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Sensitivity and Operating Characteristics of the Graseby Ionics Otto Fuel Monitor (OFM MK2)

P.A. Lancaster, D.R. Leslie
and V. Tantaro

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P.A. Lancaster, D.R. Leslie and V. Tantaro

Ship Structures and Materials Division
Aeronautical and Maritime Research Laboratory

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ABSTRACT

The response of the Graseby Ionics Otto Fuel Monitor (OFM MK2) to propylene glycol dinitrate (PGDN), the volatile, major component of Otto Fuel has been determined in a range of environmental conditions. The OFM was found capable of detecting PGDN vapour, when it is present in air, at the concentration set by the National Occupational Health and Safety Commission as the Worksafe Australia occupational exposure standard. Characteristics of the OFM that should be considered when using the monitor are discussed.

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Sensitivity and Operating Characteristics of the Graseby Ionics Otto Fuel Monitor (OFM MK2)

Executive Summary

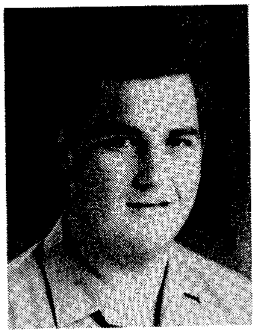
Otto Fuel is used by the Royal Australian Navy as a propellant for torpedoes. This fuel contains a volatile component, propylene glycol dinitrate (PGDN) that is known to cause undesirable physiological responses in exposed personnel, and the National Occupational Health and Safety Commission (NOHSC) has set a workplace exposure standard for contamination of air with PGDN vapour. Thus, there is a requirement to monitor the air in workplaces where there is the potential for personnel to be exposed to this vapour.

In this report, the suitability of the Graseby Ionics Otto Fuel Monitor (OFM Mk 2) as a workplace monitor for PGDN vapour was investigated. We have found that the OFM provides a sensitive, "real time" response to PGDN vapour, although extremes of temperature and humidity degrade the sensitivity of the monitor. Nevertheless, the OFM will normally detect concentrations below the NOHSC exposure standard. The OFM operates on the same principles as the Graseby CAM that is in service with the ADF as a detector and monitor for chemical warfare agent vapours. This work directly supports studies for development of procedures to maximize the field detection capability provided by CAM.

Our evaluation results in the following recommendations for the use of the OFM by ADF personnel: operators must be aware of the effect of temperature and humidity on the sensitivity of the monitor, they must be mindful of the interfering vapours that may provide false positive responses and they must adhere to operating guidelines that have been developed to avoid contamination of the monitor.

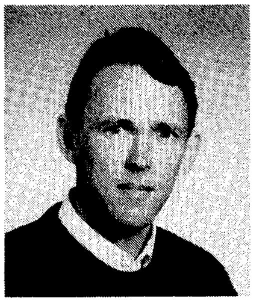
Used properly, the OFM provides the capability to alert personnel when the airborne concentration of PGDN in the workplace approaches, or exceeds the NOHSC exposure standards.

Authors



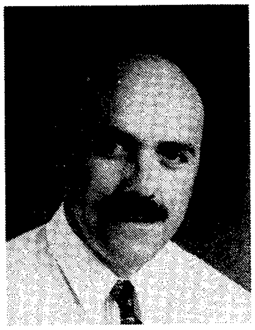
P.A. Lancaster
Ship Structures and Materials Division

Paul Lancaster, BAppSci (App Chem) is a Professional Officer at AMRL-Maribrynong. Before joining DSTO in 1994, he worked at Latrobe University School of Agriculture investigating the fertility status of crop and pasture soils. His work in DSTO includes the development of procedures for using detection and monitoring equipment for chemical warfare agents and other hazardous vapours.



D.R. Leslie
Ship Structures and Materials Division

D. Ralph Leslie, PhD, BAppSci (App Chem) is a Senior Research Scientist at AMRL-Maribyrnong. His early research experience was obtained in the field of physical organic chemistry, using nuclear magnetic resonance spectroscopy as a major investigative tool. Since joining DSTO in 1986 he has undertaken research into chemical methods for the destruction of chemical warfare agents. He is currently investigating the detection of chemical vapours by physical methods.



V. Tantaro
Ship Structures and Materials Division

Vince Tantaro has worked in the NBC area for 25 years during which he has provided technical assistance and advice in the areas of Respiratory Protection, Decontamination, Detection and analysis of Chemical warfare agents, Nuclear Effects, Field trials and training exercises involving the Australian Defence Forces.

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1. Introduction

Otto Fuel is used by the Royal Australian Navy as a propellant for torpedoes. Inhalation of the vapour of the volatile component of Otto Fuel is known to cause a range of undesirable physiological responses in exposed personnel[i]. There is, therefore, a requirement to monitor the air in the vicinity of operations where there is potential for personnel to be exposed to this vapour. The Graseby Ionics Otto Fuel Monitor (OFM) provides the capability for "real-time" monitoring of Otto Fuel vapour. The reported sensitivity of the OFM is sufficient to detect the vapour in air at concentrations well below the limits determined appropriate for occupational exposure[ii].

The OFM utilises the same technology as the Graseby Chemical Agent Monitor (CAM). CAM is known to exhibit a variability in its response to chemical vapours that depends upon temperature of the instrument and also the temperature and humidity of the air to be sampled[iii]. A similar variation in the sensitivity of the OFM with changes in its operating environment could result in the OFM failing to detect Otto Fuel vapour at concentrations that would require action to limit exposure of personnel[ii].

In this report the response of the OFM to Otto Fuel vapour over a range of temperature and humidity is investigated. The procedures applied or developed for generation of air streams containing known concentrations of Otto Fuel and water vapours are described, along with the analytical procedures used to determine these concentrations.

1.1 Otto Fuel II

Otto Fuel II is a bright red-orange liquid used as the propellant that has fuelled most torpedoes since the 1960s. It is regarded as safe for this purpose as it is non-explosive and is stable at temperatures up to 150°C. Propylene glycol dinitrate (PGDN), the major component of Otto Fuel (Table 1) has been found to be physiologically active and is considered a health hazard when present at relatively low concentrations in air.[iv] The dinitrate ester of propylene glycol, PGDN exerts many of the physiological effects of nitrate esters including inducing headaches, dizziness, nausea, dilation of blood vessels, decrease in blood pressure and laboured breathing. Sudden deaths due to circulatory failure have been reported in humans exposed to mixtures of the nitrate esters, nitroglycerine and ethylene glycol dinitrate (EGDN)[v]. Commercial Otto Fuel II also contains di-*n*-butylsebacate as a desensitiser, and 2-nitrodiphenylamine (2NDPA) as a stabiliser. 2NDPA gives Otto Fuel II its colour as pure PGDN is colourless when freshly prepared[vi].

Table 1: Chemical composition and specifications for Otto Fuel II.[a]

Component	% by weight	
	min	max
Propylene glycol dinitrate (PGDN)	75.8	76.2
di-n-Butyl sebacate	22.5	22.5
2-Nitrodiphenylamine	1.4	1.6

[a] From reference [iv]

1.1.1 Occupational Exposure Levels

For Australian workers, the National Occupational Health and Safety Commission (NOHSC) set exposure standards for airborne concentrations of chemicals which, according to current knowledge, should neither impair the health of nor cause undue discomfort for nearly all workers. The NOHSC Worksafe Australia Standard for exposure to PGDN, calculated as a time weighted average (TWA), is 0.05 ppm[vii]. This is the same value as that set by the American Conference of Governmental and Industrial Hygienists (ACGIH)[viii]. The TWA for an airborne contaminant is the maximum allowable average airborne concentration calculated over an eight-hour working day, for a five-day working week to which a worker should be exposed. For many airborne contaminants exposures to concentrations above the TWA standard are permitted provided they are compensated for by equivalent excursions below the standard during the working day.[vii] A Short Term Exposure Limit (STEL), defined as the concentration averaged over a period of 15 minutes, is set for exposure to concentrations above the TWA value. Workers should not be exposed at the STEL for longer than 15 minutes and for no more than four such periods per working day[vii]. While NOHSC does not list a STEL value for PGDN, values from 0.3 ppm to 0.06 ppm have been set in some European countries (Appendix A).

