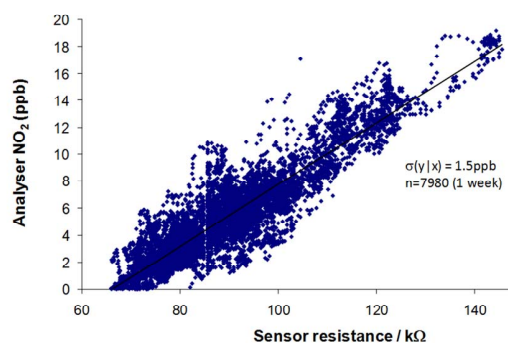


Figure 10. Correlation of sensor resistance with NO_2 concentration, measured in the atmosphere over 1 week. The reference measurement is the hourly averaged NO_2 determined by a chemiluminescence instrument

sensor instrument with analyser. Although the sensor instrument did not capture the rapid (few minute)-scale fluctuations in NO_2 concentration, it did reliably track the 1-hr running mean of the analyser signal. The sensor instrument tracked the analyser-indicated daily mean to better than 2ppb over the entire period.

The time series showing the two types of instrument tracking one another highlights two circumstances in which the measurements diverged. The first occurred occasionally during the morning rush-hour, when for a short time the semiconductor instrument could over-read in comparison with the chemiluminescence analyser. This circumstance

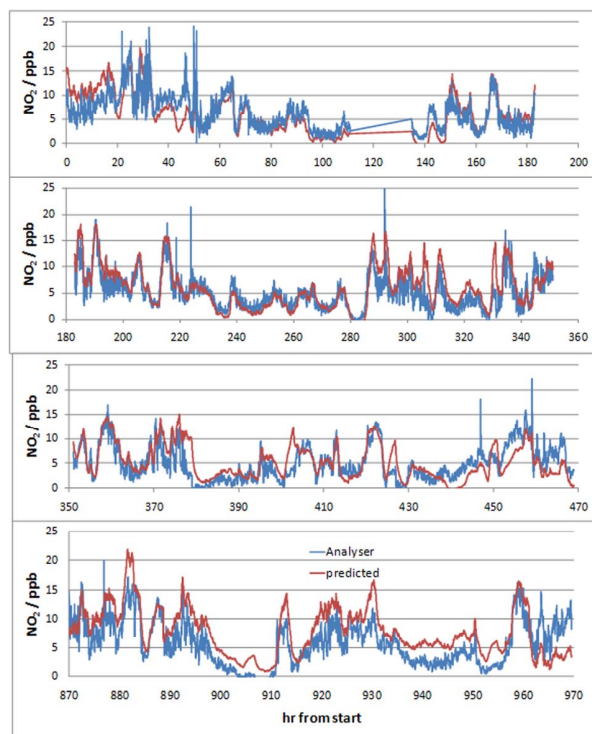


Figure 11. 1000hr-time series comparing semiconductor instrument and chemiluminescence NO₂ analyser, operating in the atmosphere. Measurements were made simultaneously on the two instruments every minute.

corresponds to a ‘non-equilibrium’ atmosphere, where the NO concentration is high and when ozone is being transported down into the urban boundary layer from higher in the troposphere. In this case, ozone can oxidize NO in the scrubber. The effect was confirmed by ozone injection into the sampling line when the NO concentration in the atmosphere was high. The second case was also associated with high NO in the atmosphere, only in this case the semiconductor instrument under-reported in comparison with the chemiluminescence analyser. Given that the sensor develops no signal to NO concentrations that are much higher (fig.8), we attributed this discrepancy to the chemiluminescence analyser. This instrument determined NO₂ by difference, between the NO signal directly measured and that measured after all the NO₂ has been catalytically reduced to NO. Our hypothesis is that, if NO is high and NO₂ low then there exists the classic case of a small error in the individual values translating to a more significant error in the difference. The observed effect would appear if some rather small amount of NO were converted to NO₂ either in the sampling lines or within the instrument other than in the chemiluminescence reaction chamber.

IV. CONCLUSION

Instruments based on WO₃ semiconductor sensors have been successfully developed for measurement of ozone and nitrogen dioxide in the atmosphere. These instruments tracked reference analysers for periods of months, without recalibration. They are eminently suitable for deployment as

low-cost devices to extend the spatial coverage of air quality monitoring networks.

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