Kromasil®

Kromasil Diol

AKZO NOBEL

A new Kromasil product, Kromasil Diol, has been developed. This new product has been developed in order to achieve high ligand coverage, and the derivatization utilizing a tri-functional silane has been optimized in order to achieve the same batch to batch reproducibility as with a mono-functional silane. Characterization has been performed using solid state NMR, elemental analysis and chromatographic evaluation.

Background

For normal phase chromatography bare silica is the major product sold on the market. However, alternatives with different selectivity and retentivity for polar compounds have been of interest for many years. In certain cases the aim is also enhanced solubility, which can be achieved by introducing alternative mobile phases. Very polar impurities can also be washed out more easily after the analysis or purification. On the market today, the most common alternative NP phases are cyano- and diol-modified silica.

Silica

Kromasil Diol is based on Kromasil 60 Å which is known to have high chemical purity and excellent mechanical stability.

Derivatization

In order to obtain a stationary phase with a pronounced diol character, a tri-functional silane was used in the derivatization. Figure 1 demonstrates the difference in chromatographic properties between a diol phase with high loading (Kromasil 60-5-Diol), a competitor with low loading, and Kromasil 60 Å silica, in the separation of six different phenols. The Kromasil Diol material with high loading provides a significantly better separation.

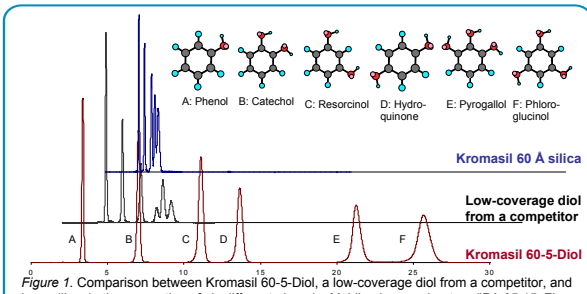

Figure 1. Comparison between Kromasil 60-5-Diol, a low-coverage diol from a competitor, and bare silica, in the separation of six different phenols. Mobile phase: n-heptane/IPA 85:15; Flow rate: 2 mL/min. Detection: UV, 224 nm.

During the development ^{29}Si CP/MAS NMR and elemental analysis have been run in order to characterize the bonded phase, together with a chromatographic evaluation. The surface coverage is high, a typical value is 3.5 µmol/m^2. The aim was to have a diol phase with "full" polymerization, which means no T^1 signals, in order to obtain a stationary phase with low silanol activity and high reproducibility. Figure 2 illustrates that this goal has been achieved due to the complete absence of the T^1 signal. The same manufacturing process is applied for both analytical and preparative materials. This is an critical factor when scaling up from analytical to process scale.

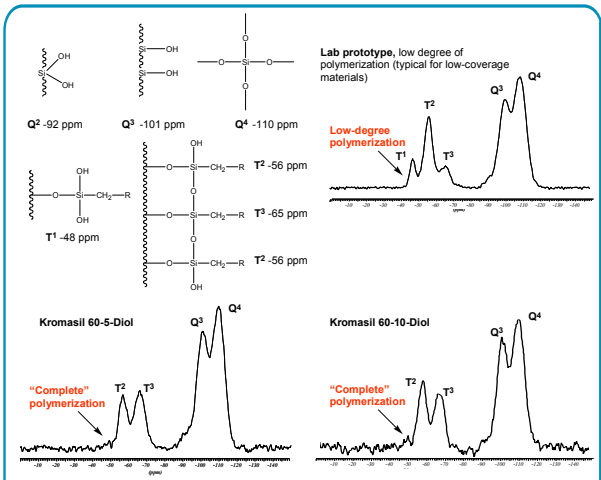

Figure 2. ^{29}Si CP/MAS NMR of Kromasil Diol compared to a lab prototype with a low degree of polymerization.

