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VAPOR TAGGING OF ELECTRIC BLASTING CAPS WITH PERFLUORINATED COMPOUNDS

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Abstract. Vapor tagging of electric blasting caps (EBC) is accomplished with the use of perfluorocarbon taggants. These taggants are absorbed in either the present EBC end closures or in substitute fluoroelastomeric end closures to approximately 5-10% of the total weight of end closure. The specific taggants have been chosen to allow a 0.5 to 5 nanoliter per minute vapor taggant emission rate from the tagged EBC over a 5 year lifetime.

The taggant emission rates from tagged EBC have been experimentally observed to be well described by a taggant emission rate model. This model provides for experimental selection of the proper taggant for projected lifetimes of ten years based on just several months of observed emission measurements.

Another model has been derived which can predict the taggant concentrations in various realistic scenarios such as room, building, lockers, etc. The model takes into consideration the effect of barriers such as boxes, suitcases, etc., in impeding the release of the taggant vapors from the tagged EBC into the scenario and the dilution effect of the scenarios air circulation system. Taggant concentrations have been experimentally determined using a 425 liter sampling chamber with various barriers and the results are used with the model to predict various scenario taggant concentrations.

Introduction

An effective technique for predetonation detection of secreted explosives would be the detection of some volatile component, e.g., a taggant, present in all explosives. Most explosives however consist of a variety of chemical compositions, physical designs and packaging making detection of a common volatile component a difficult task. Yet a common element to the majority of explosives is that they are initiated by a detonator, i.e., a blasting cap. Thus one effective strategy for predetonation detection of explosives would be to detect some volatile component of blasting caps.

Electric blasting caps (EBC as produced in the United States are basically all of a common design as shown in figure 1. with an elastomeric material

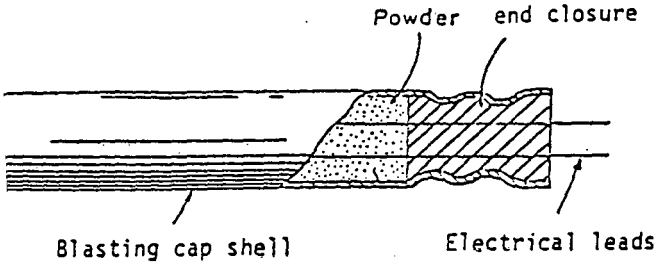

Figure 1. Basic design of an electric blasting cap.

forming the end closure of the EBC. Intrinsically the EBC's do not possess this volatile component. However this can simply be introduced into the EBC by the adsorption of a volatile taggant into the elastomeric end closure of the EBC[1,2,3]. The present end closure used in the manufacturing of EBC are substituted with the vapor taggant impregnated end closures. The subsequently emitted taggants are then detected providing for the predetonation detection of explosives.

Requirements for Vapor Taggants and Substrates

There are several requirements necessary in order to maximize the detectability of tagged EBC. The typical detection scenarios envisioned is the detection of the vapor taggant in the output air of an automated suitcase and sampling system or the detection of the taggant in a room or building containing a cache of tagged explosives. The vapor taggants should therefore be a class of chemical compounds for which there exists ultra-sensitive techniques for their detection. Specifically proposed for tagging of the EBC, is the use of perfluorinated compounds (perfluorocarbons) as vapor taggants and detection by electron capture detectors (ECD)[4]. The perfluorinated compounds also share a high sensitivity to detection by ion mobility spectroscopy, a technique similar to ECD, thus affording two separate techniques for the detection of perfluorocarbon vapor taggants.

The introduction of the vapor taggant into the EBC end closure requires the selection of a perfluorinated vapor taggant which has an appropriate solubility and emission rate in the present EBC end closures. This would minimize the impact of vapor tagging in the EBC manufacturers procedures. As a result of the above considerations, several specific requirements for a suitable vapor taggant/elastomeric substrate can be formulated and are as follows:

1. The vapor taggant must have a solubility of at least 5-10% by weight in the substrate so that the

tagged EBC will be emitting taggants over a period of 5 to 10 years. This requires that 25-50 mg of taggant be impregnated into the EBC end closures based on the present EBC design.

