

Journal of Conventional Weapons Destruction

Volume 9
Issue 1 *The Journal of Mine Action*

Article 50

August 2005

Some Steps to a Refined REST Technology

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Recommended Citation

Goss, Kai-Uwe (2005) "Some Steps to a Refined REST Technology," *Journal of Mine Action* : Vol. 9 : Iss. 1 , Article 50.

Available at: <https://commons.lib.jmu.edu/cisr-journal/vol9/iss1/50>

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Some Steps to a Refined remote explosive scent tracing Technology

by Kai-Uwe Goss | Environmental Chemistry, EAWAG and ETH |

In the Remote Explosive Scent Tracing (REST) technology, air from a suspect site is passed through a filter in order to trap the target odor. The filter is then analyzed in a laboratory environment, usually by animals. Although REST is a potentially efficient technology for area reduction, it appears that there has not yet been any strict optimization of the technical procedure. The following work gives a theoretical analysis of the REST concept and first results of a lab study on the performance of various filters for the sampling of gaseous 2,4-DNT.

ence on the performance of the REST procedure, can be characterized by a sorption coefficient, $K_{i \text{ filter/air}}$. It is defined as the equilibrium concentration of a chemical, i , on the filter divided by the equilibrium concentration of i in the air (in the following, the subscript i will be used for all parameters that depend on the considered target scent):

EQUATION 1

$$K_{i \text{ filter/air}} = \frac{C_{i \text{ filter}}}{C_{i \text{ air}}} = \frac{\text{amount of scent } i \text{ (mol) per mass of filter (g)}}{\text{amount of scent } i \text{ (mol) per volume of air (l)}}$$

For this discussion, we will assume the following:

1. A sorption/desorption equilibrium will be

scent will be sucked onto the filter. While the operator proceeds, "clean" air is sucked through the filter again until the sampling process is finished. As more and more clean air is sucked through the filter, the trapped concentration peak travels through the filter towards the filter outlet. The velocity of the scent peak traveling through the filter depends on $K_{i \text{ filter/air}}$ and the flow rate of the sampled air. While traveling through the filter cartridge, the peak also gets broader. The peak maximum will reach the filter outlet when the filtered air volume, $V_{\text{air}}^{\text{sample}}$, equals the product of $K_{i \text{ filter/air}} \times M_{\text{filter}}$. At that time, half of the scent molecules that had originally been trapped have already left the filter while the other half are still trapped on the filter. For a good performance of REST, breakthrough of more than 50 percent of the scent must be avoided, i.e., $V_{\text{air}}^{\text{sample}}$ must be smaller than $K_{i \text{ filter/air}} \times M_{\text{filter}}$.

Refining REST Technology

Obviously the REST procedure must be optimized such that any explosive scent present in the sampled field-air will eventually be detected by the sniffing animals. Thus, we can start the optimization process by requiring that the concentration of scent i released by the filter into the air, $c_{i \text{ air}}$, in the analysis procedure should be maximal. To this end, one first has to ensure that a sorption/desorption equilibrium is obtained between the filter in the filter-stand and the surrounding air within a reasonable amount of time, which is assumed for the following discussion. For the air concentration, $c_{i \text{ air}}$, available to the animal, we can then derive the following equation:¹

EQUATION 2

$$c_{i \text{ air}} = \frac{M_{i \text{ on filter}}}{V_{\text{air}}^{\text{analysis}} + K_{i \text{ filter/air}} M_{\text{filter}}}$$

($M_{i \text{ on filter}}$ is the amount of scent i trapped on the filter, M_{filter} is the amount (g) of filter material used and $V_{\text{air}}^{\text{analysis}}$ is the volume of air into which the explosive scent is eventually released again.)

This volume, $V_{\text{air}}^{\text{analysis}}$, is not easily defined. It depends on the volume of air sniffed by the animals and the air volume that passes the exposed filter due to the normal convection and circulation of air in the room where the analysis takes place. Therefore, $V_{\text{air}}^{\text{analysis}}$ is not a constant number but increases with the exposure time of the filter. It is obvious from EQUATION 2 that $c_{i \text{ air}}$ becomes

Notes

1. The following analysis aims for an optimization of REST in combination with sniffing animals. Optimization of the desorption procedure would be different if analytical instruments were used for detection.
2. The following analysis does not apply if the major load of the sampled scent is not in gaseous form but is attached to soil/dust particles that are sucked in during sampling.
3. REST has also been applied to detect explosive scents in cars or other closed containers. This application has some distinctively different features from the REST procedure that is used in demining and therefore requires a somewhat different optimization.

