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Chapter 1 : Electrochromatography - Wikipedia

Capillary Electrochromatography Supports and stationary phases for capillary electrochromatography. Keith D. Bartle, Peter Myers and Peter Myers Peter Myers and.

Packing methods for the preparation of CEC columns have been investigated. The problems inherent in the use of burned-in frits remains an obstacle, but can be at least partially overcome by minimising the length and by silanisation. The influence of a variety of mobile phase variables on aspects of CEC is in agreement with theory for: Temperature can be used as a variable to change column selectivity in CEC. The influence of pH on electroosmotic flow EOF by changing the degree of ionisation of residual silanol groups is similar for a wide range of neutral bonded groups, but is much less marked for bonded sulphonic acid groups. The EOF may be reversed for bonded groups containing nitrogen. Electrochromatography; Silica columns; Reviews; Capillary columns; Stationary phases, electrochromatography Contents 1. Packing columns with silica particles Influence of properties of the silica based stationary phase on CEC Particle size and porosity Nature of the silica-bonded stationary phase Influence of temperature on CEC on silica bonded columns Practical CEC separations on silica based columns S 00 K. A â€" 1. Introduction reasons are as follows: The separation mechanism is, therefore, a understood, and the direct transfer of HPLC methods combination of differential electrical migration of to CEC versions with improved resolution is, there- charged species, and partition between mobile and fore, most likely to employ silica based columns; stationary phases. Application alkoxylsilane derivatives are reacted with surface of a large voltage across the column generates silanol groups [9]. A number of advantages arise from electrod- rive, especially in increased column efficiency. Superposition of electromigration on partition should 2. Discussion also lead to versatile separation methods. A very wide variety of approaches is currently 2. Packing columns with silica particles being made to the fabrication and technology of columns for CEC. Electrokinetic [10] and duced, filled with polyacrylamide gels [3,4]. Such centrifugal [11] packing have been proposed, al- columns have been used in numerous application though the use of liquid slurries with ultrasonication areas, and have been shown to be highly efficient. In has been most common. Our previous work on the a second approach, a solâ€"gel process is employed to preparation of columns for micro-HPLC suggested form a silica xerogel [5] within the capillary, fol- [12] that packing with a supercritical fluid carrier and lowed by bonding of the stationary phase group; ultrasonication produced columns with very high alternatively, the separation medium itself may be efficiencies. Accordingly, 50 and mm I. Open-tubular columns in which umns for our early CEC work [13] were prepared in the wall is coated with the stationary phase, either an analogous fashion and were also found to be physically or by bonding through chemical deri- highly efficient, with plate numbers up to vatisation, have attracted attention [6,7]. A large Clearly, the above approaches are all likely to lead number of 50 mm I. At Leeds, fluid carbon dioxide or liquid carrier, each with and however, we have chosen to work with bonded silica without the use of an ultrasonic probe. The resulting columns, at least in what are still the early stages of columns were evaluated by measuring the electro- progress of a new technique, in which understanding osmotic flow EOF , migration time, column ef- of many of the basic processes is still sought. Our ficiency and retention factors for constituents of a K. The results Table 1 of this section, but at intermediate 7. It follows that, at pH significant differences between the properties of extremes, the EOF linear velocity decreases with the columns packed by different methods or in the length of the packed bed, while at pH values most success rate of preparation. Substantial contributions to originate in random variations between replicate the EOF from the capillary wall are evident. Problems with frits packed when applying ultrasonication with a liquid slurry. Although reproducibility is clearly a problem Bubble formation in CEC has been addressed in a during column preparation, CEC is highly repeatable number of ways. The original suspicion, that bubble Table 2 on an individual column. In an attempt to determine the role of the of. In fact, if heating effects are excluded, packed-bed length, the column was considered as bubble formation almost always arises at the inter- two