The various levels set for occupational exposure are consistent with findings of a study on the effects of human inhalation of PGDN vapours by Stewart et al[i]. In their study human volunteers were exposed to varying concentrations of PGDN vapour for periods up to eight hours. Subjects consistently reported headaches if exposed to concentrations of PGDN greater than 0.2 ppm for more than 2 hours. A loss of coordination was observed above 0.5 ppm PGDN and an eye irritation was noted at 1.5 ppm. No subjects reported any headaches at levels below 0.1 ppm.

The occupational exposure levels define the sensitivity required of a monitor to measure PGDN vapour in a work environment. The minimum capability needed is to provide an alert if the concentration of PGDN exceeds the TWA level of 0.05 ppm. The ability to detect lower concentrations would allow monitoring of the work-place to determine trends in PGDN vapour concentration and provide a warning if these were approaching the TWA. Similarly, the ability to monitor levels in excess of the TWA would allow the effectiveness of procedures to reduce the concentration of PGDN to be assessed.

1.2 Otto Fuel Monitor MK2

The Otto Fuel Monitor (OFM) monitor is a single (negative ion) mode Ion Mobility Spectrometer (IMS) manufactured by Graseby Ionics that closely resembles the Chemical Agent Monitor (CAM, CAM2) made by the same company. It is a hand held portable monitor weighing 1.5 kg (Figure 1a) and powered by a 6 V lithium sulphate battery having an operational life of 6 hours.

Ion Mobility Spectrometry characterises ions in the gas phase on the basis of their mobility at atmospheric pressure in an applied electric field. In this technique ions are accelerated in the electric field and are also continuously colliding with neutral molecules as they traverse the region of the field. The sum of effect of these processes results in a constant ion velocity in the electric field region. It is the ratio of this ion velocity to the magnitude of the applied electric field that is called ion mobility[ix].

1.2.1 Operating Principles of the OFM

Air being monitored is drawn into the OFM through an inlet nozzle by a pump that also creates a re-circulating air flow through the drift tube assembly (Figure 1b). The path to the drift tube assembly (DTA) is blocked by a heated membrane which selectively permits chemical vapours to permeate through it but at the same time acts as a seal between the inside of the DTA and the outside air. Within the ionisation region of the DTA, beta rays from a weak radioactive source, ^{63}Ni (nominally 370 mbq) randomly collide with all the air molecules, knocking off secondary electrons of various energies. These secondary electrons continue colliding with air molecules resulting in a continuous loss of energy as they traverse the ionization region of the DTA. As the energy of the electrons approaches the kinetic energy of the air, probability dictates that these thermalised electrons will collide with an oxygen molecule and a stabilising third molecular body forming primary O_2^- ions. The primary ions begin a series of ion-molecule reactions with trace amounts of H_2O and CO_2 in the drift gas producing secondary ion clusters, $CO_2.O_2^-$; $(H_2O)_nO_2^-$, called reactant ions. In the reaction region, these ions undergo further ion-molecule reactions (eg O_2^- transfer) with neutral organic compounds that have permeated through the membrane producing a stable product ion.

$$M + O_2^- \rightarrow (M)\,O_2^-$$

Movement of the stable product ions and reactant ions along the drift tube under the influence of the applied electric field is prevented by a grid which superimposes a strong electrical field perpendicular to that of the drift tube. Periodically, this " electrical shutter" is pulsed open to permit a packet of ions to enter the drift region of the DTA. Ions then travel under the influence of the electric field to a collector electrode. Different ions have their own characteristic velocities and arrive at the detector at different times. The time taken for a particular ion to travel to the collector is called its *drift time*. A current is therefore induced at the collector electrode that is time dependent. The resultant ion mobility spectrum (Figure 2), a plot of ion-current at the collector (quantitative measure of the analyte) against arrival times of the ion-clusters as they reach the collector (qualitative information of the analyte), may be processed to detect the presence of ion-clusters formed from particular organic vapours.

The OFM monitors the ion mobility spectra for the presence of ions that have the characteristic mobility of the ion clusters formed in the presence of PGDN. These are identified from their drift time relative to that of the reactant ion peak (RIP)[ii]. The data is processed to provide an indication of the concentration of PGDN present in the sampled air which is then presented to the operator on an 8-segment LCD display on the top of the unit. The concentration of PGDN is indicated by the number of segments showing on the LCD display.

1.2.2 Operation of the OFM

To the operator, the OFM is a simple piece of equipment with only one control, the on/off button (Figure 1a). Before switching on, the operator should ensure that the nozzle protective cap is in place. Each time the monitor is switched on a series of self checks are carried out, after which the RIP needs to be identified by the internal processor before the monitor is ready for use. If a RIP cannot be identified then the display of the monitor will display "WAIT" indicating it is not ready for use. If this display does not clear the OFM will require maintenance. Sampling of the air commences immediately the nozzle protective cap is removed.

To confirm that the OFM is functioning correctly the operator should expose it to vapours released by the confidence test sample, methyl salicylate (MS), provided with the instrument. The exposure should not exceed 1 second, and several segments of the LCD display should be activated. If the monitor does not respond to the confidence sample, the procedure should be repeated and if it still fails to respond it will require maintenance. When the display clears after a positive response to the confidence sample, the OFM is ready to monitor for PGDN. When the OFM indicates an 8 bar response it is important not to continue sampling (the OFM should be removed from the source and the nozzle protective cap replaced) as the vapour will adsorb on its inner surfaces, then be slowly released into the sample air stream. In this situation, the OFM will "correctly" indicate the presence of PGDN for an extended period even after removal from the vapour source. Before turning the monitor off, the operator should fit the nozzle protective cap to the instrument and wait until all eight segments of the LCD display are clear.

2. Results and Discussion

2.1 Reproducibility of Ion Mobility Spectra

The effect of a number of parameters on the response of the OFM was investigated in this study. The time necessary to adjust, allow equilibration and measurement of these various parameters, especially the concentration of PGDN vapour in air streams dictated that data on the response of the OFM were collected over several weeks. The stability of the OFM was demonstrated by comparing ion mobility spectra collected under standard conditions at various times.

The area of the RIP (current × time, in arbitrary units) measured with the nozzle cap in place after the OFM had been operating for at least 30 minutes at ambient laboratory temperature (21-24 °C) exhibited little variation over a 6 day period (Table 2).

Table 2: Area of Reactant Ion Peak taken over a 6 day period while sampling clean air under ambient conditions

Day	1	2	3	7	Mean
RIP Peak Area[a]	8190±420	8830±470	8870±450	8930±450	8700±450

[a] Arbitrary units. Errors are standard deviations obtained for measurements taken on each day.

This area is a measure of the number of reactant ions available for reaction with PGDN vapour and is a primary determinant of the sensitivity of an IMS instrument. The stability of the RIP peak area can be attributed to the ^{63}Ni ionisation source used in the OFM.[x] The reproducibility in the response of the OFM to PGDN matches the stability of the RIP. When the OFM was presented to a stream of dry air containing 0.118 ppm PGDN vapour 5 times over a 24 hour period the drift times and peak areas of the PGDN peak (Table 3) varied by less than 5%. These observations provide a reference against which the effect on the response the OFM caused by variations in the concentration of PGDN in air, humidity or other external parameters may be assessed.

Table 3: Analysis of the PGDN peak from the Ion Mobility Spectrometer spectrum obtained while the Otto Fuel Monitor was exposed to an air stream containing 0.118 ppm PGDN at various times over a 24 hour period.

Time Frame (hours)	Drift Time (Ms)	Peak Area[a]	Population
0:00	9099±23	2227±78	12
1:30	9045±25	2332±116	22
3:15	9022±21	2413±122	21
5:00	8983±16	2385±115	22
24:00	9057±16	2355±147	18

[a] Arbitrary units

2.2 OFM Response to PGDN Vapour in Air

OFM ion mobility spectra were recorded with the monitor exposed to air containing concentrations of PGDN vapour in the range 0.0014 to 0.0118 ppm. The area of the peak corresponding to ions containing PGDN in the spectra and the number of segments activated in the monitors LCD are given in Table 4. The manufacturer uses the term "Bars" to indicate the number of LCD segments activated, for the sake of brevity we will use the same term throughout this report. The OFM responses in the Table were obtained from PGDN vapour in air streams containing the ambient level of water vapour (≈40% RH, 21 °C), apart from the last 3 entries for which dry air (<1 ppm water) was used. The manufacturer's literature indicates that the OFM when exposed to air containing 0.01 ppm of PGDN will display 1 Bar, and 0.2 ppm will give a full 8 Bar response. The sensitivity of the OFM used in this study exceeds these values, with responses of 1 and 8 Bars to PGDN concentrations of 0.0081 and 0.0827 ppm respectively. The NOHSC Worksafe Australia TWA exposure standard for PGDN of 0.05 ppm activates 7 Bars of the OFM display.