Obviously, a maximal concentration of the target scent should be made available to the animals in the analysis procedure of REST. Intuitively, one would want to use a strong filter in order to trap as much of the scent as possible from the filtered air; however, a strong sorbing filter will eventually not release much of the trapped scent into the air inhaled by the animal. The following analysis investigates how to optimally select the filter material, the mass of filter (M_{filter}), and the volume of air ($V_{\text{air}}^{\text{analysis}}$) into which desorption takes place when the filter is eventually exposed for analysis by the animals. These and some other factors (e.g., temperature, humidity, volume of sampled air) are discussed.

Sorption Coefficients

Different filter materials (e.g., different polymers, activated charcoal) exhibit different affinities to a given chemical (i.e., the target scent). This affinity, which obviously must have an influ-

ence on the performance of the REST procedure, can be characterized by a sorption coefficient, $K_{i \text{ filter/air}}$. It is defined as the equilibrium concentration of a chemical, i , on the filter divided by the equilibrium concentration of i in the air (in the following, the subscript i will be used for all parameters that depend on the considered target scent):

2. A linear sorption isotherm is valid, i.e., $K_{i \text{ filter/air}}$ does not depend on the actual concentrations. The sorption coefficient, $K_{i \text{ filter/air}}$, depends on the filter material as well as the type of scent that is looked at and needs to be known beforehand. $K_{i \text{ filter/air}}$ further depends on temperature and, for hydrophilic filter materials, on relative humidity in the air. To begin with, we will assume that the temperature in the sampling and analysis procedure is the same and that there is no humidity effect. At a later stage in this discussion, these two points will be taken up again.

Functioning of a Filter

Let us consider an operator who collects an air sample onto a filter while moving forward in the field. When the operator passes an area where a mine is buried a concentration peak of the target

maximal if $M_{i \text{ on filter}}$ is maximal (CONDITION 1) and $V_{\text{air}}^{\text{analysis}} + K_{i \text{ filter/air}} M_{\text{filter}}$ is minimal (CONDITION 2).

CONDITION 1 can be satisfied if

- 1A. The concentration of the target scent in the sampled air is maximal.
- 1B. The sampled target scent i is trapped on the filter without major losses.

For 1A, i.e., the concentration of the target scent in the sampled air depends on the ambient conditions during the sampling procedure. A discussion of optimal ambient sampling conditions is extremely complex and beyond the scope of this manuscript. Note, however, that the scent concentration in the air analyzed by the animals in a REST procedure will always² be smaller than the original air concentration in the field under sampling conditions. This follows from a combination of EQUATION 2 and 3. Hence, it is not advisable to collect filter samples in the field under ambient conditions where air concentrations of the scent are known to be below the detection limit of a dog.

CONDITION 1B ("the target scent is completely [>90 percent] retained on the filter") can be satisfied if (see the above discussion on the functioning of a filter):

EQUATION 3

$$K_{i \text{ filter/air}} M_{\text{filter}} \geq V_{\text{air}}^{\text{sample}}$$

CONDITION 2 demands that the product of $K_{i \text{ filter/air}} \times M_{\text{filter}}$ be minimal which, in combination with EQUATION 3 from CONDITION 1B, results in the following requirement:

EQUATION 4

$$K_{i \text{ filter/air}} M_{\text{filter}} \approx V_{\text{air}}^{\text{sample}}$$

Furthermore, it follows from CONDITION 2 that $V_{\text{air}}^{\text{analysis}}$ should be small (e.g., 10 percent) compared to the term $K_{i \text{ filter/air}} M_{\text{filter}}$ in order not to substantially increase the complete term $V_{\text{air}}^{\text{analysis}} + K_{i \text{ filter/air}} M_{\text{filter}}$, resulting in the following equation:

EQUATION 5

$$V_{\text{air}}^{\text{analysis}} < 0.1 K_{i \text{ filter/air}} M_{\text{filter}}$$

Before we come to the final conclusions based on these equations, we should shortly investigate the influence of temperature and relative humidity (RH).