2. The taggant emission rate from the tagged EBC during its 5-10 year emitting lifetime should be in the 0.5 to 5 nanoliter/minute range (2 to 20 micrograms/day) in order to be detected in most scenarios.

3. The taggant must have a low ambient atmospheric concentration so as to not interfere with their detection in the scenarios. Compounds with no major industrial or commercial use are preferred.

4. The taggant should be sufficiently volatile to minimize adsorption losses, i.e., adsorption of taggants on porous materials present in the detection scenarios, such as clothing, curtains, etc. yet not as volatile so as to interfere with EBC internal timing mechanisms.

5. The physical and mechanical properties of the taggant/substrate combination should be compatible with EBC manufacturers end closure requirements.

As shall be seen there are several perfluorinated compounds which meet these requirements as vapor taggants in the present EBC end closures. If the requirement that other elastomeric substrates can be substituted as EBC end closures, then the number of acceptable perfluorinated compounds is extended.

Experimental Results

The experimental program for the development of acceptable perfluorinated vapor taggants consists of three steos. The first was an initial screening of perfluorinated compounds for those which had the requisite solubility in the present EBC end closures and in other elastomeric substrates. Vapor taggants and substrates which satisfied the first step were prepared as tagged dummy blasting caps. The emission rates of the vapor taggants from these caps where then monitored over several months in order to evaluate each taggant/substrate combination. Finally each successful taggant/substrate pair will be evaluated for their detectability in various scenarios with barriers. The above aspects are discussed in more detail as follows.

Solubility of the Vapor Taggants in the Substrates

A large number of perfluorinated compounds were examined for their solubility in the present EBC end closures and in other elastomeric substrates. The range of perfluorinated compounds included perfluoroalkanes, perfluorocycloalkanes, perfluoroalkenes, fluoroaromatics, inorganic fluorides and other miscellaneous fluorides. The solubilities were determined by exposing the substrates to the perfluorinated compounds at a variety of temperatures and pressures. A table of abbreviation for the perfluorinated compounds of interest is given in Table 1. The compounds which have at least 5% solubility (by weight) in the substrate in either one of the three present EBC end closures, Buna (DuPont), Rubber (Atlas) and Kraton (Hercules) or in Viton (DuPont) or equivalently Fluorel (3M) are tabulated in Table 2. Negative solubility represent a leaching of the substrate by the perfluorinated compound and other methods were used to verify a negligible solubility.

Table 1. Abbreviations for the Perfluorinated Compounds.

Abbreviation	Chemical Name
OFN	Octafluoronaphthalene
HFB	Hexafluorobenzene
OFT	Octafluorotoluene
DFBP	Decafluorobiphenyl
PDCB	Perfluorodimethylcyclobutane
PMCH	Perfluoromethylcyclohexane
PDCH	Perfluorodimethylcyclohexane
SF_6	Sulfur hexafluoride
PFX	Octafluoroxylene
PFP	Pentafluoropyridine
DFCH	Decafluorocyclohexene
OFCP	Octafluorocyclopentene

Table 2. Solubility of Various Perfluorinated Compound in the Present EBC End Closures and in Viton/Fluorel in % (by weight).

Perfluorinated Compounds	End Closure Substrates			
	DuPont Buna	Atlas Rubber	Hercules Kraton	Viton/ Fluorel
OFN	60.3	25.6	182.6	107.2
HFB	22.0	28.6	26.4	85.9
OFT	5.7	10.3	- 10.4	60.5
DFBP	11.2	27.8	- 4.6	60.1
PDCB	- 5.7	- 0.2	- 2.0	8.9
PMCH	- 5.3	1.7	- 5.6	7.6
PDCH	- 6.1	0.6	- 6.6	7.9
SF_6	- 0.9	1.5	2.0	7.0
PFX	8.0	6.9	- 10.3	7.3
PFP	40.9	11.5	3.0	139.0
DFCH	- 2.0	6.1	- 1.8	20.0
OFCP	- 3.0	6.6	- 6.8	24.3

The results of this initial solubility screening indicates that most perfluorinated compound have a high degree of solubility in the Viton/Fluorel fluoroelastomers whereas only the fluoroaromatics have appreciable solubilities in the present EBC end closures.

