

New method to measure stored VOC in concrete

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ABSTRACT

This paper describes a new technique to investigate the presence of stored VOCs in building materials. The method is based on a device for headspace measurement with GC-FID analysis. The technique has been successfully used in both laboratory and in situ measurements.

The paper describes an in situ measurement using this method. The measurement illustrates the distribution profile of stored degradation products in the concrete of a floor construction. From the measured values, properties that can describe the ongoing transport processes may be evaluated. In this investigation the effective diffusion coefficient for n-butanol in concrete is obtained.

INDEX TERMS

Concrete floor, contamination distribution, diffusion properties, measurement technique, VOCs.

INTRODUCTION

One of the most important sources of enhanced VOC in the Nordic building stock are moisture-damaged floorings (Kumlin, Åkerlind, Hall *et al* 1994, Hall 2001, Engström and Sjöberg 2002). In several Scandinavian investigations of SBS the floor construction has been pinpointed as the source of enhanced VOC in indoor air. VOC that are formed below the flooring during alkaline hydrolysis of flooring adhesive may be emitted into indoor air and migrate into the sub floor (Sjöberg 1999, 2000, 2001). Degradation products that migrate down into the sub floor may be stored in the concrete slab of a floor construction for a long time and may eventually be emitted into the indoor air.

It is therefore important to have the equipments and methods to measure the amount of degradation products that have migrated down into the concrete slab and are now stored there. In this paper a new method for measuring the presence of stored decomposition products and other VOCs in concrete is described. On the basis of the measured values it is possible to predict how the stored chemical substances may affect the indoor air if different renovation measures are carried out. One example of the distribution profile of n-butanol measured in situ during a SBS investigation is described and analysed in the paper.

A similar method for laboratory purposes is described by Hjellström, Wadsö and Kristensson (2002). That method is however designed to measure the total amount of a chemical substance in concrete while the method described in this paper is designed to measure the free concentration in the air in the pores of the concrete, compare headspace. According to Sjöberg (2001), the storage capacity is given by the relationship between these quantities.

METHOD

The method developed in this project is a measurement of the headspace in a glass bottle where a concrete sample is placed. The method is suitable for measuring the concentration of VOC deposited in concrete both in laboratory studies and in situ in a floor construction in a building with SBS.

The VOC stored in concrete is measured in four steps. 1) The first step is to retrieve concrete samples from the floor construction, seal them up and transport them to a laboratory. 2) The next step is to place fractions of the sample in bottles and wait for the VOC in the concrete to attain equilibrium with the bottle air. 3) The third step is to take an air sample from the bottle through an adsorbent, preferably a TENAX tube. 4) The last step is to analyse the TENAX tube by gas chromatography (GC-FID).

The first step may be performed in situ in the floor construction of a building or on a specimen in a laboratory study. Steps two to four are performed in a laboratory with proper equipment and knowledge of the method used.

SAMPLING EQUIPMENT

The equipment for headspace measurements is based on a 250 ml glass bottle with a wide neck ($\text{Ø}_{\text{int}} = 30 \text{ mm}$); Figure 1. The screw cap of the bottle has an internal teflon gasket to ensure that no VOC can be emitted or taken up through the cover. In the screw cap there are stainless steel fittings for tubes; these enable controlled replacement of air in the bottle at the time of sampling. Sampling of the air in the bottle is performed after the VOC in the concrete has attained equilibrium with the bottle air. The conditioning to equilibrium is assumed to take about 72 – 120 hours at room temperature; the temperature should not be increased.

