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Hypothetical Means to Improve GC Column Efficiency.

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ABSTRACT

For open-tubular, capillary gas chromatography (GC), the linear carrier gas velocity is a crucial parameter for determining the performance of the system with respect to resolution. However, due to the compressibility of the carrier gas and the pressure differential from the head of the column to the column exit, the linear gas velocity is non-uniform across the length of the column leading to a loss of efficiency. To alleviate this, it is considered whether a column of varying diameter could potentially yield a more uniform linear gas velocity to enhance the performance of the system.

Keywords: capillary gas chromatography, column efficiency, chemical analysis, carrier gas velocity

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INTRODUCTION

Open-tubular, capillary gas chromatography (GC) is an indispensable analytical tool that has evolved remarkably to the stage now where the performance is phenomenal in terms of its ability to resolve compounds and the technique is now considered an indispensible tool for numerous applications, particularly when in combination with mass spectrometry (MS) for sensitivity of detection and unique identification of molecular characteristics [1]. There have been many milestone improvements for the GC system itself in chromatographic terms, notably the development of open-tubular, capillary columns and temperature programming amongst many others [1, 2]. However, the insatiable need for ever greater resolution by chromatographic practitioners means that the evolution and developmental process will forever be an ongoing one. At the physical level, the chromatographic performance of the system is dependent on many factors such as adsorption, diffusion, linear carrier gas flow, etc. [2]. However, because of the never ending demand for greater and greater performance, note only for the separation of analytes per se but to also to elevate the sensitivity through narrower eluting peaks, the study of the physical processes involved in GC still continues despite the current depth of understanding, and although the primary focus generally is on the study of the physical processes themselves or the study of particular systems, the general application of these results for the improvement of GC methodology is a strong motivation [3]. Many attempts have previously been made to address the limitations of the physical peculiarities, for example pressure programming, which has had a mixed acceptance by GC practitioners in comparison to the well-entrenched temperature-programmed systems [4].

For capillary GC, the linear carrier gas velocity is crucial for determining the performance of the system, but due to the compressibility of the carrier gas and the pressure differential from the head of the column to the column exit the linear gas velocity is non-uniform across the length of the column, thereby leading to a loss of efficiency. To alleviate this, it is considered whether a column of varying diameter could potentially yield a more uniform linear gas velocity to enhance the performance of a given system.

DISCUSSION

The fundamental performance metric of a chromatographic system is the number of theoretical plates, or the height equivalent of a theoretical plate (HETP) to enable quantitative comparison of different systems independent of the column length [2, 5]. Plots of HETP versus the average linear carrier gas velocity for GC systems (Van Deemter plots) readily espouse the performance of various systems with regard to temperature (isothermal conditions), carrier gas type, column inner diameter (id) etc. However, it is the average linear gas velocity that is measured and used for the construction of these plots. But whilst it is readily apparent that the system performance will vary with the mean rate of carrier gas flow, which is a trivial measurement to make in practice, what is not generally well appreciated for practical purposes is that the carrier gas velocity is not constant and varies dramatically in concert with the pressure differential across the length of the column due to the compressibility of the carrier gas. The introduction of pressure programming was an attempt to address the problem of pressure changes caused, in part, by the use of temperature programming [4] which led to a perturbation of the average linear gas velocity

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from the ideal value which had been measured and set by the GC practitioner. Nonetheless, despite temperature programming and pressure programming there are still limitations on column efficiency due to the physical properties of the system and carrier gas.

For example, if one considers a normal open-tubular column as depicted in **Fig. 1a**, at the head of the column the high pressure present at that point compresses the carrier gas and as a consequence the initial linear gas velocity is below that of the optimum, mean, value for the particular carrier gas in use at the particular temperature in use. Towards the exit end of the column as the pressure nears to ambient pressure or the vacuum of an MS ionization source, the expansion of the carrier gas results in a linear gas velocity that is above the desired optimal value. So although the average linear carrier gas velocity over the entire length of the column may be equated to an ideal value, only in the mid sections of the column does it actually correspond to such a value wherein the imparted resolution is consequently also optimal. At either end of the column, however, the resolution being imparted to the analytes can be considerably less than optimal.

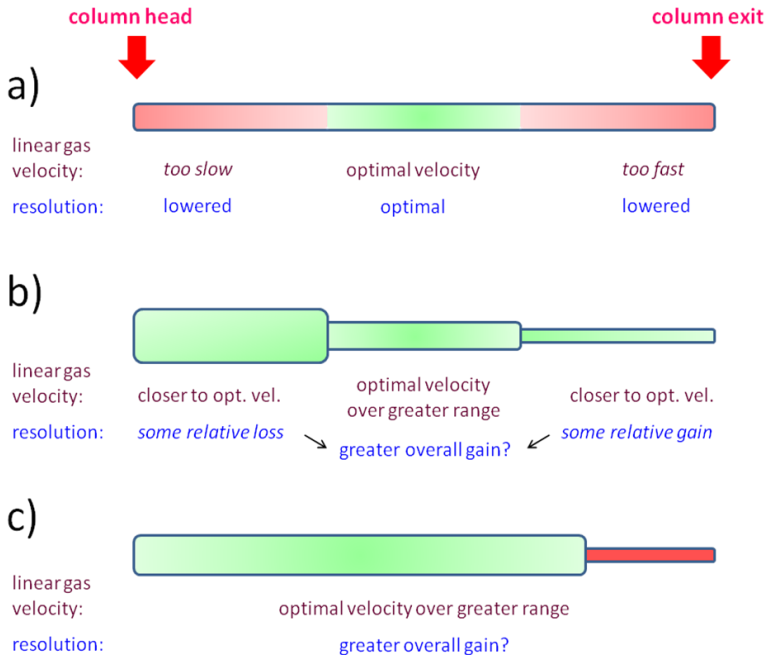

Fig. 1: Depiction of various column geometries and their purported characteristics in terms of the linear gas velocity at various points along the column and the likely resultant resolution at those points for: a) normal uniform id GC column; b) a system with a step-wise diminution in column id; and c) a normal column end-capped with a restrictive short segment.

Interestingly, there do not seem to be any clear reports on attempts to address the question of the non-uniformity of the carrier gas across the length of the column. One means to alleviate this would be to construct a column of decreasing id so that as the gas progresses along the column, the more restricted column limits the expansion of the carrier gas. However, such a column geometry may be challenging to manufacture. An alternative approach would be a step-wise change in id. This is a practical option due to the availability of column joiners normally used to repair broken columns or to assemble extra long or mixed-type columns, thus enabling columns of different id to be joined together. Thus in **Fig. 1b**, whilst the optimal linear carrier gas velocity is again generally attained in the midsection of the column system, a greater portion of the column system can be

maintained nearer this optimal value. Starting with a larger relative id column section may lead to some nominal reduction in the resolution for that part since resolution is related inversely to column id, but this will be balanced by the superior gas velocity for that section in comparison to a constant id column, and also by the greater resolution imparted by the narrower column segment at the end of the column system, particularly with a superior comparative gas velocity also for that segment.

Alternatively, rather than trying to effect a less dramatic pressure differential across the entire column length, the majority of the pressure drop could be shunted to a small part of the column system by employing a short end-column segment of much narrower id (**Fig. 1c**) resulting in a more uniform flow along the major column section. Sacrifice of resolution is made in the short column segment to attain a greater overall gain in the larger column segment.

These are conceptual ideas that we will be pursuing experimentally in our laboratory in the near future. It is hoped that some gains in performance will be made, and that these will not only be applicable to new GC systems but that they can be applied to already established systems.

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