Relative Humidity

If adsorbents with a hydrophilic surface (e.g., clay minerals, silica) were used, then RH would have a strong (i.e., exponential) impact on $K_{i \text{ filter/air}}$ due to the competition of the water vapor with the target scent for the adsorption sites on the sorbent surface.^{3,4} This effect can also occur unintentionally if soil particles accumulate on the filter during sampling and eventually surpass the original filter in its sorption capacity. Adsorbents like organic polymers that are used as filter material are not expected to depend much on RH in their absorption capacity. Humidity may of course also strongly effect the actual con-

centration of the target scent in the field-air, but this effect does not directly impact the filter performance and is therefore beyond the scope of this paper.

Temperature

In general, $K_{i \text{ filter/air}}$ decreases with increasing temperature. In case the air-temperature deviates from the filter-temperature, it is the latter that determines the sorption coefficient. The actual temperature sensitivity depends on the specific target scent and the type of sorbent. The $K_{i \text{ filter/air}}$ -value for 2,4-DNT will roughly decrease by a factor of three per temperature increase of 10 C for any typical polymer (this value also applies to the sorption on minerals, but only if relative humidity stays constant).

Having this knowledge on the impact of temperature, we need to reconsider the above analysis of optimal REST conditions. Obviously the danger of a filter breakthrough can be further minimized (CONDITION 1B) if the filter is cooled during sampling; due to technical constraints however, this option does not seem feasible. It seems more realistic to assume that REST sampling will always take place at ambient temperature. We can thus rewrite the condition defined by EQUATION 4 with a temperature-specific sorption coefficient:

EQUATION 6

$$K_{i \text{ filter/air}}^{T_{\text{sampling}}} M_{\text{filter}} \approx V_{\text{air}}^{\text{sample}}$$

It is also clear that any increase in the filter temperature during the analysis procedure will increase the release of the target scent to the animal's nose. A temperature increase of 20 C would increase the air concentration for the animals by almost a factor of 10, provided that the criterion of EQUATION 5 is still met. The upper temperature limit will be given by the physiology of the animals and by the condition given in EQUATION 5, which becomes more difficult to comply with at higher temperatures. Hence, we also need to rewrite EQUATION 5 with a temperature-specific sorption coefficient ($K_{i \text{ filter/air}}^{T_{\text{sampling}}}$):

EQUATION 7

$$V_{\text{air}}^{\text{analysis}} < 0.1 K_{i \text{ filter/air}}^{T_{\text{sampling}}} M_{\text{filter}}$$

With EQUATIONS 6 and 7, we have reached the final conditions that tell us how to optimize the technical procedure of the REST method.

Just as with humidity, ambient temperature may also strongly affect the actual concentration of the target scent in the field-air. Again, this

effect does not directly impact the filter performance and thus is beyond the scope of this paper.

Analysis Procedure by Sniffing Animals in REST

Filters should be heated during the analysis procedure. The upper temperature limit mainly depends on the animal's physiology and still has to be established. A temperature increase of 10 C can increase the air concentration provided to the animal's nose by about a factor of three. The technical equipment of the filter stand, filter holder and filter itself should be designed such that the complete filter material is able to equilibrate within a short time (minutes) with a small volume of surrounding air.

This air volume, $V_{\text{air}}^{\text{analysis}}$, should be kept below a threshold (given by EQUATION 7) in order to allow a maximal air concentration ($c_{i \text{ air}}$) to be established. If the requirement of EQUATION 6 has been met during the sampling procedure, then EQUATION 7 is equivalent to

EQUATION 8

$$V_{\text{air}}^{\text{analysis}} < 0.1 V_{\text{air}}^{\text{sample}} / 3 (T_{\text{analysis}} (^{\circ}\text{C}) - T_{\text{sampling}} (^{\circ}\text{C})) / 10$$

For example, if we assume that $V_{\text{air}}^{\text{sample}}$ equals 100 L (under ambient pressure) and the analysis temperature is 20 C above the sampling temperature, then $V_{\text{air}}^{\text{analysis}}$ should be kept smaller than 1 L in order to comply with EQUATION 8. Note, that there is no advantage in reducing $V_{\text{air}}^{\text{analysis}}$ any further because then the term $K_{i \text{ filter/air}}^{T_{\text{analysis}}} M_{\text{filter}}$ dominates the air concentration, $c_{i \text{ air}}$ (see EQUATION 2).