The area of the "PGDN peak" in the ion mobility spectra is approximately linearly dependent upon the concentration of PGDN over the concentration range investigated (Figure 3). This range was selected as it provides the full range of responses (0-8 Bars) on the OFM display. The linear dependence is consistent with reports that IMS instrumentation have a linear response to analyte concentrations spanning 1-2 orders of magnitude.[x] The linear range in IMS is limited by the finite number of reactant ions available for reaction with the vapour to be detected. Increasing the analyte vapour concentration in the air stream above that which results in transfer of the total charge associated with the RIP to the analyte peak will not increase the area of the analyte peak. Although the 0-8 Bar response of the OFM occurs within the concentration range that provides a linear response in the raw ion mobility spectra, the Bar response is a logarithmic function of concentration (Figure 4).

Table 4: Otto Fuel Monitor response to PGDN vapour.

[PGDN] generated (mg/m^3)	(ppm)	PGDN IMS Peak Area[a]	Otto Fuel Monitor Bar Response
0.010	0.0014	88	none
0.023	0.0034	104	none
0.055	0.0081	123	1
0.072	0.0106	124	2
0.125	0.0184	223	3
0.190	0.0279	273	4
0.218	0.0320	597	5
0.264	0.0389	712	6
0.264	0.0389	675	6
0.311	0.0470	1040	7
0.347	0.0512	1119	7
0.562	0.0827	2128	8
0.598	0.0880	1704	8
0.694	0.1020	2068	8
0.799	0.1180	2850	8

[a] Arbitrary units

2.2.1 Effect of Temperature on OFM Response

Increasing the temperature of the OFM results in decreased sensitivity of the monitor. When sampling an air stream containing 0.032 ppm PGDN, the Bar display decreases from 5 to 0 as the temperature of the monitor is increased from 21.0 °C to 46.5 °C (Table 5a). The diminished response of the OFM does not result from a decrease in the inherent sensitivity of the instrument. While the amplitude and width of the RIP is dramatically altered (Figure 5) the area (a measure of the number of reactant ions available for reaction with PGDN) is largely unaffected by temperature, decreasing by only 20% as the temperature of the monitor is raised from 20.5 to 50.0 °C.

Table 5a: Effect of temperature on the response of the Otto Fuel Monitor to air containing 0.032 ppm PGDN[a].

OFM Temperature (°C)	PGDN Peak Area[b]	OFM Bars Displayed
21.0	497	5
29.5	314	4
35.5	188	3
39.5	108	1⇒2
46.5	20	0

[a] Air stream maintained at 28°C, 40% RH.
[b] Arbitrary units

In contrast, the same temperature change causes an 85% reduction in area of the PGDN peak for a monitor sampling air containing 0.083 ppm PGDN. Thus, the decreased response of the OFM must arise from a change in equilibria between reactant and product (PGDN containing) ions. This may arise from an increase in the concentration of water vapour in the DTA (see below). The air within the DTA is recirculated by a pump through a path that includes molecular sieve to remove water vapour. Increasing temperature of the sieve will reduce its ability to remove water vapour.

Table 5b: Effect of temperature on the response of the Otto Fuel Monitor to air containing 0.083 ppm PGDN[a].

OFM Temperature (°C)	20.5	25.5	30.5	35.5	37.5	41.0	44.5	50.0
PGDN Peak Area[b]	2030	1940	1450	1230	1110	660	440	255

[a] Air stream maintained at 53°C, 34% RH.
[b] Arbitrary units

2.2.2 Effect of Humidity on OFM Response

The response of the OFM to PGDN vapour in air was found to decrease with increasing relative humidity of the air stream (Table 6). This occurs despite the presence of a membrane separating the DTA from the air being sampled. The role of the membrane is to provide a barrier between the DTA and the environment to prevent gross contamination by dust, aerosols or liquids while selectively allowing organic vapours to enter the DTA for analysis. Ideally, the membrane would be permeable to only the vapour of interest (PGDN). Clearly, water vapour also diffuses through the membrane.

Table 6: Effect of water vapour on Otto Fuel Monitor response to air containing 0.056 ppm PGDN[a].

Relative Humidity %	[Water] in Vapour (g m^{-3})	PGDN Peak Area[b]	OFM Bars Displayed
38	15.4	1270	7⇒8
50	20.3	1070	7
63	25.6	850	6
70	28.4	710	5⇒6
80	31.3	215	3
87	35.3	120	2

[a] Air stream temperature maintained at 35°C,
OFM temperature maintained at 21°C.
[b] Arbitrary units

The response of the OFM when exposed to an air stream containing 0.056 ppm PGDN, slightly higher than the NOHSC Worksafe Australia TWA exposure standard of 0.05 ppm, reduces from between 7 and 8 Bars to 2 Bars as the water concentration in the air stream increases from 15.4 to 35.3 g/m^3 (Table 6). The same trend is also observed at a higher concentration of 0.081 ppm PGDN (Figure 6). For both concentrations of PGDN the decrease in the area of the PGDN peak is approximately linear with increasing water concentration.

Qualitatively, this reduced sensitivity can be explained by considering the "equilibrium" distribution of charge that results from the totality of the various ion-molecule reactions to be given by the following approximate relationship, in which Q_T is the total number of ions, ε_I is a relative measure of charge affinity for the neutral vapour and [I] is the concentration of the vapour, I.[X] The products ε_I [I] are therefore representative of the relative concentration or number of ions formed from the various neutral species.

$$Q_T = \varepsilon_A [A] + \varepsilon_B [B] + \varepsilon_C [C] + \varepsilon_D [D] + \ldots\ldots\ldots\ldots$$

All of the vapours present in the air stream contribute to the ion-molecule reactions, including those that form the ions of the RIP. Because Q_T remains constant, an increase in the concentration of one vapour will favour formation of ions from it at the expense of ion formation from other vapours. In practice, the situation is more complicated as changing the concentration of neutral vapours may result in processes other than redistribution of charge between the species present. For example, charged and neutral species form clusters in which the number of constituent molecules is dependent upon the concentration of the various species in the reaction region; in particular, the number of water molecules in an ion cluster will vary with water concentration. Nevertheless, the relative number of ions formed from a particular species in the reaction region will depend upon its concentration relative to all other species in the sampled air.

2.3 OFM General Performance

2.3.1 Response Time and Recovery Time

The response time of the OFM when exposed to PGDN vapour in air and the time required to clear after its removal from the PGDN vapour were typically only a few seconds. For concentrations of PGDN that resulted in 4-5 Bars displayed, the maximum response was obtained within 1-2 seconds and even the 1 Bar response obtained when exposed to low concentrations of PGDN was displayed within 10 seconds.

After removal from PGDN vapour the OFM does not instantaneously clear to a zero Bar display as time is required to purge the airways and to allow purging of vapour adsorbed on associated surfaces. The recommended operating procedure to minimise the "clear-down" time for IMS monitors such as the OFM is to cease sampling by replacing the nozzle protective cap as soon as the maximum 8 Bar response is obtained. Continued exposure can provide no additional information and will result in swamping of the monitor that may cause it to display a positive response for many minutes after its eventual removal from the vapour source. In this study the OFM was allowed to sample air streams containing PGDN vapour for more than two minutes. Even when the maximum number of Bars were displayed by sampling over liquid Otto Fuel the monitor cleared within 20 seconds (Figure 7).

