TSKgel® ODS-100V and 100Z for the analysis of basic compounds at low and neutral pH values

Introduction

ODS phases are synthesized by chemically bonding C18 alkyl chains to silica gel. After the reaction has been completed, a relatively large number of silanol groups remain unreacted on the silica gel surface due to steric hindrance. These residual silanol groups tend to affect sample retention and peak shape.

Two new ODS-type columns have been developed by Tosoh Bioscience - TSKgel® ODS-100V and TSKgel ODS-100Z - with different surface properties utilizing highly efficient bonding and endcapping procedures. This efficient endcapping reduces the number of accessible, residual silanol groups, resulting in reduced peak tailing and improved analyte quantitation.

Results

Using three TSKgel ODS columns differing in their endcapping procedures, changes in the retention and peak shape of desipramine were compared at various mobile phase pH values. The hydrophobicity of desipramine increases at higher pH as fewer amino groups are protonated, affecting the retention of this compound.

Figure 1 shows that as the pH of the mobile phase increases so does the retention time of desipramine on all TSKgel ODS columns. At pH ≥5, retention gradually increased for all three columns, but increased most for TSKgel ODS-80T $_{\rm S}$ QA. The silica used in TSKgel ODS-80T $_{\rm S}$ columns is not as pure as that used in the new TSKgel ODS-100V and ODS-100Z columns. Also, in comparison with TSKgel ODS-100V and TSKgel ODS-100Z, the endcapping efficiency of TSKgel ODS-80T $_{\rm S}$ QA is not optimal, leaving a relatively high number of residual and accessible silanol groups. Thus, when the pH of the mobile phase increases, the electrostatic interaction between the dissociated silanol groups and the secondary amine functional group of desipramine becomes more pronounced.

Figure 2 shows how the asymmetry factor of desipramine changes as a function of mobile phase pH. With ODS-80T_S QA, the higher the mobile phase pH, the greater the asymmetry factor for the desipramine peak. However, with TSKgel ODS-100V, and to a lesser extent with TSKgel ODS-100Z, there were no distinct changes in the asymmetry factor, and, irrespective of mobile phase pH, there was minimal peak tailing. The reasons for this are (again) the purity of the base silica and the effectiveness of the endcapping reactions of TSKgel ODS-100V and TSKgel ODS-100Z, which reduces the number of accessible, residual silanol groups. Because there is very little peak tailing for basic compounds with both TSKgel ODS-100V and TSKgel ODS-100Z when using a neutral pH mobile phase, it is possible to analyze basic compounds even when they are strongly retained.

Figure 1. Retention as a function of pH for a basic compound on three TSKgel ODS columns.

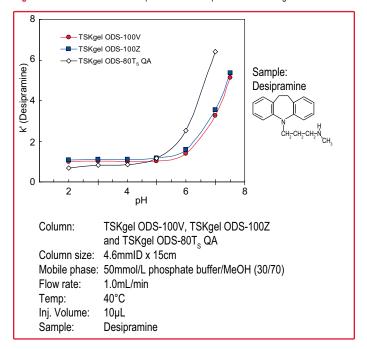
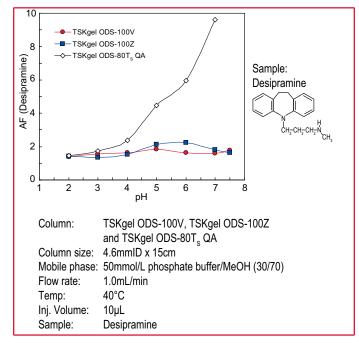


Figure 2. Asymmetry factor of a basic compound as a function of mobile phase pH.



Conclusion

Because of the purity of the base silica and the effectiveness of endcapping procedures, TSKgel ODS-100V and TSKgel ODS-100Z columns are premier C18 columns for the analysis of basic compounds at low and neutral pH values.

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