resistors in series, corresponding to the packed face between packed and unpacked sections of the and open sections [15]. The results formation of bubbles. A â€" Fig. Electropherograms showing how the formation of bubbles is affected by the length of the frit and the applied voltage. However, the problem can carbon containing groups which results from the high be tackled at source by: The bonded groups are easily restored sleeve a piece of empty capillary with a length of to the silica frit by treatment with, for example, polyimide removed to form a detector window; and chlorodimethyloctadecylsilane, thus minimising the K. Electropherograms showing how the recoating of stationary phase after formation of a frit can reduce the formation of bubbles. The bead was trapped bubble formation Fig. In our capillary to fill the tapered section of the glass tube approach [18] tubes approximately 40 mm in length until the blocking bead was reached Fig. The two mm access holes formed a such fluctuations resulted in a large degree of band tight fitting sleeve for fused-silica capillaries. Such a dramatic reduction in capillary single mm silica bead was inserted into the diameter can be avoided, however, by locating a tapered glass tube prior to the fitting of a packed and single mm diameter silica particle between two Fig. Schematic showing how the capillary, bead and glass taper were combined to make a frit. If a packed section of capillary of I. This method results in an improved column ef- 3 mm Spherisorb ODSl â€" [19] 3 mm Nucleosil C 18 91 â€" [20] ficiency, although not yet as consistently high as that 3 mm Spherisorb C 18 PAH Up to [13] seen for capillaries containing burnt-in frits. Influence of properties of the silica based 3 mm CEC Hypersil â€" [23] stationary phase on CEC used by manufacturers to produce different particle The majority of CEC separations so far reported sizes. Table 3 compares tial improvement in column packing is all important results from the literature for the isocratic CEC and that significant problems are involved in packing separation of polycyclic aromatic hydrocarbons smaller particles as well as larger ones. Knox has the packing process is again probably important; also pointed out [24] that reduced plate heights, h, although all the packings are listed as 3 mm diam- below unity can be obtained in HPLC for larger eter, the particle size distribution is unlikely to be particles, whereas the practical minimum for 3 and 5 uniform, and this distribution will vary with the mm particles is nearer 2. Number, area or volume bonded silica showed that, while h was approximate- distribution may be used to characterise particles for ly 1. If number distributions are compared, because of less uniform packing. These conclusions fine material below 2 mm is evident in virtually all may have a significant influence on the results of nominal 3 mm and 5 mm materials, and this is both CEC experiments with small-diameter particles. The likely to cause difficulties with packing and to be early theoretical work of Knox and Grant showed difficult to remove via the usual air classification how very highly efficient CEC should be attainable Fig. Schematic showing the construction of a PTFE coupled bead fit. A â€" on sub-micron particles, but there have been conflict- ing reports on the practical applicability of small non-monodisperse ,2 mm particles in CEC. In our hands, the preparation of columns packed with such materials was largely unsuccessful, in spite of the use of ultrasonication. Agglomeration of packing material and discontinuities in the packed bed were evident, leading to poor column durability and performance, in stark contrast to results with 3 mm particles. Monodisperse stationary phases in the range 0. For example, 3 mm ODS1 par- ticles packed into 30 mm I. Mobile phase parameters The dependence of EOF mobility, m EOF , on mo- bile phase properties has been well explored theoret- ically, and confirmed in a series of experiments in our laboratory [28] on CEC columns packed with Fig. Separation of acetyl salicylic acid A and salicylic acid silica and ODS1 bonded silica. The well-known fall-off of m EOF with led off and began to drop for silica packings with decreasing pH consequent on the decrease in silanol pore size 8 nm while continuing to rise in CE. From theory, the double- was borne out for binary mixtures of acetone, layer thickness and hence zeta potential and m EOF are acetonitrile, methanol and 2-propanol with water; proportional to I The increase of m EOF with I While, for example, retention [29]. The most commonly used stationary phases for 6. Deviations from the unit gradient line may arise capillary electrochromatography CEC are silica from the choice of thiourea as retention marker. Particle size and porosity Siâ€"O 2 groups of the particles and column wall. In acid groups, in either strong cation-exchange SCX fact, the velocity should be independent of particle or so-called mixed-mode phases [33] which incorpo-

size, as has been demonstrated [31] for monodisperse rate both sulphonic acid and alkyl groups. Pore size 8 based on partition by an electromigration either with nm compared to 30 nm however had no significant or against the EOF. A strong anion-exchange SAX Fig. Variation with pH for two amino-type stationary phases, acetonitrileâ€" 50 mM phosphate buffer Influence of temperature on CEC on silica bonded columns 2. Practical CEC separations on silica based columns Column temperature is easily changed in CEC, and increased temperature reduces the mobile phase The great majority of applications of CEC so far viscosity and hence increases m EOF so that more reported have involved high-resolution reversed- rapid analysis is possible for a given voltage [34] phase separations on bonded 3 mm or 5 mm particles. Separations of such lines may differ for different related com- neutral molecules have predominated in the field of pounds, making selectivity changes possible by environmental and pharmaceutical analysis, and changing the temperature [34]. A peak capacity of over is possible on a 50 the relative retention of chlorothalidone and hydro- cm long column packed with 3 mm particles, making flumethizide is reversed with increasing temperature. The effect of more rapid include separations of oxygen heterocyclic com- K. Temperature effects on the separation of diuretics by CEC. Acetonitrileâ€"waterâ€" 50 mM phosphate buffer, pH 2. A â€" pounds in citrus oils by both reversed-phase and under the same conditions [34]. Beyond this number non-aqueous CEC. More rapid analysis than by there was substantial degradation of column per- HPLC was achieved [37] by both methods. Partial restoration of EOF velocity and Work has also been reported on the use of silica efficiency was achieved by flushing the column with based packings for CEC with pore sizes up to buffer for 2 h, but it is clear that the small mass of nm. These materials should be capable of supporting stationary phase in CEC columns imposes stringent through-particle electroosmosis, and hence signifi- sample clean-up requirements if analytical capability cant increases in column efficiency. A second conse- is to be maintained. Efficient separation of a series of narrow molecular 3. Conclusions mass Mr distribution polystyrene fractions on 30 nm pore size 3 mm silica particles was achieved with Columns packed with bonded silica particles are dimethylformamide as mobile phase; elution was in currently commonly used in CEC. An evaluation of sequence of decreasing number-average Mr Mn. Measure- ments of currents during CEC suggest that the fused- 2. Column lifetime robustness silica column wall contributes substantially to the EOF. The problems arising from burned-in frits in Over consecutive successful injections of CEC columns can be minimised by reducing the frit neutral test mixtures were possible at 30 kV on 25 length and re-bonding stationary phase groups lost cm mm I.

