



Editorial

Editorial on “The challenges of the analysis of basic compounds by high performance liquid chromatography: Some possible approaches for improved separations” by D.V. McCalley

HPLC columns have been continuously improved during the last forty years, both in terms of their kinetic performance based on the reduction of particle size of packing materials and also in their chemical properties. The so-called silanol effect on silica-based phases, however, has still been one of the major topics of chromatographic studies even after the development of advanced stationary phases that were supposed to provide symmetrical peaks for amines. The performance of an HPLC column for the separation of basic compounds is of much practical importance, because many pharmaceuticals possess basic functionalities.

Prof. David McCalley has been studying various aspects of silanol effects on the elution of basic compounds from stationary phases for reversed-phase HPLC since the early 1980s until today. In fact, his interest in chromatography began with gas rather than liquid phase separations, working with glass capillary columns, and their application to the analysis of sterols as pollution indicators in environmental samples. In those days, pyrex glass columns had to be drawn from a 1m length of thick-walled glass tube, deactivated and coated, all by the user, employing techniques that required some considerable patience and dexterity. He still retains an active interest in gas chromatography, and his research group was the first to identify (using GC-negative chemical ionization mass spectrometry) the presence of pollutant estrogens in the River Thames in 2001. These compounds have been shown to be responsible for sex changes and sterility in male fish. This work attracted much attention in the international news media such as the BBC and the Financial Times due to its perceived connections with human fertility [1].

He began his studies in HPLC with the analysis of alkaloids in the early 1980s, being intrigued as to why some columns yielded good peak shapes, while others showed broad tailing peaks or even irreversible adsorption of the same solutes. Since then, he has published more than thirty papers on subjects related to the secondary retention processes in reversed-phase HPLC and silanol effects. His interests included every aspect of these effects, including the influence of stationary phase structure as well as the probe compounds used, including base strength (pK_a values) and steric factors, the effects of elution conditions (type of organic modifier, its concentration, temperature, buffer type, pH and flow rate of mobile phase), the effect of sample mass and overloading on the appearance of the effects. His investigations concerning the overloading of ionized compounds have shown it to be a particularly nefarious problem that has not been solved by the introduction of the new Type B silica

stationary phases. The mechanism of overloading remains intriguing and is of much current interest, as evidenced by the dozens of recent publications by Prof. Georges Guiochon in the journal on this subject. He has also reported characterization methods for stationary phases and interpreted the results of these evaluations using chemometric procedures.

Recently Dr. McCalley has been working on characterization of a broader range of HPLC columns and chromatographic phenomena, including new types of chromatographic support (small-sized silica particles, hybrid materials, superficially porous particles, and monolithic columns), HILIC stationary phases, and retention processes in ultrahigh pressure LC. He feels privileged to have published some of this work in collaboration with a number of the most distinguished researchers in LC. His work is always related to the fundamentals of liquid chromatography, and is of much importance.

These are the reasons why the author has been invited to write this review article, and why the subject will be of much interest to Journal of Chromatography A readers. In this article he mainly addressed the issue of how to obtain optimum results in the separation of basic compounds. It will help readers interested in separation of basic substances, or pharmaceuticals in general. The readers will get both fundamental and advanced knowledge of the materials and conditions for the separation of basic compounds.

Dr. McCalley loves hot springs, particularly those in Japan where he can use some of his Japanese vocabulary talking with his neighbors in hot waters. He feels very fortunate in having been able to visit parts of the world through his work that he would never have seen otherwise, and has given invited lectures in the last year in places as diverse as London, Cork, Dresden, Marseille, Baltimore, and Kyoto. He must be a good traveler, having curiosity in every aspect of life. He is a good organist and pianist loving especially Bach and Art Tatum, and once considered music as a career. I hope readers will enjoy this article, and understand from it how to deal with the separation of basic substances. I also hope Dr. McCalley will continue to extend his work elucidating the fundamentals of chromatography in a practical sense.

Reference

- [1] <http://news.bbc.co.uk/1/hi/health/1495908.stm>.

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