Application Note No. 052

Determination of Breakthrough Volume for Optic Liners

When analysing very low level samples it is necessary to trap analytes from large volumes of sample. Under these conditions knowledge of the breakthrough volume of an analyte on the trap is important if confidence in the analytical results is to be maintained.

When trapping analytes on a packed Optic liner, the packed liner can be considered as a very short packed column so under any conditions of temperature and flow, an analyte will have a finite (but hopefully very large) retention time. By knowing the sample flow rate and the retention time of the analyte under consideration, the breakthrough volume can easily be calculated.

The retention time of an analyte can be determined in one of two ways, either method can give valid results so long as some simple precautions are taken. Neither method is best and tends to be judged on a case-by-case basis. However, the first method gives good results if the Optic liner has been packed with materials that can be considered as traditional column packing, such as Tenax or Porapak. Examples of both techniques are shown.

Method 1

The first method uses the packed Optic liner as a real packed column, the outlet of the injector being connected to the detector via a short length of uncoated fused silica transfer line. The carrier gas flow rate is set to a high value 40-60cm³/min. The column oven temperature is set to a value between 50-100°C. Samples of the analyte in question are injected into the top of the Optic and the time taken for the analyte to start eluting from the Optic are taken at a range of Optic temperatures. The breakthrough volume at any temperature of interest is determined graphically by plotting log (Retention Volume) Vs Optic temperature and extrapolating to the temperature of interest.
The following points must be borne in mind when using the technique:

i. When making the injection, the syringe needle must not penetrate the trap bed otherwise a falsely low breakthrough volume will be obtained.

ii. The capacity of the trap must not be exceeded otherwise it will behave as a short packed column.

iii. The carrier flow rate will vary with Optic temperature and it must be accurately measured at each Optic temperature used and incorporated into the calculation of retention volume.

This method has the potential to give results more quickly than the second method but has two associated problems. Firstly it requires the column configuration to be changed from the normal analytical configuration and secondly to be able to accurately determine when the analyte is first detected, particularly at (relatively) low Optic temperatures where analyte peaks are very broad.

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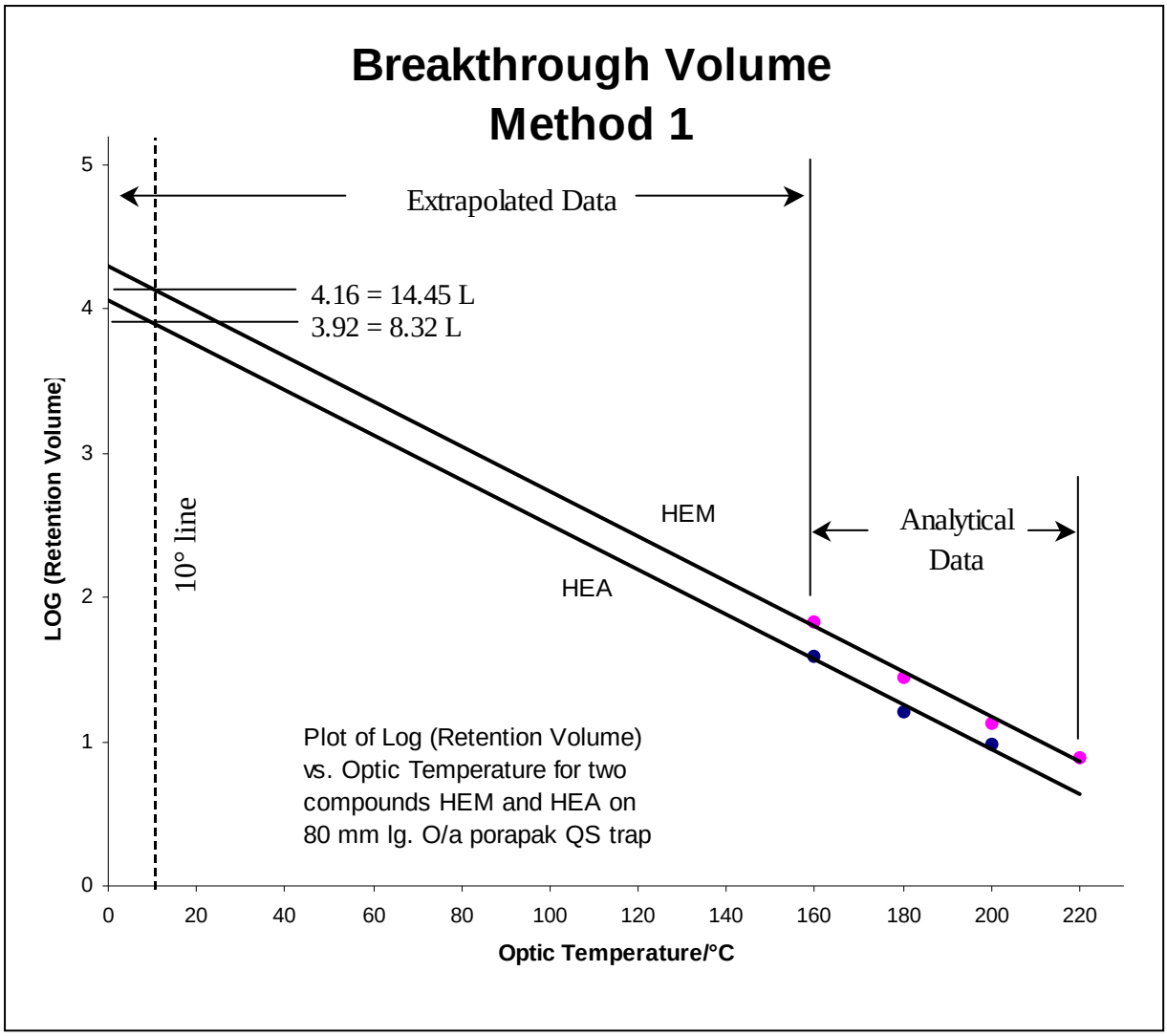

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Method 2

The second method is more consuming but does not require the column configuration to be changed.