2.3.2 Response to Other Vapours

The "confidence test" sample provided by the manufacturer contains methyl salicylate (MS). The drift time for the ion species formed from MS is very similar to that for PGDN ion clusters (Figure 8), hence the confidence test will produce a response on the OFM Bar display. Clearly, other sources of MS could also produce a positive response by the OFM. Methyl salicylate is widely used in the perfume industry, medicinally in the form of local analgesic or anti-inflammatory ointments and in confectionery.[xi] Ethylene glycol is also identified by the manufacturer as providing a positive OFM response. Ethylene glycol is an anti-freeze agent and could be found in coolants, brake fluids etc. Other vapours previously identified to cause CAM2 to respond in H-Mode operation[xii,xiii] may also provide a source for false positive responses by the OFM.

2.3.3 Other Operating Characteristics

The ability of the OFM to detect PGDN vapour in air relies upon the formation of ions from this vapour by transferral of charge from the RIP. If all of the charge originally associated with the RIP is transferred to a vapour other than PGDN the OFM may not provide a Bar response. This possibility has been recognised by the manufacturer, and the operator is alerted to this condition by activation of a segment of the LC display that shows as a vertical arrangement of three dots (⁝) at the lower right hand edge of the display panel. This display will also be activated if the charge of the RIP is completely transferred to PGDN, but in this case the Bar segments of the display will also be activated. Operators should be aware of the status of the three dot element of the display to avoid false negative responses to PGDN vapour.

The response of the OFM may deteriorate between routine maintenance operations. Within the OFM is a molecular sieve-pack that removes water vapour from the recirculating air stream within the DTA. In time, the capacity of this sieve-pack may become exhausted causing a loss of sensitivity associated with relatively high water vapour concentrations within the DTA. Replacement of the sieve pack is part of the maintenance schedule for the OFM.

3. Conclusion

The Graseby Ionics OFM is ideally suited to the point source detection of PGDN vapours as it is light, compact, battery powered and requires no external equipment for its operation. It provides a rapid response to PGDN vapour at the TWA exposure limit (0.05 ppm), set by the NOHSC, under moderate temperature and humidity conditions. Concentrations of PGDN in air as low as 0.008 ppm provide a positive response on the Bar display. After removal from PGDN vapour the Bar display of the OFM quickly cleared to zero Bars. The stability of the OFM under ambient conditions was found to be very good, with little variation in response found over extended periods. The sensitivity and response characteristics of the OFM allow it to be used as a monitor for PGDN vapour. It can provide a real time response to changing concentrations of PGDN in air provided it is not exposed to gross contamination by PGDN or other chemical vapours. Implementation of correct operational procedures will avoid contamination of the OFM.

Under conditions of high temperatures the response of the OFM to PGDN vapour was significantly reduced. At a temperature of 46.5 °C the OFM showed no response to air containing 0.032 ppm PGDN. This same concentration provides a 5 Bar response at ≈20 °C and 1-2 Bar response at 40 °C. The presence of high concentrations of water vapour in the air being sampled also reduces the sensitivity of the OFM. Nevertheless, the presence of PGDN at the TWA exposure level (0.05 ppm) provides a 2 Bar response with the a water vapour concentration of 35 g/m^3. *Such high levels of water can only be achieved with air temperatures in excess of 33 °C (ie 100% RH at this temperature corresponds to a water vapour concentration of 35 g/m^3).* Operators should be aware of the effect of temperature and humidity on the response of the OFM. The OFM should not be left in an environment where it will heat up (eg, in direct sunlight).

Consideration of the effect of temperature and humidity on the response of the OFM clearly shows that the OFM can not be used to provide a quantitative measure of PGDN vapour. Nevertheless the Bar response can be used as a trend monitor with changing PGDN concentrations resulting in corresponding changes in the Bars displayed on the OFM.

4. Experimental

4.1 PGDN Vapour Generation

Two methods were used to generate controlled concentrations of PGDN vapour in air. A Graseby Ionics Vapour Generator Type GI 10 was used to generate concentrations in the range of 0.118 to 0.045 ppm. In this generator, PGDN vapour released from a permeation (or diffusion) tube housed in a heated, constant temperature source chamber is collected in a constant flow of carrier gas which is then uniformly mixed with a greater flow of diluent gas. The required concentration at the outlet nozzle is established by adjusting the source temperature and gas flows. Our need to generate air streams with lower concentrations of PGDN vapour necessitated the use of another method of vapour generation. A system based on the procedure described by Fortuin,[xiv] and previously shown in this laboratory to produce low, reliable constant vapour concentrations during the evaluation of chemical warfare agent detector systems was used. PGDN vapour is introduced into an air stream at a rate controlled by its diffusion up a vertical cylindrical tube from a pool of liquid in the bottom of the diffusion tube. This rate is controlled by adjusting the temperature of the liquid, the diameter of the diffusion tube and the distance of the liquid level below the top of the tube.

A flow diagram of the "Fortuin" apparatus used to generate air streams with concentrations of PGDN vapour in the range of 0.0014 to 0.0827 ppm is shown in figure 9a. A stream of clean air passes, via a buffering vessel (E), through a needle valve (T) followed by two adsorption towers (A1 and A2) before passing through a rotameter (W1). The air stream then enters a constant temperature box, passes through a heat transfer coil and enters the diffusion tube chamber (J) where it passes over a vertical cylindrical tube containing Otto Fuel. The air stream containing PGDN vapour exits the constant temperature box and enters the first of two dilution chambers (X1) where it is diluted by mixing with an air stream controlled by the combined needle valve and flowmeter, B1. The diluted PGDN vapour enters a fractionating chamber (S) that has a port allowing a portion of the air stream, controlled by a needle valve, to exit the system via the vent Q2 which contains a charcoal trap to prevent release of organic vapours. The remaining portion of the diluted vapour passes through a rotameter (W2) and into the second dilution chamber (X2) where it is diluted again with an air stream controlled by the valve/flowmeter B2.

The vapour then enters a large sampling chamber (N) containing a primary sampling point large enough for the inlet nozzle of the Otto Fuel monitor to be introduced to the sample vapour, and an auxiliary sampling point where the vapour can be sampled for analysis.

Air streams of constant PGDN concentration and varying humidity required for this study were obtained by modifying a "Fortuin" vapour generator used previously in this laboratory in trials of chemical warfare agent detector systems (Figure 9b). This system produces two constant flow air streams that are combined in a mixing chamber before entering the sampling chamber. One stream contains PGDN vapour of constant concentration, the other water vapour of variable concentration. Thus, a stream of clean air passes through a buffering vessel (E) and two absorption towers (A1 and A2) before it is split into three. One stream passes through the flow controller B1 and is conditioned to provide air with a controlled concentration PGDN vapour to the mixing vessel (P). Another air stream, controlled by needle valve T2, is saturated with water vapour by passing it through a series of water towers (a1, a2, and a3) packed with glass wool to maximise contact time in the water. This stream then combines with the third air stream, the flow of which is controlled by the needle valve T1, and the total flow of these two streams is measured by flowmeter B2. By varying the flow of the individual streams a constant flow of air with variable humidity can be supplied to the mixing vessel (P). The percentage relative humidity (%RH) of the air stream leaving the mixing vessel is obtained by measuring the temperatures of a wet bulb thermometer (Z) and a dry bulb thermometer (Y) and converting these readings to %RH using the appropriate tables[xv]. Maintaining a constant flow of humid air allows the %RH of the final outlet stream of air to be adjusted without affecting the concentration of PGDN at the outlet of the mixing chamber.

The "Fortuin diffusion tube" style vapour generators were found to require approximately 1 hour for the PGDN vapour concentration to reach a stable value after the temperature or air flows were adjusted. A similar period was necessary for equilibration of relative humidity when the air streams controlling this property were adjusted in the modified apparatus. The GI-10 vapour generator required up to 12 hours for the equilibration of PGDN concentration in the air stream after the temperature or air flows in the GI-10 were altered. The vapour streams were deemed to have reached their equilibrium concentrations of PGDN and water vapour when the area of the PGDN peak in the ion mobility spectrum produced by the OFM (see below) reached a constant value.

4.2 Quantitation of PGDN Vapour in Air Streams

The concentration of PGDN vapour in air streams produced by the three vapour generators was determined by collecting (trapping) the PGDN contained in a known volume of air, then quantifying this trapped mass by Gas Chromatographic analysis.