Sampling Procedure in REST

EQUATION 6 defines the conditions that one should obey in the sampling procedure. It indicates that there is not one optimal filter material, but for every material there is an optimal amount of filter that should be used. For example, if we assume again that $V_{\text{air}}^{\text{sample}} = 100$ L (under ambient pressure), then we can calculate the filter mass that should be used for a given filter material if 2,4-DNT is the target scent (see TABLE 1). (Note that the pressure in the filter during filtration is smaller than ambient pressure due to the applied vacuum. Hence, if the filtered air volume $V_{\text{air}}^{\text{sample}}$ is measured under pressure conditions that are different from that in the filter, then a pressure correction is needed before the number for $V_{\text{air}}^{\text{sample}}$ can be used in the above equations.)

If more than the calculated filter mass is used (i.e., if the product $K_{i \text{ filter/air}}^{T_{\text{sampling}}} M_{\text{filter}}$ is larger than

Sorbent-Polymer	$K_{\text{polymer/air}}$ (L/g) at 20 C	M_{filter} (g)
Cotton Wool	2.5	=> 40.0
Polyester Wool	15	=> 6.6
PU foam	8,700	=> 0.012

TABLE 1: Mass of filter (g) that should be used in order to retain 50 percent of the sampled 2,4-DNT on the filter at a sampling temperature of 20 C. Results have been calculated according to EQUATION 6 assuming that $V_{\text{air}}^{\text{sample}} = 100$ L. The sorption coefficients, $K_{\text{polymer/air}}$, for 2,4-DNT were determined in our lab for a linear flow rate of 30 ml/min and RH of 91 percent (see experimental section below). Note that filter masses required to retain 50 percent of 2,4,6-TNT under the same conditions would be more than a magnitude smaller.

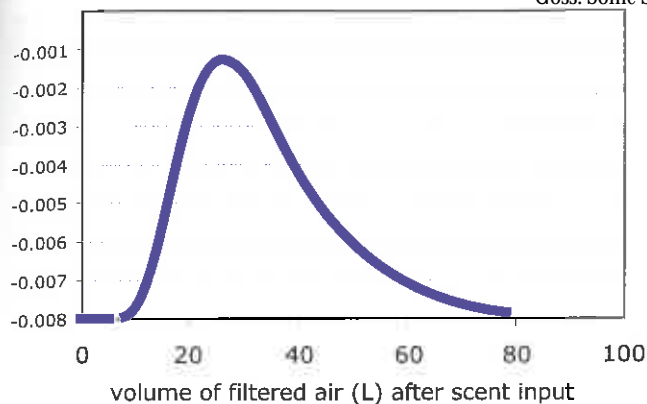


FIGURE 1
Breakthrough curve for 2,4-DNT on 2.3 g polyester wool filter at 20.0 C and 91 percent RH.

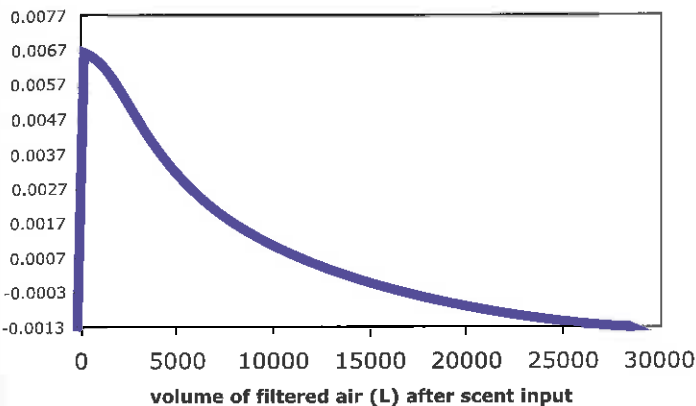


FIGURE 2
Breakthrough curve for 2,4-DNT on 6.8 g PVC gauze filter at 20.0 C and 91 percent RH.