Taggant Emission Rates

Dummy tagged blasting caps were prepared from taggant/substrates combinations which were chosen on the basis of the solubility results. The end closure substrates were obtained from the EBC manufacturers and the Viton/Fluorel substrates from rubber compounders. The end closures were the same size as is presently used. The taggants were impregnated into the substrates using procedures to insure uniform impregnation of the substrate. The resulting end closures were crimped into blank EBC shells with a DuPont crimper in order to simulate as close as possible the present manufacturing techniques. Triplicate sets of tagged blasting caps were prepared for emission rate determinations at room temperature, 45°C and 65°C. The tagged caps were tare weighed periodically on a microbalance for a period of several months in order to determine gravimetric loss data i.e., the rate of weight loss of the tagged cap due to taggant emission. Occasionally the emission rates determined from the gravimetric loss data were veri-

fied by a chromatographic determination of the emission rates with good agreement between the two methods.

The taggant emission rates as derived from the gravimetric loss data were obtained over a time period up to 6 months. In order to fully evaluate the various taggant/substrate combinations an emission rate model was derived which would be able to predict emission rates at time periods up to 5 years based on a few months of gravimetric loss data. The taggant emission rate model was derived by applying the diffusion equation to the geometry of a tagged blasting cap with the following model assumptions.

1. The rate of taggant emission from the tagged blasting cap is determined by
   a. The rate of taggant diffusion in the substrate plug as characterized by a taggant/substrate diffusion constant.
   b. The rate of taggant mass transfer across the substrate plug end as characterized by a taggant mass transfer coefficient.
2. The taggant diffusion constant is independent of the initial taggant concentration in the substrate plug.
3. The substrate plug is impregnated uniformly with the taggant, at least in the axially direction, at the time of impregnation.

Solution of the differential diffusion equation with these model assumptions leads to the following expression for the taggant emission rate

$$\frac{dc(t)}{dt} = - \frac{c(o)}{Z_0}\left(\frac{D}{\pi t}\right)^{\frac{1}{2}}\left(1+2\sum_{n=1}^{\infty}(-1)^n \exp\left[-\frac{n^2 Z_0^2}{Dt}\right]\right) \qquad (1)$$

in which dc(t)/dt is the taggant emission rate (units: milligrams of taggant per second), c(o) the amount of taggant impregnated into the substrate plug (units: milligrams of taggant), D the taggant/substrate diffusion constant (units: square centimeters per second) and t the age of the tagged blasting cap, i.e., the elapsed time after impregnation (units: seconds). This solution reflects the experimental observation that the taggant mass transfer rate across the plug end is negligible when compared to the taggant diffusion rate in the substrate plug. In most situations the emission rate expression, equation (1), can be approximated by the first term, i.e.,

$$\frac{dc(t)}{dt} \cong - \frac{c(o)}{Z_0}\left(\frac{D}{\pi t}\right)^{\frac{1}{2}} \qquad (2)$$

yielding a simple inverse square root of time dependence for the taggant emission rate.

Integration of equation (2) leads to the following approximate expression for c(t), the total amount of taggant remaining in the substrate plug as a function of elapsed time after impregnation, i.e.,

$$c(t) \cong c(o) - 2c(o)\left(Dt/\pi Z_0^2\right)^{\frac{1}{2}} \qquad (3)$$

This expression is used to obtain the taggant/substrate diffusion constants from the experimentally obtained gravimetric loss data. With this diffusion constant equation (1) permits the prediction of the taggant emission rates from a tagged blasting cap up to 5 to 10 years after impregnation based on data from a few months of following the weight change of a tagged blasting cap.

An example showing the agreement between the experimental gravimetric loss data and agreement to equation (3) is given in figure 2.