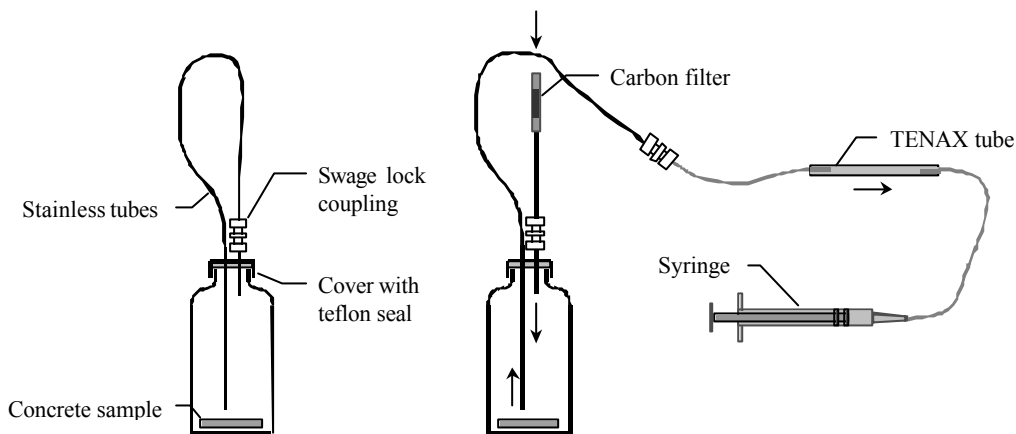


Figure 1. Bottle during conditioning and sampling.

During sampling a 20 ml plastic syringe is used to draw the correct volume of air through the TENAX tube. The different tubes and the syringe may be connected with teflon hoses, using Swage lock type couplings or silicon hose.

MEASUREMENT

The measurement performed in this investigation took place in a 0,57 m thick haunch at the edge of the concrete slab in a 20 year old building. It was suspected that the flooring adhesive that attached a PVC flooring to the top of the concrete had been chemically decomposed by the moist concrete. In that case decomposition products may have migrated into the concrete. The building investigation is carefully described by Engström and Sjöberg (2003)

The sample was extracted by dry core drilling, i.e. no water was used to cool down the core drill. If cooling water is used some water soluble organic compound may be washed away. It is best to drill a few cm at a time to allow the drill to cool down during the cooling time an approx. 1 cm thick layer of concrete was removed from the core with hammer and chisel. A couple of concrete pieces from each layer were sealed in separate bottles or in several layers of aluminium foil directly after they had been removed. Each bottle contains about 20- 50 g concrete.

The samples were quickly transported to the laboratory under thermally stable conditions. Under no circumstances must the seal on the sample be broken during transport. If condensation is visible on the glass bottle this must be taken into consideration when evaluating the results. The concentration in the headspace may be higher due to evaporation from the concrete and hysteresis effects if condensation has taken place in the bottle.

After the bottles had reached the laboratory they have to be conditioned and stabilised for 72 – 120 hours at room temperature. The intention is to enable the VOC in the concrete sample to attain equilibrium with the air in the bottle at room temperature. It is therefore important not to raise the temperature of the sample before or during the conditioning period or at the time the bottle air is sampled.

When the air is sampled the Swage lock coupling on the screw cap is opened and a carbon filter is attached to the air inlet tube. To the other tube a TENAX tube for air sampling is connected. During sampling a 20 ml plastic syringe is used to slowly draw a 20 ml volume of air through the TENAX tube. The TENAX tube is then analysed by gas chromatography (GC-FID) according to the procedure in Sjöberg (2001).

MEASUREMENT UNCERTAINTY

No complete validation of the uncertainty of the methods has been performed so far. In Sjöberg (2001) the total standardised measurement uncertainty for the method was assessed at $<\pm 20\%$. Based on Sjöberg (2001) and the writer's unpublished experience the total uncertainty may be estimated to be around 20 – 30% including the GC-FID analysis.

RESULTS

The results of the situ measurements of the concentration of one chemical compound that arises from degradation of floor adhesive in a moisture damaged floor construction is summarised in Table 1.

Table 1. Values of the stored decomposition product n-butanol measured at different depths in the haunch of a moisture damaged concrete slab.

