

Introduction to Functionalized Silica Gel and Alumina RediSep® Columns

Application Overview

This application provides an introduction to Isco's RediSep® Columns loaded with functionalized silica gel and basic, acidic, and neutral alumina.

The RediSep functionalized columns are as follows:

Amine

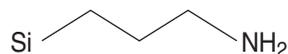


Figure 1: Amine compound

The amine functionalized silica can be used in either normal or reversed phase conditions. It is useful in purification of compounds with amine substitution. This is particularly true if spiking with triethyl amine (TEA) is required for purification with normal phase silica.

The use of a RediSep Amine column can eliminate the need to spike with TEA, and as a result, reduce time required to remove solvent from purified fractions.

Cyano

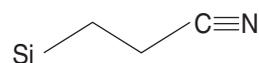


Figure 2: Cyano compound

The cyano functionalized silica acts very similar to normal phase silica when using similar solvents. In reversed phase conditions, it is similar to Isco's C-18 reversed phase columns, although the elution order of compounds may be different.

Fluoro-tagged

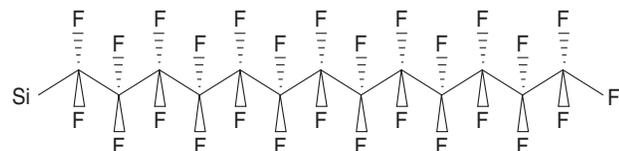


Figure 3: Fluoro-tagged compound

Isco's fluoro-tagged columns are packed with a poly-fluorinated-tridecyl group for separation of fluorinated compounds as well as a separation of degrees of fluorination. That is, it will separate something that has three fluorine molecules from something that has five fluorine molecules in the compound.

Alumina

There are three different alumina columns available: Basic, Acidic, and Neutral.

Since normal phase silica is slightly acidic, some acid-sensitive compounds will break down. In those cases, the use of neutral alumina can be helpful for separation of compounds without running the risk of acidity breaking down a compound.

Compounds that have an acidic or basic moiety may streak or tail with normal or reversed phase silica. Streaking or tailing will ultimately cause multiple fractions and overlapping fractions.

Typically, chemists spike their solvents with either triethyl amine (TEA) if they have a basic component, or acetic acid (AcOH) if they have an acidic component in their target compound. The problem here is twofold: first solvents have to be swapped and primed before compounds can be separated, as well as change the system and purge after the run. The second problem is that TEA or AcOH must be removed after the run. This involves an additional extraction and washing with a suitable solvent, or concentrating the mixture down to an oil and placing the oil on a high vacuum overnight.

With an acid or base moiety covalently bound to the stationary phase, the need to switch solvents is eliminated. Additionally, TEA or AcOH are not used so they don't need to be removed after the purification is complete.

Applications

Application guidelines for the functionalized gels are listed in Table 1.

Reuse Instructions

If reusing a functionalized column, always flush the column with the highest polarity solvent that was used in the previous separation or purification. After flushing the column, purge it with plenty of air. Purging with air and keeping in a desiccator when not in use will often increase the life expectancy of the columns.

If these procedures are followed, the columns can be used up to approximately ten times. However, cross contamination can occur.

Table 1: Application Guidelines for RediSep Functionalized Columns

Functionalization	Suitable Solvents	Loading (w/w)	Functional Groups to Purify	Reusable?
Amine	Hexanes, Ethyl Acetate, Methylene Chloride, Chloroform, Methanol, Ethanol, Isopropanol, Water, Acetonitrile	1%–10%	Most organic compounds. (Usually have a base function on target compound)	Yes, treat with highest polar solvent used in separation.
Cyano	Hexanes, Ethyl Acetate, Methylene Chloride, Chloroform, Methanol, Ethanol, Isopropanol, Water, Acetonitrile	1%–10%	Most organic compounds.	Yes, treat with highest polar solvent used in separation.
Fluorous-tagged	Methanol, Water. Also used: ethanol, isopropanol, acetonitrile, dimethyl formamide (DMF), dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), ethyl ether, chloroform, methylene chloride, hexanes, benzene, and toluene.	5%–15%	Separate organic compounds from fluorinated compounds and separation of fluorinated compounds by degrees.	Yes. Flush with tetrahydrofuran (THF). Can be reused up to 10 times. Cross contamination may occur, though.

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