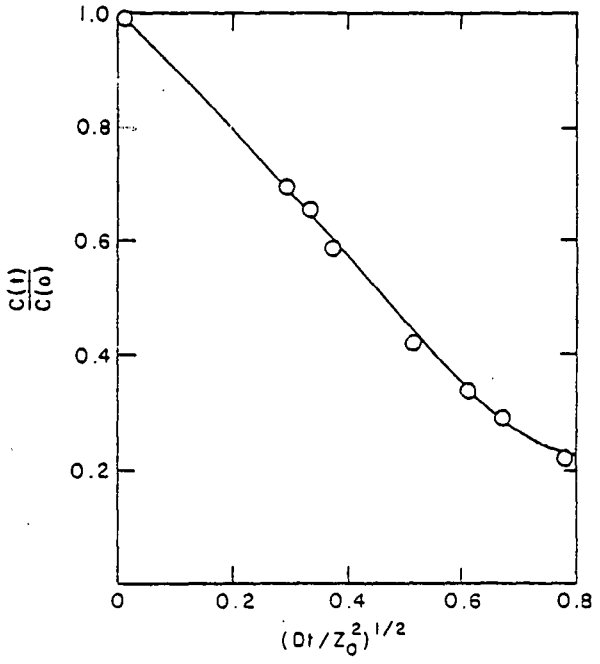

Figure 2. Experimental fractional loading c(t)/c(o) plotted as a function of the reduced time $(Dt/Z_o^2)^{\frac{1}{2}}$ for the PDCB/Fluorosilicone rubber combination. The solid line is a best theoretical fit with D = 2.5 x 10^{-7} cm^2/sec.

All of the taggant/substrate combinations studied to date show equally a good fit between the gravimetric loss data and the theoretical emission rate model. On the basis of this success, taggant emission rates can be confidently predicted using the experimentally obtained diffusion constants for each taggant/substrate combination. Thus the evaluation can be made of the taggant emission rates.

An example calculation of emission rate using the model is given in Table 3 for taggant/substrate combination with suitable emission rates. As can be seen in Table 3, after 5 years a little less than half of the initially impregnated OFN still remains in the tagged cap. At 5 years the OFN emission rate is 0.41 mg/min within the requirements for the taggant emission rates. Taggant emission rates can be increased or decreased by simply impregnating more or less taggant into the blasting cap since the emission

Table 3. Emission Rates from an OFN/DuPont Buna Tagged Blasting Cap as a Function of the Age of the Tagged Cap. Fifty mg OFN Initially Impregnated.

Age of Tagged Blasting Cap	Taggant Emission Rate (nanoliters/minute)	Amount of Taggant Remaining in Cap (milligrams)
1 day	17.5	49.39
10 days	5.55	48.06
1 month	3.15	46.59
1/2 year	1.29	41.71
1 year	0.919	38.27
2 years	0.649	33.42
5 years	0.410	23.78

rate has a linear dependence on the initial amount of taggant c(o). This is possible since experiments have been performed verifying the model assumption that the diffusion constant is indeed independent of the initial amount of impregnated taggant. This assumption has been verified up to at least 25% initial taggant impregnate (25% of the weight of the substrate).

Taggant/substrate combinations which meet the taggant emission rate requirements (0.5 to 5 nℓ/min over a 5-10 year period) are presented in Table 4. There are at least two taggants with suitable solubilities and taggant emission rates in each of the two present EBC manufacturers, DuPont and Atlas. An evaluation of the taggants for the third manufacturer, Hercules, is not yet available due to an earlier unavailability of the Hercules end closure material in a suitable form. However it appears, based on preliminary estimates, that OFN will satisfy the requirements in the Hercules material. If for other reasons the above taggants and the present EBC end closures cannot be used, then the Viton/Fluorel substrate can be used as an EBC end closure material.

Gravimetric loss data obtained from tagged blasting caps stored at 45°C and 65°C has similarly been analyzed using the emission rate model. Diffusion constants exhibited the expected temperature, i.e., an Arrhenius equation dependence

$$D = D_0 \exp\left(- E_d/RT\right) \qquad (4)$$

(D the measured diffusion constant at temperature T, D_0 a pre-exponential factor, E_d the activation energy for diffusion and R the gas constant). An example is shown in figure 3 for the OFN/DuPont Buna blasting cap combination. Least squares analysis of the diffusion constants gives an activation energy of 18.7 ± 2.3 kcal/mole. This value is comparable with other values in the literature. This gives further verification to the taggant emission model used in the analysis.

Table 4. Acceptable Taggant/Substrate Combinations.

