Abstract for Fabrice Gritti Lecture

Achieving High-resolution Chromatography Under Extreme Operating Conditions : Vacuum-Jacketed Chromatographic Columns.

Under extreme operating conditions in either ultra-high pressure liquid chromatography (UHPLC, 1 kbar, 1 mL/min → eluent heating) or supercritical fluid chromatography (SFC, pure CO_2, temperatures > 50^oC, outlet pressures < 100 bar → eluent cooling), thermal effects take place during the steady-state decompression of the mobile phase and deteriorate the resolution power of the column. This is due to the circulation of a steady radial heat flux from the column wall (stainless steel) to the external environment (lab air or oven compartment) and the subsequent presence of radial temperature gradients across the bed, which are distorting the chromatographic bands.

In this presentation, a new technology is proposed, validated on both fundamental and experimental grounds, and applied for the development and final design of new user-friendly column hardware to combat against these performance losses. The technology is based on high-vacuum ($P_{air}=10^{-5}$ Torr) and low-emissivity materials (ex: aluminum) which sets the chromatographic column under quasi-adiabatic conditions.

From a fundamental viewpoint, heat transport models predicted that the amplitude of the temperature difference between the center and the wall of the bed could be reduced by a factor as large as 30. In theory, the maximum resolution power can then be achieved due to the existence of nearly uniform flow and retention profiles across the column diameter under such adiabatic conditions.

Experimentally, a large vacuum-oven was first built to reveal experimentally that the standard efficiency of a UHPLC column packed with sub-2 μm particles and fully embedded in this adiabatic oven may increase by +35% for a heat power released of 12 Watt/m (900 bar, 0.8 mL/min, 10 cm long column). Additionally, the same vacuum-based oven was used to maintain the quasi-Gaussian peak shape of volatile compounds in SFC when using pure CO_2 above 100 ^oC inlet temperature and at 100 bar outlet pressure. Secondly, a series of user-friendly vacuum-jacketed column hardware were designed to achieve the same column performance as those observed with the large adiabatic vacuum-oven. Experimental results are presented to show that UHPLC columns equipped with this new column hardware and run under elevated heat power (12 Watt/m) retain efficiencies within 5% of their maximum theoretical efficiency expected under isothermal conditions. Finally, applications are shown for the fast UHPLC analysis of enolase digest and the SFC separation of volatile compounds such as terpenes in herbs and alkanes in gasoline samples.

CV, Fabrice Gritti

Fabrice G. Gritti received a B.S. degree in Chemistry and Physics from the University Joseph Fourier of Grenoble (France) in 1995, a graduate Engineering school degree in Chemistry and Physics from the University of Bordeaux I (France) in 1997, and a Ph.D. in Chemistry and Physics from the University of Bordeaux I (France) in 2001. He came in the U.S. in 2002 for a post-doctoral visit at the University of Tennessee in the research group of Prof. Georges Guiochon. He worked there as a Research Scientist in the Department of Chemistry until 2014. In 2015, he joined Waters Corporation as a principal research scientist in the Instrument/Core research/Fundamental department.

Dr. Gritti's research interests involve liquid/solid adsorption thermodynamics and mass transfer in heterogeneous media for characterization and design optimization of new HPLC column/instrument technologies. He developed experimental protocols that helped refine (1) the models of adsorption isotherms used in preparative chromatography for the prediction of the band profiles of neutral and ionizable compounds and (2) the theory of band broadening along modern analytical columns in linear chromatography. Dr. Gritti has been invited to give 25 seminars, workshops, or tutorials on general chromatographic sciences at universities, discussion groups, or at international meetings. He has delivered 35 invited keynote lectures at international Symposia and published over 250 peer-reviewed scientific papers. Dr. Gritti was the recipient of the 2013 Chromatographic Society Jubilee Medal for his important contribution to the development of chromatographic science.