Level No:	Depth [mm]	n-butanol [$\mu\text{g}/\text{m}^3$]
1	0-10	14 500
2	20-30	8 900
3	40-50	1 900

Note that the concentration nearest the surface is much higher than deeper down in the concrete. This behaviour is often seen in these measurements and indicates that the sources of the chemical compounds are at top of the concrete. The source is described in Sjöberg (2001)

to be a degradation of the adhesive; degradation products may migrate deep down into the concrete and form the type of distribution profile shown in figure 2. The “error bars” at the measured values in the figure indicate the span of the depth of the sample.

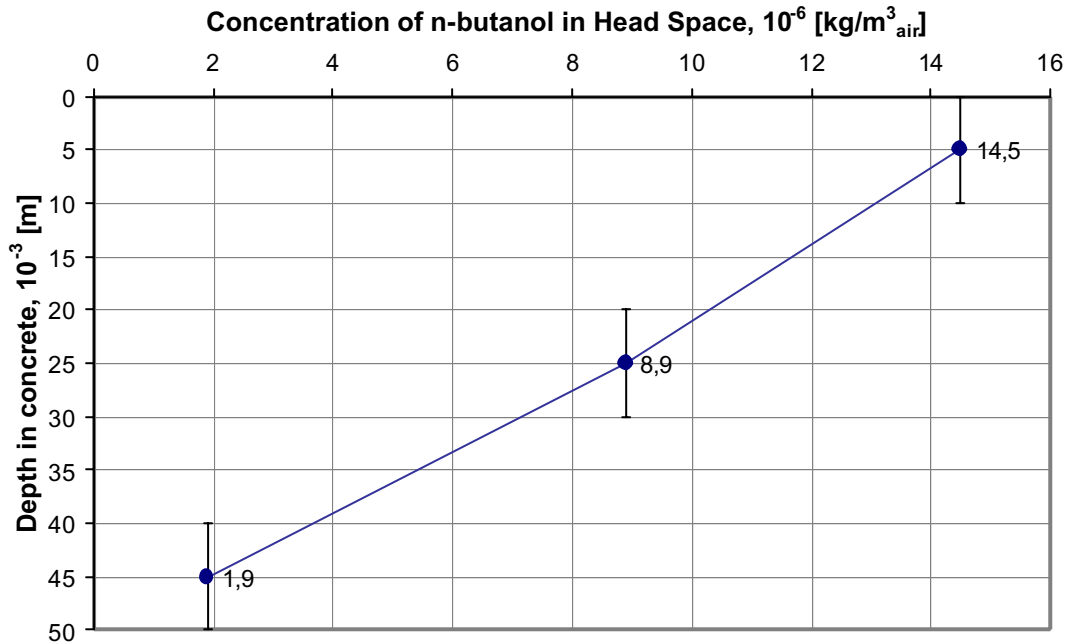


Figure 2. Distribution profile of n-butanol in the concrete slab of a moisture damaged floor construction.

EVALUATION

The distribution profile of decomposition products in the concrete slab below the flooring shows that n-butanol from decomposition of the adhesive had penetrated deep into the concrete. According to Crank (1975), the rate of the transportation process can be described with reference to a few properties. One of these is the effective diffusion coefficient D_{eff} that can be evaluated with reference to Fick's second law. For such an evaluation it is only necessary to know the concentration of the penetration profile for a chemical compound which was formed during incremental changes in a semi-infinite medium.

With the simplification that the surface concentration c_s in the test specimen had changed in stages from the initial value 0 to a constant value which then remained unchanged during the whole period, the fundamental incremental change requirement is satisfied. In view of the fact that the concentration is very low at 40 – 50 mm depth in the concrete, the 570 mm thick slab may be considered infinite in this respect.

Fick's second law can then be solved with Equation 1 according to Crank (1975); this is the complement to the "error function".

$$c(x, t) = c_s \cdot \left[1 - erf \left(\frac{x}{2 \cdot \sqrt{D_{eff} \cdot t}} \right) \right] \quad [kg/m^3_{air}] \quad (1)$$

where c is the concentration in the air in the pores of the concrete (headspace) at depth x at time t . c_s is the “ headspace” concentration at the surface and D_{eff} is the effective diffusion coefficient.