Taggant/Substrate	Diffusion Constant (10^{-9} cm^2/sec)	Emission Rate at 1 year (nℓ/min)
OFN/DuPont Buna	3.2 ± 1.7	0.92
DFBP/DuPont Buna	7.4 ± 1.2	1.4
OFCP/Atlas Rubber	17.6 ± 0.5	2.2
OFBP/Atlas Rubber	21.4 ± 0.3	1.9
PFP/Viton A	9.07 ± 0.06	3.0
OFCP/Viton A	0.62 ± 0.05	0.62
DFCH/Viton A	0.283 ± 0.026	0.34
PFX/Viton A	0.68 ± 0.07	0.59
HFB/Viton A	3.25 ± 0.03	0.81
OFT/Viton	1.23 ± 0.02	0.63

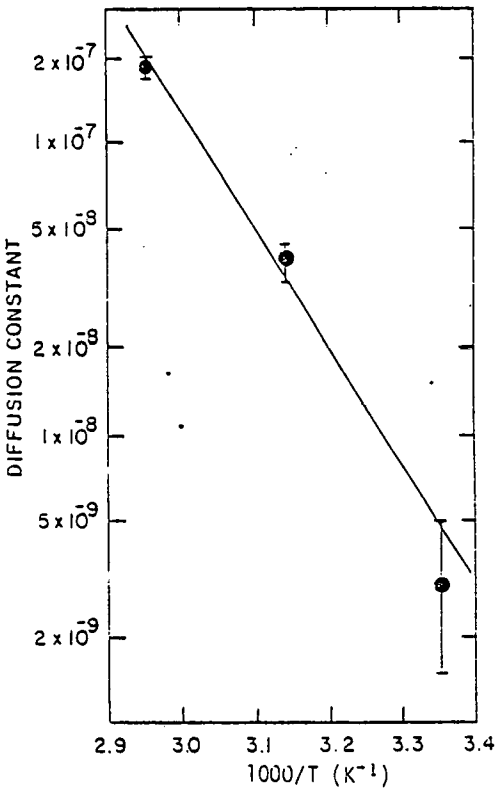

Figure 3. Experimental obtained diffusion constants for the OFN/DuPont blasting cap as a function temperature. Diffusion constant in cm^2/sec.

Expected Taggant Concentration in Scenarios from Tagged Blasting Caps in Various Barriers

As has been seen in the preceding sections it is entirely possible to prepare a vapor tagged EBC with the appropriate taggant emission rates which will emit over a 5 year period. However the successful use of tagged blasting caps or in general tagged explosives is dependent on the ability to detect the taggants in various detection scenarios. In order to realistically assess the capabilities of tagged blasting caps, expressions have to be available to predict the

taggant concentrations in the detection scenarios given certain known scenario and barrier parameters. Consequently an expected taggant concentration model has been derived. The model has been kept as general as possible so as to be applicable to the majority of realistic scenarios.

Conceptually the barriers and detection scenarios will be formulated as follows: A tagged blasting cap with a known taggant emission rate is placed within a barrier enclosure, realistic examples of which are boxes, cans, plastic bags, suitcases, etc., which in turn are placed in some scenario. By a scenario it is meant a defined space with a known volume and ventilation rate in which the taggant will be detected. Examples of scenarios are lockers, rooms, buildings, etc. A ventilation rate is included to reflect the constant exchange of air within the scenario with air from outside the scenario. In the majority of bombing situations there is a time delay between the introduction of the explosive into the barrier enclosure and the introduction of the barrier enclosure into the detection scenario. This time delay has been included as a variable in the model.

Expressions for the expected scenario taggant concentration have been derived with the following assumptions:

1. The taggant emission rate from the tagged blasting cap is constant over the time interval from the introduction of the cap into the barrier enclosure to the detection of the taggant in the scenario.
2. The emission of the taggant from the barrier enclosure is diffusion controlled, i.e., best described by Fick's first law of diffusion, and characterized by a barrier parameter k.
3. There is a uniform mixing of the taggant with the air in the barrier enclosure and in the scenario.
4. The air exchange rate due to ventilation of the scenario is constant with time.

