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A lean six sigma approach to the improvement of the selenium analysis method

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Since reliable results represent the 'pinnacle assessment of quality' of an analytical laboratory, 'variability' is considered to be a critical quality problem associated with the selenium analysis method executed at Western Cape Provincial Veterinary Laboratory (WCPVL). Due to the narrow margin of safety between toxic and deficient doses of the trace element for good animal health, the elimination and control of 'variability' is undoubtedly of significant importance. To overcome the adverse effect of variation, steps towards analytical process improvement using a quality methodology known as Lean Six Sigma, was believed to present the most feasible solution. Lean Six Sigma represents a form of scientific method type, which is empirical, inductive and deductive, systematic, relying on data and is fact-based. The Lean Six Sigma methodology comprises of five macro-phases, namely, Define, Measure, Analyse, Improve and Control (DMAIC). Both qualitative and quantitative laboratory data were collected in terms of these phases. Qualitative data was collected using quality tools, namely an Ishikawa diagram, Pareto chart, Kaizen analysis and a Failure Mode Effect analysis tool. Quantitative laboratory data, based on the analytical chemistry test method, was collected through a controlled experiment. Laboratory results obtained from the controlled experiment was analysed using statistical methods, commonly associated with quality validation of chemistry procedures. Analysis of both sets of data yielded an improved selenium analysis method, believed to provide greater reliability of results, in addition to a greatly reduced cycle time and superior control features. Lean Six Sigma may therefore be regarded as a valuable tool in any laboratory, and represents both a management discipline, and a standardised approach to problem solving and process optimization

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Binding of methacrylate-based monoliths to PEEK supports for HPLC separations

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Porous monolithic materials are becoming increasingly popular as stationary phases for miniaturized separation techniques. Monoliths have high permeabilities, which enables excellent performance for the fast separation of large molecules. From a chemical viewpoint, the typical monolith consists of a cross-linked polymer backbone that carries the functional groups providing the dedicated selectivity. Monoliths are mostly prepared in fused silica capillaries to be used as separation media in capillary HPLC and electrochromatography. For this purpose, silanization of the inner surface is required to assure binding of the monolith to the silica support. This avoids both ejection of the monolith under external pressure and peak tailing due to gaps located close to the support walls. However, the small inner diameters of silica capillaries (75-520 µm) prevent their use in conventional HPLC. Therefore, there is a need of developing methods to covalently bind monoliths to other supports commonly available in much larger inner diameters than silica capillaries. In this work, procedures to modify the surface of Poly Ether Ether Ketone (PEEK) tubes so that they bind with poly methacrylate-based monoliths for HPLC separations, are described and compared. For this purpose, 0.75 mm i.d. PEEK tubes were used. After selection of binding procedure, several polymerization variables (monomer/cross-linker ratio, type and content of initiator, among others) were studied in order to tailor the chromatographic performance of the resulting columns. Chromatographic tests were applied to provide information about the anchoring of the monolith to the tube wall. Also, mixtures of alkylbenzenes and organophosphorus compounds were used to characterize the chromatographic performance of the developed stationary phases.

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