4.2.1 Vapour Trapping

After the air streams were allowed time to equilibrate a portion of the air stream at the outlet of the vapour generator was drawn through two glass tubes (connected in series), each containing 100 mg of Porapak adsorbent,[xvi] by a low pressure air pump. A previous study, conducted at these laboratories, had shown Porapak to be effective in trapping PGDN vapour. Flow through the adsorbent tubes was controlled by a critical orifice, chosen to provide a constant air flow through the adsorbent material. The air stream was sampled for an accurately timed period of approximately 50 minutes, after which the tubes were eluted with 2 mL of hexane, desorbing the PGDN into solution for quantitation by gas chromatography (GC). The Porapak tubes proved efficient in trapping the PGDN with no apparent breakthrough of the PGDN vapour into the second of the Porapak tube used in series. A single 2 mL wash of hexane was determined sufficient to desorb all of the adsorbed PGDN from the Porapak tubes.

4.2.2 Gas Chromatography Analysis

Quantitative analysis of PGDN in hexane was achieved using a Varian 3700 gas chromatograph fitted with a constant current pulse modulated ^{63}Ni Electron Capture Detector (ECD). This detector is highly sensitive to electron capturing groups such as nitrate esters but insensitive to amines and hydrocarbons. The experimental gas chromatographic conditions are given in Table 7. Chromatographic data was collected and analysed using a PC running a Delta Junior, *Ver* 4.32 software and interface package. Software analysis provided retention time and integrated area for peaks which were used for quantitative analysis.

Table 7: Gas Chromatographic conditions.

Varian 3700 GC	
1μL sample size 10:1 split injections	
Temperatures:	Injector: 200°C Detector: 220°C Column: 120°C - Isothermal
Column:	SGE 25QC-3-BP5-0.5 column flow = 38 cm/sec
Detector:	Constant current pulse modulated Ni^{63} electron capture detector
Carrier gas:	Ultra high purity nitrogen
Run time:	≈4 min.

An analytical working curve for quantitation of PGDN trapped from air was constructed from the peak areas of PGDN in standards containing 0.02 to 4.0 μg/mL in hexane (Figure 10) which, under the gas chromatographic conditions used, corresponds to 2 to 400 picograms (10^{-12} g) of PGDN presented to the ECD. Standards were prepared from Otto Fuel II shown to contain 69.1% PGDN by ^{1}H nuclear magnetic resonance spectroscopy. The working curve was linear ($R^2 = 0.998$) across the entire concentration range. Standards prepared from Otto Fuel have been shown to be suitable for the construction of calibration curves for gas chromatographic analysis of PGDN[V]. All analyses, including those of the calibration standards were performed in triplicate and the average values taken to minimise experimental errors in quantitation.

4.3 Otto Fuel Monitor Response

The operation of the OFM was monitored by recording the response indicated on its 8 segment LCD display and the raw data ion mobility spectra from which the output to the display was derived. Data were collected under a range of conditions, with the concentration of PGDN in the air stream being sampled, the temperature and relative humidity of the air stream and the temperature of the OFM varying. The procedure for data collection involved equilibration of the OFM in a constant temperature environment for 120 minutes prior to it sampling the air streams. In addition, the OFM was turned on, the self check procedure completed and allowed to operate with the nozzle protective cap in place for at least 30 minutes before data collection. Ion mobility spectra were recorded until there was no significant variation in peak areas between successive spectra, and at this time the LCD response which corresponded to the maximum achieved when sampling the particular air stream was also recorded. Typically, stable ion mobility spectra and maximum LCD readings were obtained within 10 seconds of exposure to the air stream.

4.3.1 Collection of Ion Mobility Spectra from OFM

The OFM has an analogue output of the amplified signal created by arrival of ions at the collector at the end of the drift tube. A timing pulse coincident with the opening of the electrical shutter and introduction of ions into the drift region of the DTA is provided at a separate output. Ion mobility spectra were recorded from these signals by a personal computer using a commercial interface card and software package developed specifically for this purpose (Graseby Analytical ASP processor board and Graseby Ionics WASP, *Ver* 1.35 software).

Ion mobility spectra were recorded by collecting 512 data points sampled at 20 kHz with sampling commencing 2 ms after the timing pulse. Signal averaging was employed to improve the signal to noise, with 32 spectra summed to provide each stored spectrum.

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6. Glossary

OFM	Otto Fuel Monitor
DTA	Drift Tube Assembly
RIP	Reactant Ion Peak
LCD	Liquid Crystal Display
CAM / CAM2	Chemical Agent Monitor / improved model
H-Mode	Chemical Agent Monitor operating in negative ion mode
IMS	Ion Mobility Spectrometry
PGDN	propylene glycol dinitrate
EGDN	ethylene glycol dinitrate
2NDPA	2-nitrodiphenylamine
MS	methyl salicylate
NOHSC	National Occupational Health and Safety Commission
ACGIH	American Conference of Governmental and Industrial Hygienists
TWA	Time Weighted Average
STEL	Short Term Exposure Limit
RH	Relative Humidity
GC	Gas Chromatography
ECD	Electron Capture Detector
PC	Personal Computer
ASP	Advanced Signal Processing
WASP	Waveform Analysis Software Package
ppm	parts per million

Appendix A

NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH RTECS[viii]

87255. 1,2-PROPANEDIOL, DINITRATE
RTECS N^{o}: TY6300000
GAS N^{o}: 6423-43-4
UPDATE: 9301
MW: 166.11
MF: $C_3H_6N_2O_6$

SYNONYMS:
PGDN, PROPYLENE DINITRATE, PROPYLENE GLYCOL 1,2-DINITRATE,
1,2-PROPYLENE GLYCOL DINITRATE, PROPYLENE GLYCOL DINITRATE (ACGIH, OSHA).

SKIN & EYE IRRITATION DATA & REFERENCES:
eye: rabbit; 100 mg ; MILD; TXAPA9 22,128,72; Toxicol Appl Pharmacol

TOXICITY DATA & REFERENCES:
inhalation;
human; TCLo:200 ppb/6H; BEHAVIOURAL (Headache); TXAPA9 30,377,74; Toxicol Appl Pharmacol
TCLo1500 ppb/5M; SENSE ORGANS & SPECIAL SENSES(Conjunctiva irritation);
TXAPA9 30,377,74; Toxical Appl Pharmacol
intraperitoneal;
guinea pig; LD50: 402 mg/kg; NUTRITIONAL & GROSS METABOLIC (Weight loss or decreasing weight gain); AIHAAP 34,526,73; Am Ind Hyg Assoc J
mouse; LD50: 1047 mg/kg; LUNGS, THORAX OR RESPIRATION (Respiratory depression); BLOOD (Methemoglobinemia-Carboxhemoglobinemia); VASCULAR (BP Lowering not characterised in autonomic section); AIHAAP 34,526,73; Am Ind Hyg Assoc.
rat; LD50: 479 mg/kg; LUNGS, THORAX OR RESPIRATION (Respiratory depression); BLOOD (Methemoglobinemia-Carboxhemoglobinemia); VASCULAR (BP Lowering not characterised in autonomic section); AIHAAP 34,526,73; Am Ind Hyg Assoc.
oral;
rat: LD50: 250 mr/kg; LUNGS, THORAX OR RESPIRATION (Respiratory depression); BLOOD (Methemoglobinemia-Carboxhemoglobinemia); VASCULAR (BP Lowering not characterised in autonomic section); AIHAAP 34,526,73; Am Ind Hyg Assoc.