The technique relies on trapping sample for longer and longer times and plotting analyte peak area vs. volume of sample passed through or exposure time at a constant pump rate for dynamic sampling. A straight-line relationship should be obtained at sample volumes or exposure time below breakthrough. At sample volumes or exposure times above breakthrough the peak areas obtained settle to constant values as analyte input at the trap inlet is balanced by analyte loss at the trap outlet. The intersection of the two lines so obtained gives the breakthrough volume at that trap temperature.

The following points must be borne in mind:

i. Analyte concentration in the sample used must be sufficiently low that the trap capacity is not exceeded at sample volumes below true breakthrough.

ii. It must be necessary to decrease amplifier sensitivity at large sample volumes to prevent the analyte peaks becoming flat topped and giving falsely low peak areas.

This method only gives an analyte breakthrough volume at one trapping temperature and must be repeated if other trapping temperatures are contemplated.

It is usual to perform these measurements using a single component in nitrogen test sample. That component usually being the first to elute in the chromatogram obtained from the test mixture under consideration.

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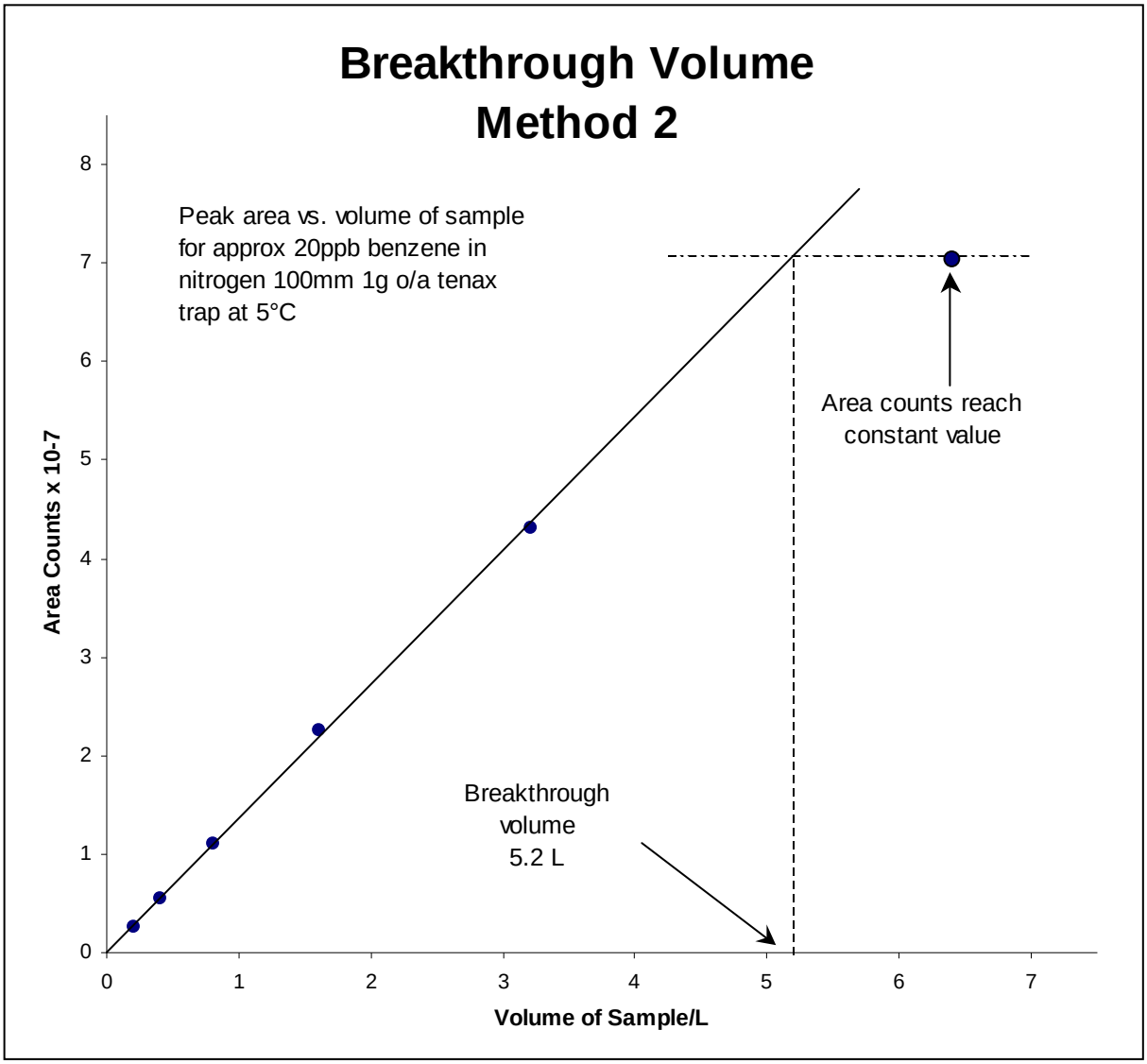

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