Comparison Study and Applications

A comparison of Kromasil Diol with other commercial Diol phases, using a test mixture of five different phenols, clearly shows that Kromasil Diol is superior regarding selectivity and symmetry, see Figure 3.

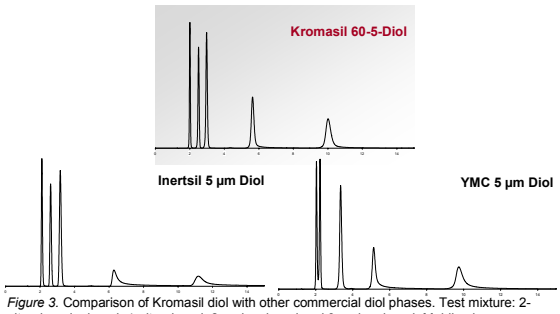

Figure 3. Comparison of Kromasil diol with other commercial diol phases. Test mixture: 2-nitrophenol, phenol, 4-nitrophenol, 2-aminophenol and 3-aminophenol. Mobile phase: n-heptane/IPA 75:25; Flow rate: 2 mL/min. Detection: UV, 254 nm.

In order to investigate if Kromasil Diol is a complement to the standard NP phases, Cyano and bare silica, the selectivity and elution order of a mixture of different phenols and anilines were studied. Figure 4 clearly illustrates that each phase has its own unique selectivity. Interesting studies are also reported in the literature that illustrates this phenomenon.[1,2]

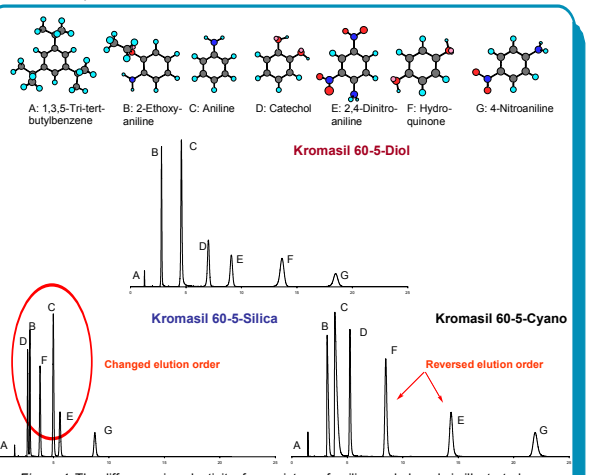

Figure 4. The difference in selectivity for a mixture of anilins and phenols is illustrated for Kromasil Diol, Kromasil Cyano, and bare silica. Mobile phase: heptane/IPA 85/15, Flow rate: 2 mL/min. Detection: UV, 224 nm.

Kromasil Diol is also an interesting stationary phase for RP chromatography, offering alternative selectivity and wettability under 100% aqueous conditions. Figure 5 illustrates an application under RP conditions.

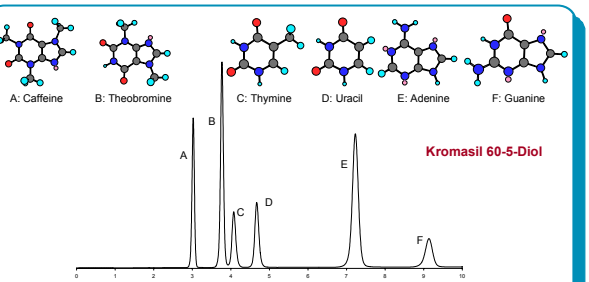

Figure 5. Kromasil Diol under RP conditions. Mobile phase: ACN/25 mM Potassium phosphate buffer, pH 7.85, 80/20. Flow rate: 1 mL/min. Detection: UV, 270 nm.

References

[1] Waksmundzka-Hajnos, M., *J. Liq. Chrom. & Rel. Tech.*, **2004**, 2247-2267.
[2] Waksmundzka-Hainos, M. *J. Chromatogr. A*, **2001**, 919, 39-50.