LITERATURE REVIEW:
ACGIH THRESHOLD LIMIT VALUE TWA 0.05 ppm (skin); 85INA8 5,502,86; Doc Threshold Limit Values

STANDARDS & REGULATIONS:

OSHA PEL FINAL: 8H TWA 0.05 ppm(0.3 mg/m^3); FEREAC 54,2923,89; Fed Regist

OEL-AUSTRALIA;	TWA 0.05ppm(0.3 mg/m^3);Skin	JANUARY 1993
OEL-BELGIUM;	TWA 0.5ppm(0.35 mg/m^3);Skin	JANUARY 1993
OEL-DENMARK;	TWA 0.02ppm(0.2 mg/m^3);Skin	JANUARY 1993
OEL-FINLAND;	TWA 0.02ppm(0.2 mg/m^3);	
	STEL 0.06 ppm (0.06 mg/m^3)Skin	JANUARY 1993
OEL-FRANCE;	TWA 0.05ppm(0.3 mg/m^3);Skin	JANUARY 1993
OEL-GERMANY;	TWA 0.05ppm(0.3 mg/m^3);Skin	JANUARY 1993
OEL-SWEDEN;	TWA 0.1ppm(0.7 mg/m^3);	
	STEL 0.3 ppm (2 mg/m^3);Skin	JANUARY 1993
OEL-SWITZERLAND;	TWA 0.05ppm(0.3 mg/m^3);Skin	JANUARY 1993
OEL-THE NETHERLANDS;	TWA 0.05ppm(0.3 mg/m^3);Skin	JANUARY 1993
OEL-UNITED KINGDOM;	TWA 0.2ppm(1.2 mg/m^3);	
	STEL 0.2ppm(1.2 mg/m^3);Skin	JANUARY 1993

OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA, NEW ZEALAND, SINGAPORE, VIETNAM

NIOSH STANDARD DEVELOPMENT & SURVEILLANCE DATA:
  NIOSH REL TO PROPYLENE GLYCOL DINITRATE-air:10H TWA 0.05 ppm (Sk);
  NIOSH DHHS #92-100,92 Natl Inst Occup Saf Health
STATUS:
  EPA TSCA CHEMICAL INVENTORY, JUNE 1990
  EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, JANUARY 1993.

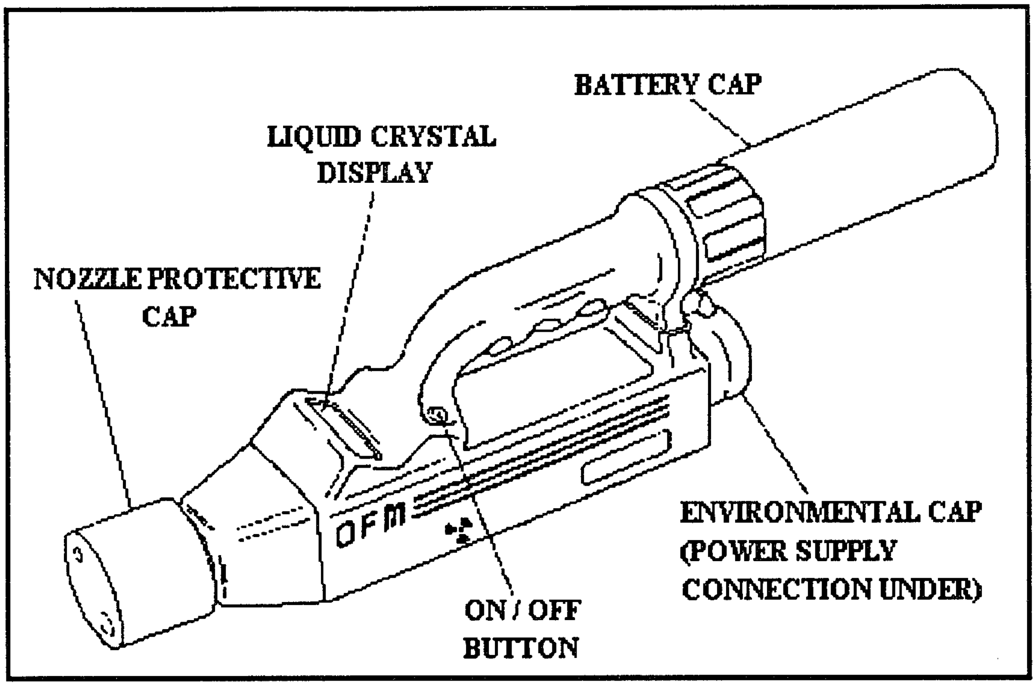

Figure 1a. Otto Fuel Monitor Components[ii]

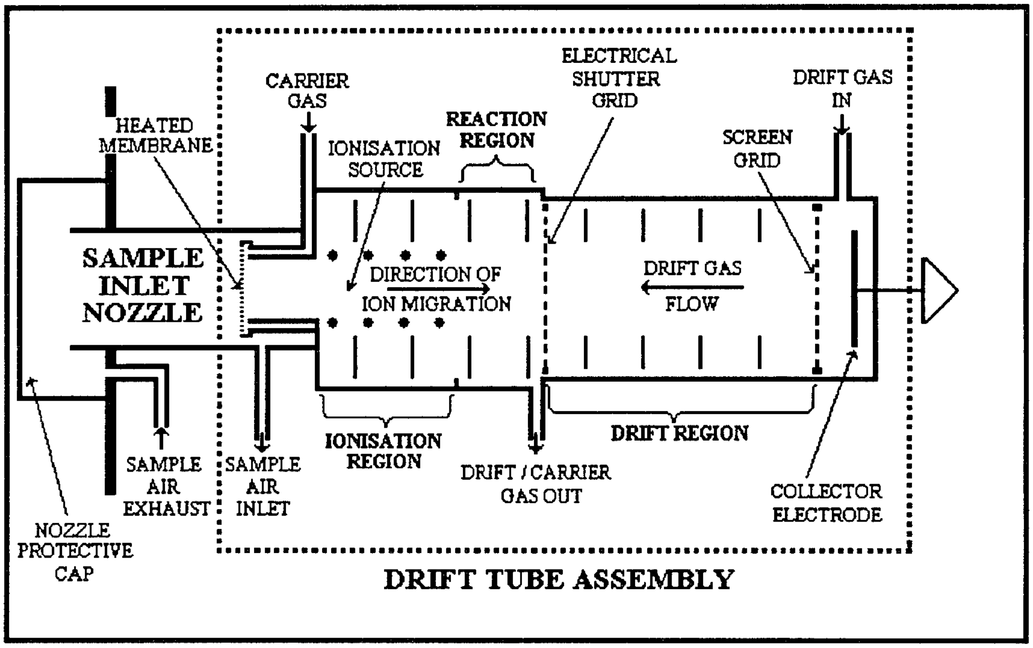

Figure 1b. Otto Fuel Monitor - Block Schematic Diagram

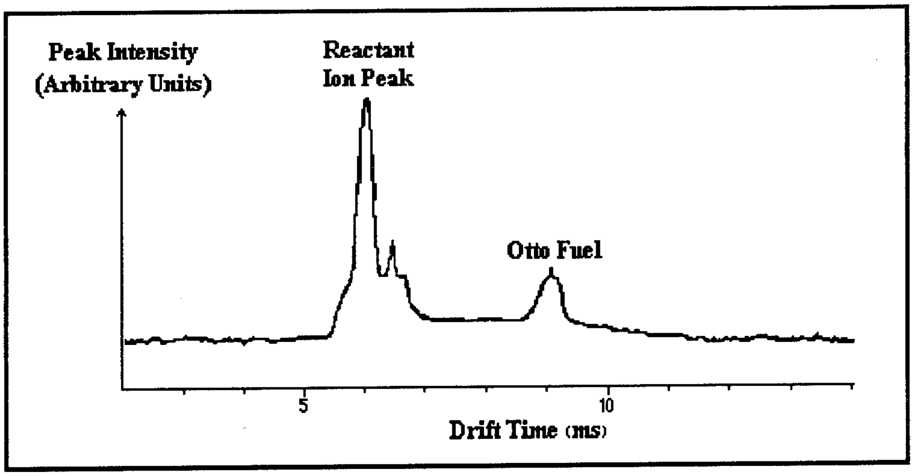

Figure 2. Ion mobility spectrum of Otto Fuel Monitor sampling Otto Fuel.