In Fig. 3, the complement to the error function has been calculated for a number of different combinations of $D_{eff} \cdot t$. The profiles for n-butanol have been calculated on the assumption that the surface concentration has been $17 \cdot 10^6 \text{ kg/m}^3$. The “error bars” at the measured values indicate the span of the depth of the sample.

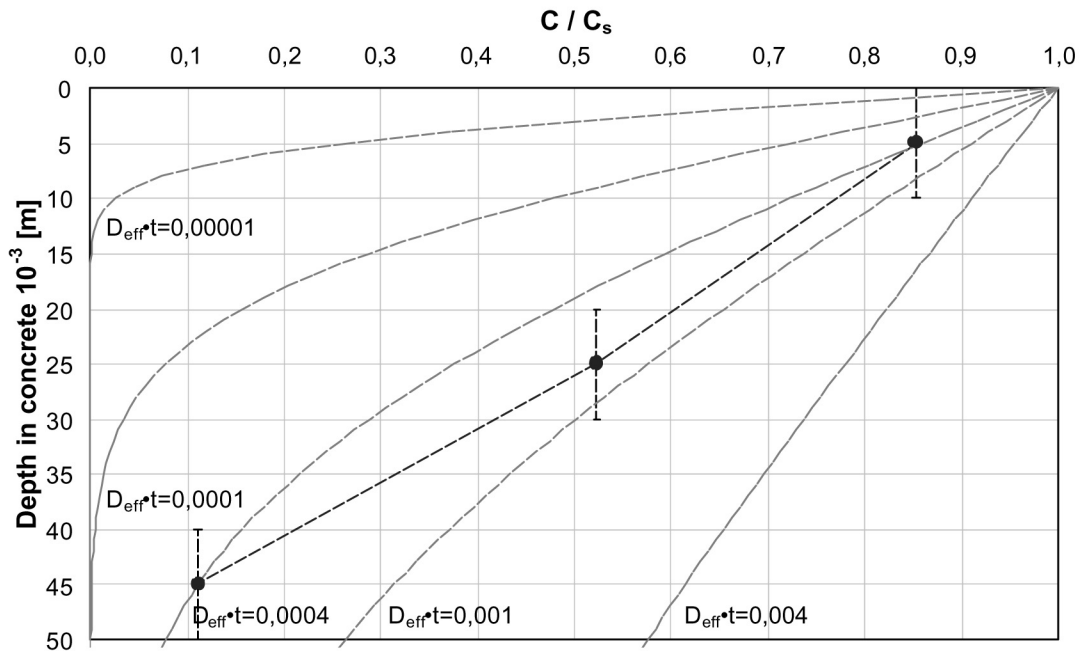


Figure 3. Measured values compared with the solution of Fick's second law. The figures along the curves are the values of $D_{eff} \cdot t$; see Equation 1.

DISCUSSION

The distribution profile for n-butanol in this measurement shows good, but not exact, agreement with Fickian behaviour, the “error function”. The deviation may be due to many different reasons which can be mainly divided into three groups: A) The measured values are not correct; the large uncertainty in the method supports this theory. B) The Fickian theory is not good enough in this case, the transport mechanism is governed not only by Fickian behaviour. C) The circumstances are not those assumed, e.g. the surface concentration has not been stable at the same value for the whole period.

Some unpublished experience states that measurements at several depths at the same place lower the uncertainty of the method. This is because the values next to each other are verifying each other. The shape of the distribution profile may not deviate from the expected “error function” without a reason.

The measurements in this method should only be performed in a laboratory that is used to this kind of headspace measurement at room temperature and has experience of this. Different laboratories tend to use slightly different variants of the method according to their equipment and earlier experiences. It can therefore be difficult to compare the results from different laboratories. Standardisation of this method may alleviate this problem.

CONCLUSION AND IMPLICATIONS

The method has proved to be useful to practitioners when investigating whether the adhesive had decomposed when the floor construction is assumed to be the source of enhanced VOCs in a SBS object. The method however needs to be developed and improved to lower the uncertainty that now appears to be rather large.

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