The barrier parameter k can be expressed as

$$k = AD/\ell \qquad (5)$$

in which A is the total area of the cracks in the barrier, ℓ is an average depth of the crack and D is the taggant-air diffusion constant. This barrier parameter cannot be a priori determined for barriers such as boxes, briefcases, cans, etc., but rather has to be determined by experiment and extracted with the use of the expected taggant concentration model. However, an order of magnitude estimation can be made. Given in Table 5 is a listing of barrier severities corresponding to various values of the barrier parameter k as based on experimental evidence.

Table 5. Barrier Parameter Severities.

Degree	Barrier Parameter k (liters/min)
Severe	2×10^{-4}
Moderate	2×10^{-2}
Slight	2

An example of a barrier enclosure is a six inch diameter paint can with a 1 micron (1×10^{-6} m) crack about the circumference of the lid. In this instance the crack area is 0.48 mm^2. Thus with the taggant-air diffusion constant being approximately 5 cm^2/min and assuming the cracks etc. to be 1 mm in depth we obtain a barrier parameter of 2.4×10^{-4} ℓ/min, which is indeed classified as a severe barrier.

The expected taggant concentrations in both the scenario and inside the barrier can be calculated from expressions derived with the use of the above assumptions. A fuller discussion is given elsewhere.[5] The resulting expressions are applied to two specific examples as follows.

A. Expected taggant concentrations inside a briefcase. This detection scenario is relevant in sampling of briefcases, suitcases, etc. The assumption here is that a tagged cap has been placed within a briefcase and that the briefcase is not exposed for any length of time to any defined volume in which the taggant concentration can build up, i.e., the briefcase is constantly in an environment that is rapidly changing. The only assumed mechanism for the loss of taggant vapors from the briefcase is diffusion through the seams of the briefcase.

Table 6 gives the results of the calculation assuming a taggant emission rate of 1 nanoliter per minute and for two degrees of barrier severity. It shows that the taggant concentration is at least in the ppb range. Thus if the taggant vapors are diluted by a factor up to 1000 by some sort of briefcase/suitcase sampling scheme, it is still possible to detect the taggant vapors with a real time continuous instrument having a sensitivity of at least 1 ppt. Naturally the dilution factor will be greater for a severe barrier enclosure than for a slight one.

B. Expected taggant concentrations in a moderately sized room. A moderate sized room (10' x 20' x 8' or 1600 cu ft) with a ventilation rate of 54 cubic feet per minute (corresponding to a complete exchange of air every 30 minutes, as specified by modern building ventilation standards) has been chosen to illustrate a volume sampling scenario. A tagged blasting cap contained within a barrier enclosure the size of a briefcase is placed into the room. The expected taggant concentrations inside the room calculated from the model assuming a barrier parameter for the briefcase of 2×10^{-2} ℓ/min (a moderate barrier) and a taggant emission of 1 nℓ/min are given in Table 7. The taggant concentrations are given as a

Table 6. Expected Taggant Concentrations Inside a Briefcase Have Various Degrees of Barrier Severity.

Time Elapsed	Expected Taggant Concentration, ppb 10^9 Severe Barrier, $k = 2 \times 10^{-4}$ ℓ/min	Moderate Barrier, $k = 2 \times 10^{-2}$ ℓ/min
15 min	1.32	1.31
30 min	2.65	2.58
1 hr	5.29	5.02
2 hrs	10.6	9.54
5 hrs	26.4	20.6

Table 7. Scenario Taggant Concentrations in a Moderately Sized Room with a Moderate Barrier.

Time after Introduction of Barrier Into Scenario	Taggant Concentration, pp 10^{12} (ppt) Time Delay 1 hr	2 hrs	4 hrs
15 min	.0295	.0527	.0924
30 min	.0535	.0902	.153
1 hr	.0919	.140	.223
2 hrs	.147	.196	.282
4 hrs	.250	.291	.362

function of the time delay between the placing of the tagged explosives into the briefcase and the placing of the briefcase in the room. Typically this time is of the order of 1 hour. As can be seen in Table 7 all of the taggant concentrations are within the detectability of a concentrating taggant detection system, i.e., greater than parts in 10^{16}.6 The greater the time delay between placing the cap into the barrier and the barrier into the scenario the faster the initial approach to the taggant steady state concentration.