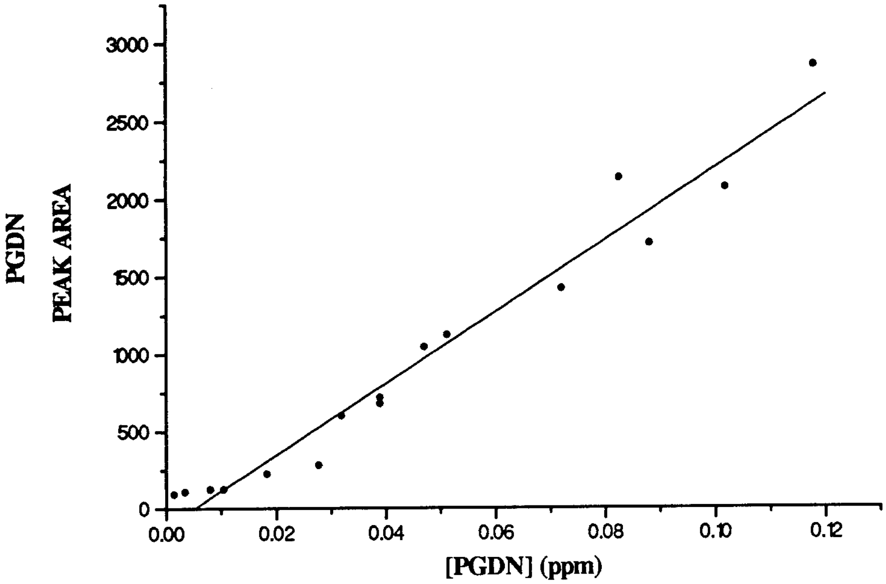

Figure 3. Calibration of Otto Fuel Monitor. Vapour concentration (ppm PGDN) verses Otto Fuel Monitor output (PGDN Peak Area). Linear correlation coefficient of least squares fit = 0.977

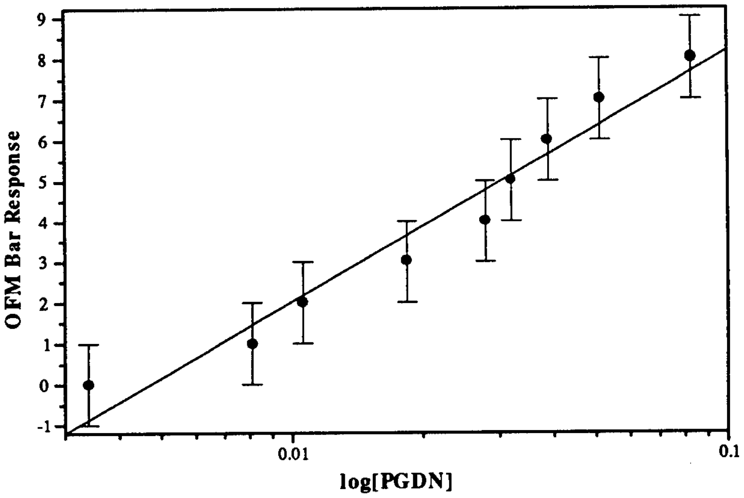

Figure 4. Calibration of Otto Fuel Monitor. Otto Fuel Monitor bar response as a logarithmic function of PGDN vapour concentration. Linear correlation coefficient of least squares fit = 0.978

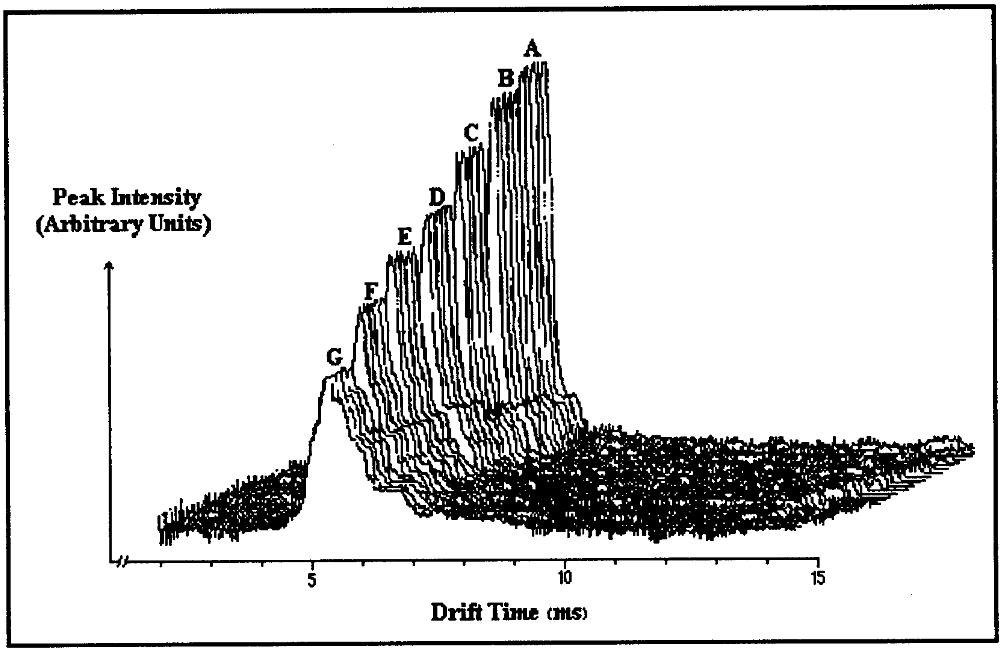

Figure 5. Reactant Ion Peak for Otto Fuel Monitor sampling clean air while the Otto Fuel Monitor temperature (°C) was held at:

A. 20.5	*B. 25.5*	*C. 30.5*	*D. 35.5*
E. 37.5	*F. 41.0*	*G. 44.5 .*	

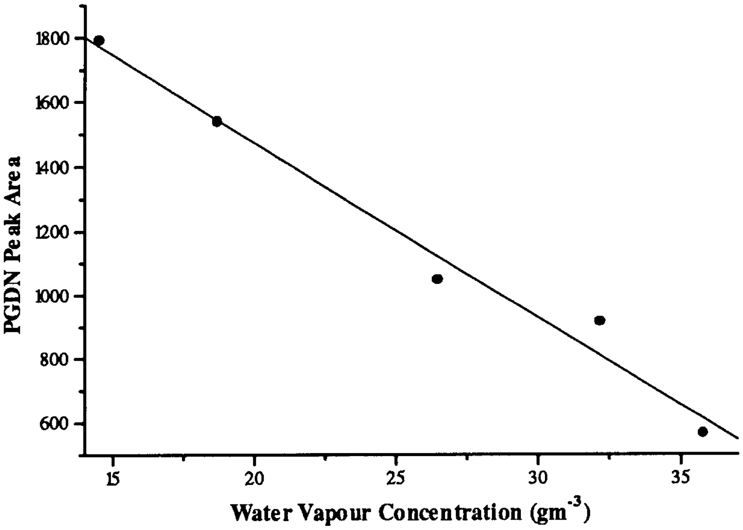

Figure 6. Effect of water vapour on the response of the Otto Fuel Monitor sampling 0.081 ppm PGDN vapour. Linear correlation coefficient of least squares fit = 0.990

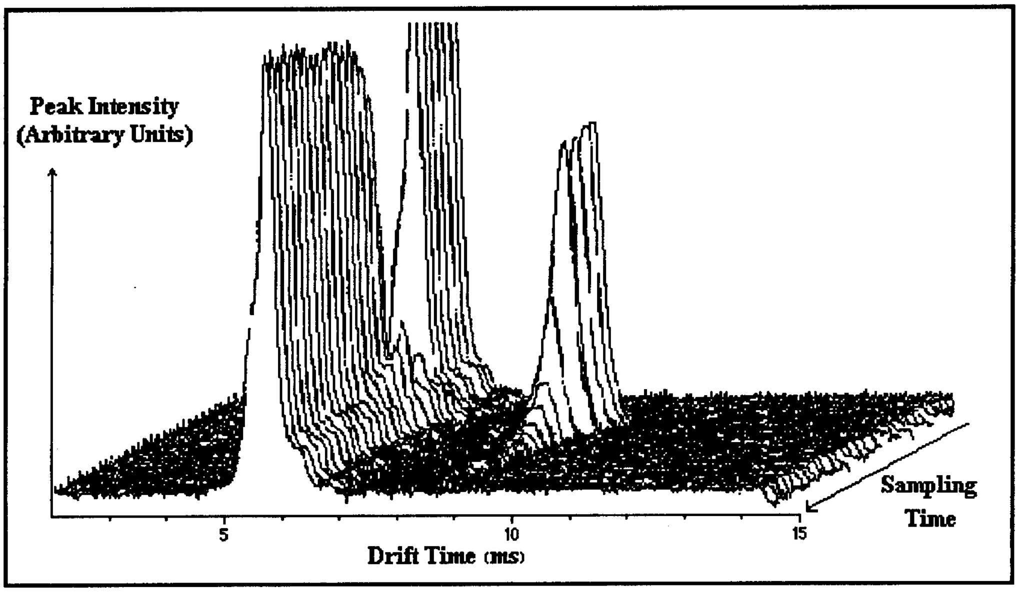

Figure 7. Ion spectra of Otto Fuel Monitor sampling over liquid Otto Fuel showing the monitors relative quick clear down time. (Spectra recorded over a 60 second period)

