**Introduction**

Activated carbon filters in respiratory protection devices form a reliable means of protection against toxic gases when used under proper conditions. Because their sorption capacity is limited, the time that filters can be used is restricted. Many factors influence the time of use, which complicates the estimation of this time beforehand. It is, therefore, extremely useful for the wearer of a mask with an activated carbon filter, to have a detection method to warn for the imminent exhaustion of the filter. At TNO Prins Maurits Laboratory a new approach is under development to realize such a detection method, which is known as an End of Service Life Indicator or ESLI (US patented [1]).

**Principle**

The principle of the End of Service Life Indicator is the detection of the release of a pre-adsorbed signal compound from a special adsorbent as a result of the adsorption of a toxic (e.g. a chemical warfare agent) or any other atmospheric contaminant, see Figure 1. This adsorbent (probably a very special activated carbon) is integrated into the normal filter near the air outlet and will be exposed to the agent/contaminant once the capacity of the filter in front of it has been depleted. The ‘adsorbent loaded with the signal compound’ (plug) may be at different positions in the filter bed, dependent on the required sensitivity. The agent/contaminant adsorbs on the plug, which causes the signal compound to desorb. The signal compound must be released in a sufficient concentration for observation either by the human nose or by an instrumental detector. The use of an instrumental detector has the advantage of being independent of the subjective observation by the wearer of the mask.

A number of variables come into play in realizing the principle of the ESLI, amongst others: the selection of the adsorbent and its dimensions, its position in the activated carbon filter, the selection and load of the signal compound, and the development of a suitable detector. In this work the attention is directed to the development of the combination of adsorbent and signal compound.

**Experimental**

In a first series of experiments several adsorbents were tested with the same signal compound. Two activated carbons, one consisting of cylindrical extrudates (ELC)
the other of spherical particles (Kureha), and two activated carbon cloths (ACC) were examined. The selected signal compound was tetrahydrothiofene (THT). Experiments were carried out in a microbalance using about 20 mg of adsorbent. The adsorbents were loaded with approximately 0.1 g THT/g carbon. First the influence of the temperature on the desorption behavior of THT was investigated. Under a continuous flow of helium the temperature was increased stepwise with 25°C, starting at 25°C up to 125°C. Both the mass of the adsorbent and the concentration THT in the effluent were followed in time. Second the desorption of THT was followed at constant temperature (25°C) while a helium flow was passed by that contained toluene, cyclohexane or butanol.

In a second series of experiments various organics were tested as signal compound on two activated carbon cloths. The tested compounds were benzaldehyde, 1-pentanol, tetrahydrofurane, toluene and xylene. Again the influence of the temperature on the desorption behavior of the signal compound was investigated.

Finally, a series of breakthrough experiments were performed in which several activated carbon cloths loaded with THT (~0.05 g THT/g carbon) were exposed to a flow of air at various relative humidities. The bed length was equal to the thickness of the cloth, typically between 0.5 and 1.0 mm, the bed diameter was 5 cm, and the linear velocity was 6 cm/s. The relative humidity was varied between 0 and 90%.

Results

Figure 2 shows the influence of the temperature on the desorption behavior of THT on three adsorbents. The Kureha carbon starts to desorb THT at a lower temperature than the two other adsorbents. The ACC-1 performs slightly better than ELC.

![Figure 2: Influence of the temperature on the desorption behavior of THT on three adsorbents.](image)

Figure 3 shows the desorption of THT at constant temperature (25°C) while a helium flow was passed through that contained toluene or butanol. Clearly, THT is released in all cases. Toluene as well as butanol desorbs THT at a higher rate from the ACC-1 adsorbent than from the two other adsorbents. THT is released much slower and at lower peak concentrations from the Kureha and ELC carbon than it is from the ACC-1 carbon.

The results so far indicated that activated carbon cloth adsorbents are more promising than particle based carbons for the application as carrier of a signal compound. Both the temperature stability and the desorption characteristics were best for the activated carbon cloth. Because the signal compound is an interesting and important factor of the ESLI-principle a series of experiments were performed with various organic compounds on two activated carbon cloths. Only the results of 1-pentanol are shown here. Figure 4 shows the influence of the temperature on the desorption behavior of 1-pentanol. The initial decrease in mass is caused by desorption of water that was present on the sample in a small amount. The ACC-2 sample starts to release 1-pentanol at 100°C whereas the ACC-1 sample starts to release already at 75°C and at a much higher rate. Although the amount of pre-adsorbed 1-pentanol was not the same on the two cloths, 0.153 g/g on ACC-1 and
0.096 g/g on ACC-2, the observed difference in behavior is too large to be solely caused by this fact. Apparently, ACC-2 performs even better than ACC-1 in case 1-pentanol is the signal compound (at least in so far the temperature dependence is concerned).

**Figure 4:** Influence of the temperature on the desorption behavior of 1-pentanol on two types of ACC.

Breakthrough experiments were performed in which several activated carbon cloths loaded with THT were exposed to a flow of air at various relative humidities. The results are shown in Figure 5. Desorption from the materials NBC and Rus is less influenced by water vapor, while these two materials release THT at a significantly lower concentration. Because water vapor is always present in the air, the extent at which water desorbs the signal compound from the special adsorbent is a very important design factor. Therefore, an adsorbent must be found or developed that is very hydrophobic, preferentially even more hydrophobic than the materials tested so far.

**Figure 5:** Influence of relative humidity on the desorption of THT from several activated carbon cloths.

**Conclusion**

The ESLI warns the wearer of the respirator that the filter element needs to be replaced. The principle of the ESLI is the detection of the release of a pre-adsorbed signal compound from a special adsorbent as a result of the adsorption of the toxic agent or any other atmospheric contaminant.

A number of variables come into play in realizing the principle of the ESLI. The attention has been directed to the development of the combination of adsorbent and signal compound. Important characteristics are: the signal compound must remain adsorbed while an air flow is passed through, have a good temperature stability, and be able to withstand high relative humidities. Generally, activated carbon cloth materials showed a better temperature stability and better desorption characteristics compared to particle based carbons. The various demands make the development of a suitable plug a complicated task, while a number of design factors can be varied to reach a set of optimal conditions.

The results so far illustrate the potential of the underlying principle as a method to achieve an End of Service Life Indicator. Recently, the attention has been directed to the development of highly hydrophobic materials. In the presentation the principle will be explained and demonstrated on the basis of the latest experimental results.

**References**