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Chapter 2 : Capillary electrochromatography (eBook,) [calendrierdelascience.com]

Capillary electrochromatography (CEC) is a new and exciting hybrid separation technique that seeks to exploit the combined advantages of both capillary electrophoresis (high efficiencies) and HPLC (mobile and stationary phase selectivity).

Deyl Z, Svec F Capillary electrochromatography. Novotny M, Ishii D Microcolumn separations: Barth, Leipzig, p Wistuba D, Schurig V Electrophoresis Liu CY Electrophoresis Yan C US Pat 5 Sawada H, Jinno H Electrophoresis Fujimoto C Anal Chem Fujimoto C Analusis Liao JL Adv Chromatogr High Resolut Chromatogr Fields SM Anal Chem Fujimoto C J. Wulff G Angew Chem Maruska A, Pyell U Chromatographia Boehringer Gasse 5â€”11, Vienna, Austria Monolithic supports represent a novel type of stationary phases for liquid and gas chromatography, for capillary electrochromatography, and as supports for bioconversion and solid phase synthesis. As opposed to individual particles packed into chromatographic columns, monolithic supports are cast as continuous homogeneous phases. They represent an approach that provides high rates of mass transfer at lower pressure drops as well as high efficiencies even at elevated flow rates. Therefore, much faster separations are possible and the productivity of chromatographic processes can be increased by at least one order of magnitude as compared to traditional chromatographic columns packed with porous particles. Besides the speed, the nature of the pores allows easy access even in the case of large molecules, which make monolithic supports a method of choice for the separation of nanoparticles like pDNA and viruses. Finally, for the optimal purification of larger biomolecules, the chromatographic column needs to be short. This enhances the speed of the separation process and reduces backpressure, unspecific binding, product degradation and minor changes in the structure of the biomolecule, without sacrificing resolution. Short Monolithic Columns SMC were engineered to combine both features and have the potential of becoming the method of choice for the purification of larger biomolecules and nanoparticles on the semi-preparative Keywords.

Chapter 3 : Theory of capillary electrochromatography | Peter Myers - calendrierdelascience.com

Journal of Chromatography A, () calendrierdelascience.com / locate / chroma Review Theory of capillary electrochromatography Keith D. Bartle, Peter Myers School of Chemistry, University of Leeds, Woodhouse Lane, Leeds LS2 9 JT, UK Abstract The present state of the theory of capillary electrochromatography (CEC) is reviewed.*

Chapter 4 : Frantisek Svec - Capillary Electrochromatography - 5

IntroductionCapillary electrochromatography (CEC) is a recently developed analytical separation technique combining, in principle, advantages of high-performance liquid chromatography (HPLC) and capillary electrophoresis (CE).

Chapter 5 : Capillary electrochromatography - Wikipedia

Capillary electrochromatography (CEC) is a chromatographic technique in which the mobile phase is driven through the chromatographic bed by electroosmosis. Capillary electrochromatography is a combination of two analytical techniques, high-performance liquid chromatography and capillary electrophoresis.

Chapter 6 : Capillary Electrochromatography (RSC Publishing)

A new polymer device for use with conventional particulate stationary phases for on-chip, fritless, capillary electrochromatography (CEC) has been realized.

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Chapter 7 : Capillary electrochromatography - University of Manitoba Libraries

Capillary electrochromatography (CEC) is a separation technique in which the flow of the mobile phase or buffer is driven through a chromatographic column by an electric field, rather than by an applied pressure.

Chapter 8 : New stationary phase Publications | PubFacts

This work describes the use of mixed-mode stationary phases which exhibit both strong ion-exchange (either cation-exchange, SCX, or anion-exchange, SAX) and reversed-phase chromatographic characteristics in capillary electrochromatographic separations of pyrimidine derivatives.