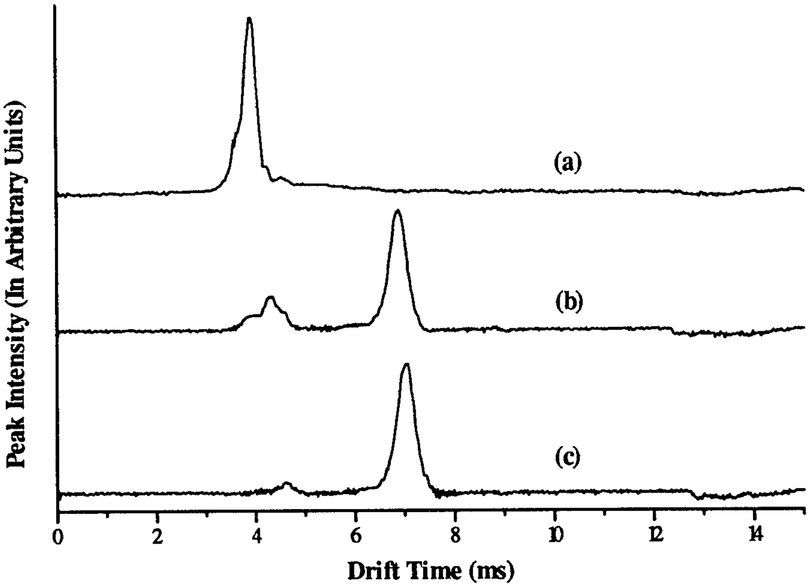

Figure 8. Ion mobility spectra produced by the Otto Fuel Monitor sampling:

(a). Clean air.
(b). methyl salicylate.
(c). Otto Fuel.

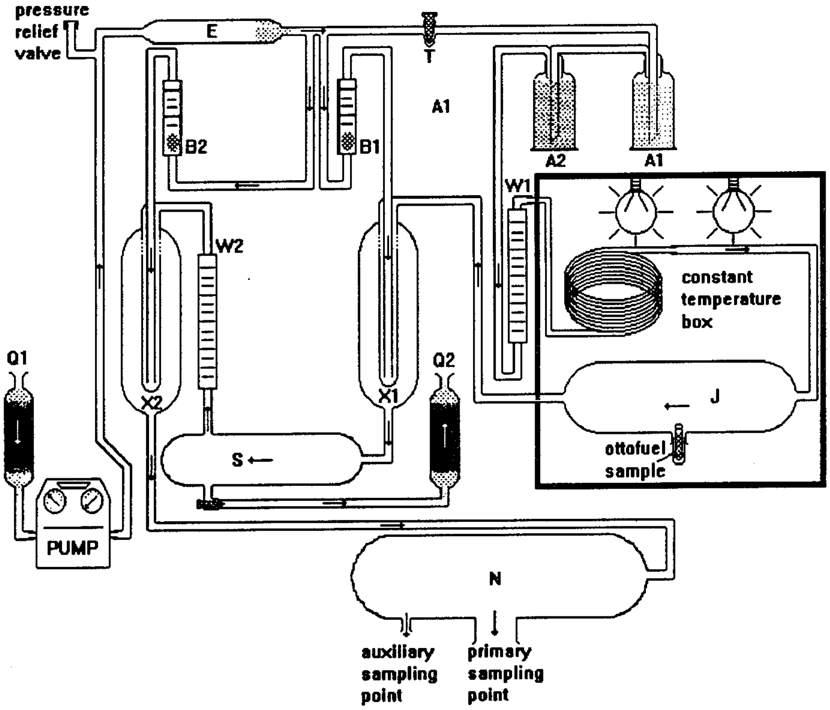

Figure 9a. "Fortuin" style apparatus used to generate air streams of constant PGDN vapour concentrations.

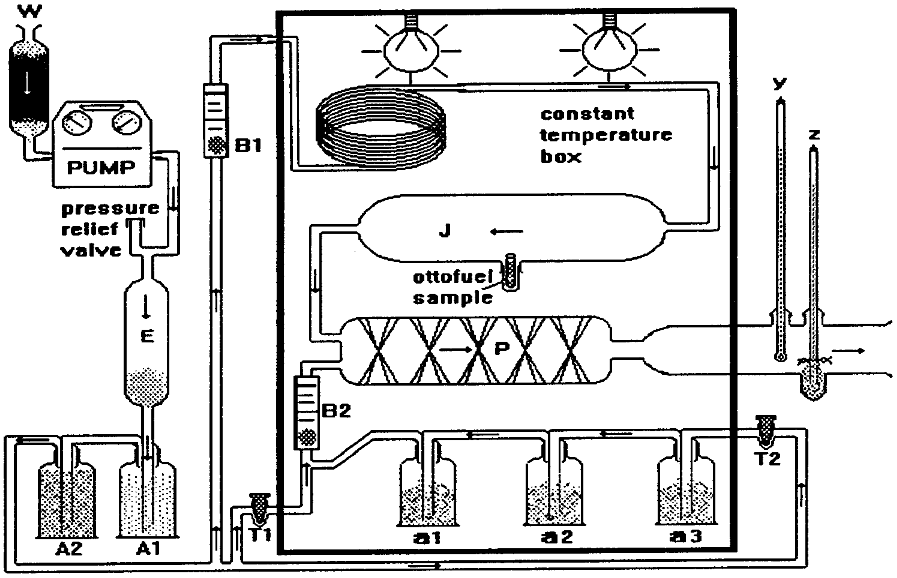

Figure 9b. "Fortuin" style apparatus used to generate air streams of constant PGDN concentration over a range of humidities.

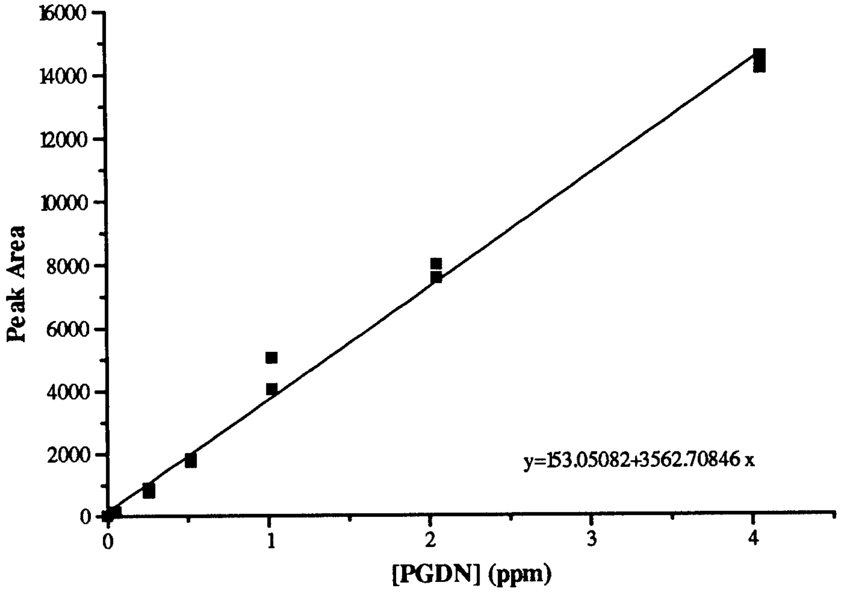

Figure 10. Gas chromatographic analytical working curve used to quantify the PGDN trapped from the air stream of the vapour generators.

Sensitivity and Operating Characteristics of the Graseby Ionics Otto Fuel Monitor (OFM MK2)

P.A. Lancaster, D.R. Leslie and V. Tantaro

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20. ABSTRACT

The response of the Graseby Ionics Otto Fuel Monitor (OFM MK2) to propylene glycol dinitrate (PGDN), the volatile, major component of Otto Fuel has been determined in a range of environmental conditions. The OFM was found capable of detecting PGDN vapour, when it is present in air, at the concentration set by the National Occupational Health and Safety Commission as the Worksafe Australia occupational exposure standard. Characteristics of the OFM that should be considered when using the monitor are discussed.