$V_{\text{air}}^{\text{sample}}$), then the target scent will be sorbed more than necessary so that its release into the analyzed air will decrease unnecessarily. If less than the calculated filter material is used, then some of the target scent might be lost due to filter breakthrough (especially if the scent was sucked onto the filter right at the beginning of the sampling interval), which in turn also lowers the concentration of the scent in the air that is to be analyzed by the animals. In order to meet the requirement from EQUATION 6 as precisely as possible, it is advisable to measure $K_{\text{polymer/air}}$ (L/g) for the considered filters under typical field conditions (temperature, volume flow rate) instead of using data from the literature. (The sorption behavior of polymers also depends on additives and other factors, so the same nominal material may perform quite differently depending on subtle differences in the production.)

It must also be noted that the air concentration produced in the analysis procedure is almost inevitably smaller than the originally sampled concentration in the field air. Hence field conditions that are unfavorable for a direct use of sniffing animals are also unfavorable for the REST technology.

So far, the volume of sampled air, $V_{\text{air}}^{\text{sample}}$, was taken as a given number. It will usually be defined by various practical aspects of the REST method, i.e., the technical equipment, the pace of the operator collecting the sample, etc. However, this volume also has an impact on the performance of the REST procedure. The smaller the sampled air

volume, i.e., the more frequently the filter cartridge is changed under otherwise constant sampling conditions, the higher the air concentration, c_{air} , that can be obtained eventually in the analysis step will be. Note, though, that this statement only holds if the filter mass, M_{filter} , and $V_{\text{air}}^{\text{analysis}}$ are adjusted to the smaller sampling volume, $V_{\text{air}}^{\text{sample}}$, according to EQUATIONS 6 and 7.

Laboratory Experiments

In a series of laboratory experiments, the following filter materials were tested for their breakthrough behavior with respect to 2,4-DNT: PVC gauze, cotton wool, polyester wool and polyurethane foam. The performance of the last three did not depend substantially on RH or the linear gas velocity during sampling and the results can be summarized in the sorption coefficients reported in TABLE 1 (see FIGURE 1 for a typical breakthrough curve).

The PVC gauze exhibited quite different behavior from the other materials (see FIGURE 2 for the breakthrough curve). A small amount of compound would break through immediately after it had entered the filter. The long tailing of the peak indicates that sorption is kinetically hindered due to very slow diffusion into the PVC material. Only a very small fraction of those molecules that have diffused into the PVC material will eventually be desorbed to the air in the analysis step. The PVC gauze is therefore less suitable for sampling gaseous scents than any of the other filters tested (however, see below for sampling of dust).

Storage of the Filter Samples

Obviously, storage conditions must be such that no scent is lost from the filter. Unfortunately, the plastic explosive (PE) containers currently used have a higher sorption capacity than any of the filters. Thus, there will be a redistribution of the scent from the filter to the container during storage. This redistribution is unfavorable because the container will not readily release much of these scent molecules in the analysis procedure. Wrapping the filters in aluminum foil would help to minimize this problem. Biodegradation should not be a problem as long as the filters are kept dry.

Even if no scent is lost from the filter, storage time may still have an impact on the result: If a concentration peak is trapped on the filter during sampling, it may be confined to a certain section of the filter, depending on the aerodynamic properties of the filter. It may therefore be necessary to expose the complete filter (e.g., by cutting it open) to the sniffing animals. During storage, the scent will start to spread over the whole filter material due to diffusion. The velocity of this process highly depends on the type of filter that is analyzed. The same amount of trapped scent will produce a higher air concentration in the analysis procedure the more concentrated it is on a particular filter section, and it will produce lower air concentrations the more it is spread over the whole filter material. Above calculations for the selection of an optimal filter were based on the conservative assumption that the scent molecules are evenly distributed on the filter.

Sampling of Soil and Dust Particles

A REST procedure that would collect soil and dust particles with sorbed scent would always give better results (i.e., higher air concentration in the analysis procedure) than any vapor sampling could. In addition, it would perform equally well for all tracer scents while vapor sampling can only be optimized for a single vapor at a time. A derivation of these statements is not given here but follows the same lines as the above analysis for vapor collection on filters.

In fact, during a recent REST workshop organized by APOPO in Morogoro, Tanzania (July 27–30, 2004), most participants agreed that the good field performance of the PVC gauze as a filter in REST is probably due to the effective sampling of soil and dust particles (note again that the lab analysis had shown the PVC gauze not to be a suitable filter for sampling the gas phase for explosive vapors). ♦

See "References and Endnotes" on page 109

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