Experimental Determination of the Barrier Parameter

The barrier parameter k has to be determined experimentally for each particular class of barriers, i.e., for briefcases, boxes, plastic bags, etc., with the chosen vapor taggants. Figure 4 is the results of an experiment to determine the barrier parameter of a tightly sealed paint can barrier containing a tagged EBC emitting at a known emission rate. The resulting taggant concentration is chromatographically determined in the air of a 426 liter sampling chamber (representing a known volume scenario) containing the paint can. The solid line represents the best fit of the expected taggant concentration model to the experimentally determined taggant concentrations and corresponds to a barrier parameter of $(6.0 \pm 0.7) \times 10^{-5}$ ℓ/min for the paint can barrier, indeed one of the most severe barriers that can be planned to be expected in realistic detection scenarios.

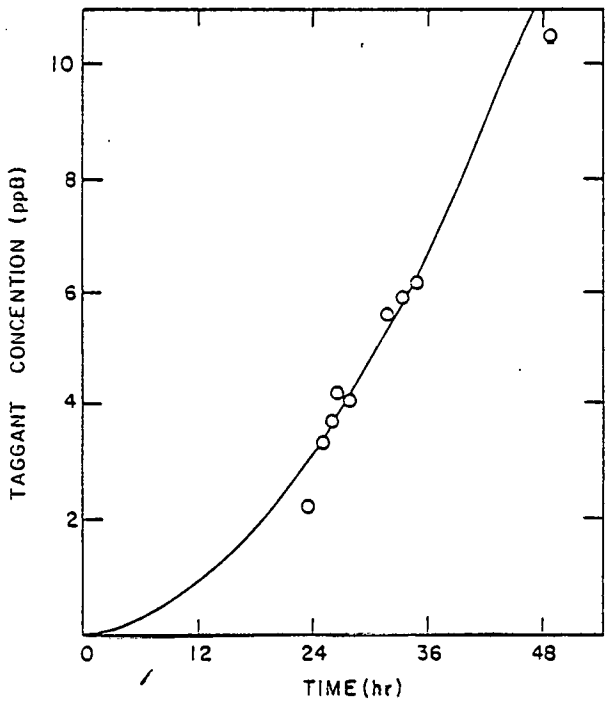

Figure 4. Determined taggant concentrations in a 425 liter sampling chamber as emitted from a paint can enclosed tagged EBC. The time is in hours after introduction of the paint can into the sampling chamber.

This experimental system will also be used for the examination of the extent of adsorption losses of the vapor taggants on porous surfaces present in the barriers and in the detection scenarios, e.g., clothing, in suitcases, packing material in boxes, etc., which would tend to reduce the expected tagged concentrations. These losses would be minimal for taggant with a high degree of volatility and this is work presently in progress.

Conclusions

The feasibility of tagging electric blasting caps with perfluorocarbon vapor taggants has been experimentally demonstrated. Specifically there are two taggants available for the EBC manufactured by DuPont, two taggants available for those manufactured by Atlas and at least one possible taggant available for those manufactured by Hercules. These taggants when impregnated into the EBC end closures can be expected to emit the vapor taggants at emission rates between 0.5 to 5 nanoliters per minute up to at least five years after the manufacture of the EBC. These vapor taggants can be sensitively detected by electron capture detectors and by ion mobility spectroscopy which has a slightly smaller degree of sensitivity when compared to ECD. If for some reason the present EBC end closure impregnated with the vapor taggants cannot be used, then an alternate end closure material (Viton or Fluorel fluoroelastomers) will provide a suitable substrate for impregnation with any of six acceptable vapor taggants.

The taggant concentration within briefcases/suitcases from a 1 nanoliter per minute tagged cap has been calculated to be of the order of parts per 10^{9} for almost all degrees of briefcase/suitcase barrier severities. This will allow real-time continuous detection of the taggants in a sampling system tolerating a dilution factor up to 1000 with detectors having sensitivities of the order 1 ppt (pp 10^{12}).

The calculated taggant concentrations in a moderately sized room is sufficient so as to detect a 1 nanoliter per minute cap with a concentrating detection scheme 15 minutes after introduction of a severe barrier enclosure containing the cap which had been placed into barrier one hour earlier. A moderate barrier will almost allow real-time continuous detection under the same circumstances.

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