Method Development in Solid Phase Extraction using Polar ISOLUTE® SPE Sorbents for the Extraction of Non-aqueous Samples

Chemistry Data Sheet

This Chemistry Data Sheet includes specific information on the use of polar ISOLUTE® sorbents for the extraction of polar analytes from non-aqueous samples (pages 1-2), and a general discussion on polar sorbents (page 3).

ISOLUTE® Polar Sorbents: SI, NH2, PSA, DIOL and CN

The ISOLUTE family of polar sorbents are used to extract polar organic compounds from non-polar matrixes. See **Figure 1** for structures of ISOLUTE polar phases.

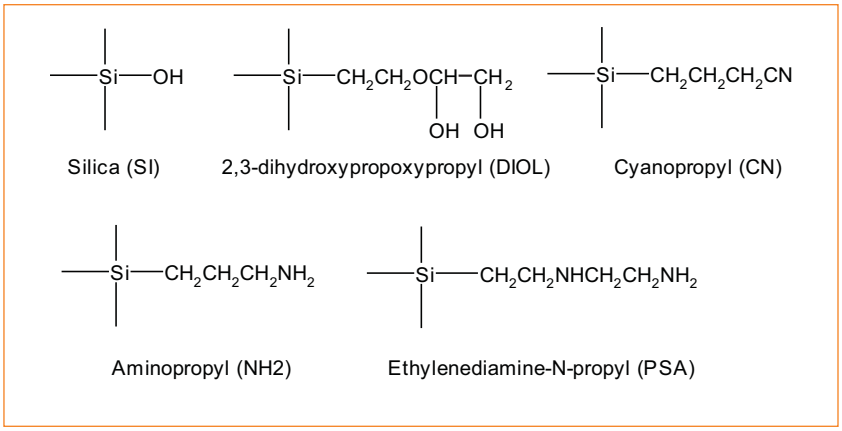


Figure 1. *Structure of ISOLUTE polar phases*

In Method Development using Polar ISOLUTE Sorbents, the Following Points are Important:

Sample Pre-treatment

For optimum retention of polar analytes, the sample should be in a non-polar matrix. If the matrix contains a significant concentration of polar solvent (e.g., acetone or acetonitrile), a non-polar solvent such as hexane should be added to dilute the sample and maximize the non-polar character of the matrix. Other suitable solvents include dichloromethane, chloroform and isooctane.

For clean-up of samples originating from non-liquid matrices, the analytes should first be extracted into a non-polar solvent using a liquid extraction techniques such as soxhlet, ASE or similar.

Biotage

Column Solvation and Equilibration

Polar columns should be solvated with a non-polar solvent similar to that used to dilute the sample. This will maximize the polar interactions between column and analyte.

Sample Loading

For polar columns, typical flow rates are 1 mL/min for 1 mL columns, 3 mL/min for 3 mL columns and 7 mL/min for 6 mL columns. Loading rates may be increased after method chemistry is established.

Interference Elution

A typical solvent for interference elution is the equilibration solvent. Another good choice of solvent is one in which the interferences are soluble, but the analyte is not.

Analyte Elution

Analytes can be eluted using a mixture of polar (e.g. methanol, isopropanol, acetonitrile, acetone) and non-polar (e.g. hexane ethyl acetate) solvents. The polar fraction should be minimized to ensure the greatest selectivity, and hence, the cleanest extract. For some applications, the polar fraction can be as little as 1, 2, or 5% (v/v).

N.B. The activity of unbonded silica (SI) is moisture dependant and can be affected by atmospheric humidity conditions or solvents containing some moisture. It is very important to ensure that solvents used in polar SPE procedures are dry and do not contain any polar modifiers (e.g. ethanol stabilizer in DCM).

Bonded polar sorbents (NH2, PSA, CN and DIOL) are more resistant to changes in activity due to moisture, and SPE procedures using these sorbents can be more robust.

The Range of ISOLUTE Polar Sorbents

Sorbent	Sorbent reference number
SI	460
NH2	470
PSA	480
CN	420
DIOL	430
Florisil	712

Most of these sorbents are available in column configurations ranging from 25 mg/1 mL to 10 g/70 mL, plus high throughput 96-well plates. Please refer to the latest catalog or contact Biotage for more information.

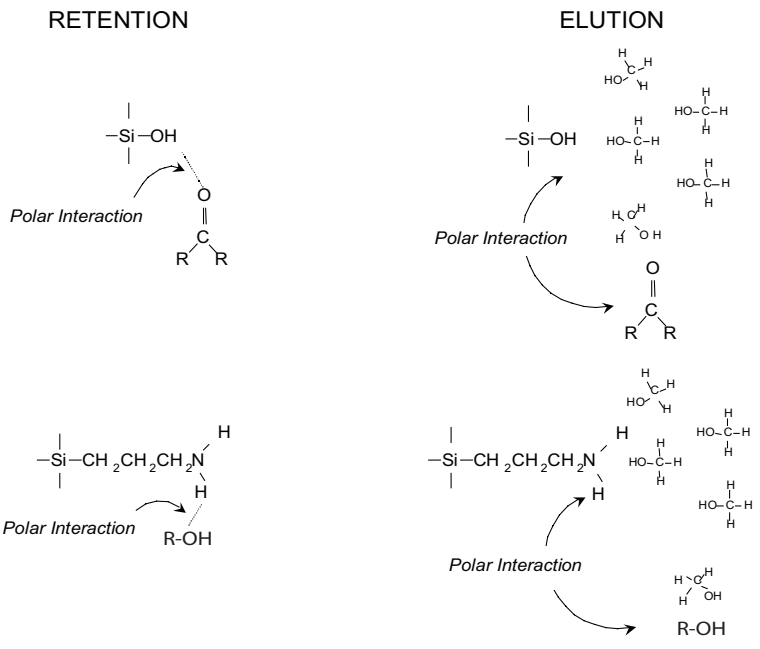
Appendix

SI, NH2, PSA, DIOL and CN (see Figure 1) are polar sorbents that vary in their ability to undergo polar interactions. There are many functional groups that can exhibit polar interactions, and have different selectivities. Some of the polar interactions that exist include dipole/dipole, dipole/induced dipole and hydrogen bonding. Polar interactions are particularly useful for the separation of molecules that are very similar in structure (e.g., structural isomers).

Silica (SI) has the greatest capacity for polar interactions, and is well suited for sample clean-up. SI is capable of exhibiting three different types of interactions: hydrogen bonding, dipole-dipole, and, in the presence of trace amounts of water, ion exchange. Aminopropyl (NH2), ethylenediamine-N-propyl (a primary/secondary amine, PSA) and dihydroxypropoxypropyl (DIOL) sorbents can participate in hydrogen bonding and dipole-dipole interactions. Cyanopropyl (CN) can participate only in dipole-dipole, and is suitable for analytes that are difficult to elute from the other polar sorbents.

Retention and Elution Characteristics of ISOLUTE Polar Sorbents

See **figure 2** for retention and elution using ISOLUTE SI and NH2. The remaining sorbents behave similarly.



RETENTION: Polar interactions between the analyte and sorbent retain the analyte during loading.

ELUTION: A polar solvent, such as methanol, will compete for interaction with the surface, and elute the analyte.

Figure 2. *Retention and Elution Characteristics of ISOLUTE Polar Sorbents*

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