



The use of ISOLUTE® HM-N for Loading a Reaction Mixture that is Insoluble (or sparingly soluble) in Non-polar Solvents onto an ISOLUTE Flash SI II Column

This Chemistry Data Sheet describes the use of ISOLUTE® HM-N as a support material for pre-absorption of a reaction mixture prior to flash chromatography. This approach is used as a solution to loading reaction mixtures that are only soluble in polar solvents onto an ISOLUTE Flash SI II or Flash NH2 column.

The Problem

The reaction mixture is only soluble in a polar solvent such as isopropyl alcohol or acetone. When using normal phase chromatography, if such a sample is applied to a silica flash column, the separation will be adversely affected due to the polar nature of the solvent, allowing migration of the components through the column prior to the complete application of the sample. The chromatographic efficiency of the column will be reduced or lost completely.

Traditional Method

Traditionally, this problem has been overcome by using silica to pre-absorb the sample; loose silica is added to the reaction mixture and the solvent subsequently evaporated. However, the silica used can affect the separation if:

- Silica from a different source to that used in a flash column is used to pre-absorb the sample (different surface pH, surface area etc).
- There are activity (water content) differences between the silica used to pre-absorb the sample and that packed in the flash column.
- Retention of the compounds on the silica requires a strong solvent for desorption, due to interactions between the silica sorbent and the compounds.

The Solution - ISOLUTE HM-N for Pre-absorption

ISOLUTE HM-N is a modified form of diatomaceous earth with an extremely high surface area. The compounds from the reaction mixture are absorbed onto the surface of the ISOLUTE HM-N as described. Provided all compounds are soluble in the initial solvent, the compounds will be released from the diatomaceous earth onto the silica flash column. The components will then be pre-concentrated at the top of the flash column.

Procedure (see Figure 1)

ISOLUTE HM-N is added to the reaction and mixed. The solvent is then evaporated or removed in a gas stream or on a rotary evaporator. Any volatile solvent is suitable; feedback has indicated that dichloromethane, chloroform, acetone and methanol work well.

The sample to ISOLUTE HM-N ratio is optimal at 2 : 3. This refers to the mass of dry sample. For a sample mass of 100 mg or less, at least 250 mg of ISOLUTE HM-N is recommended to facilitate easier sample handling.

After solvent evaporation, the ISOLUTE HM-N is dry and free flowing. It can be poured onto the top of a flash column. The column should be gently tapped to evenly distribute the material. An additional frit is then placed onto the ISOLUTE HM-N bed using a frit inserter. It is important to lightly press the frit onto the diatomaceous earth to avoid crushing. The flash chromatography separation can then be carried out as normal.

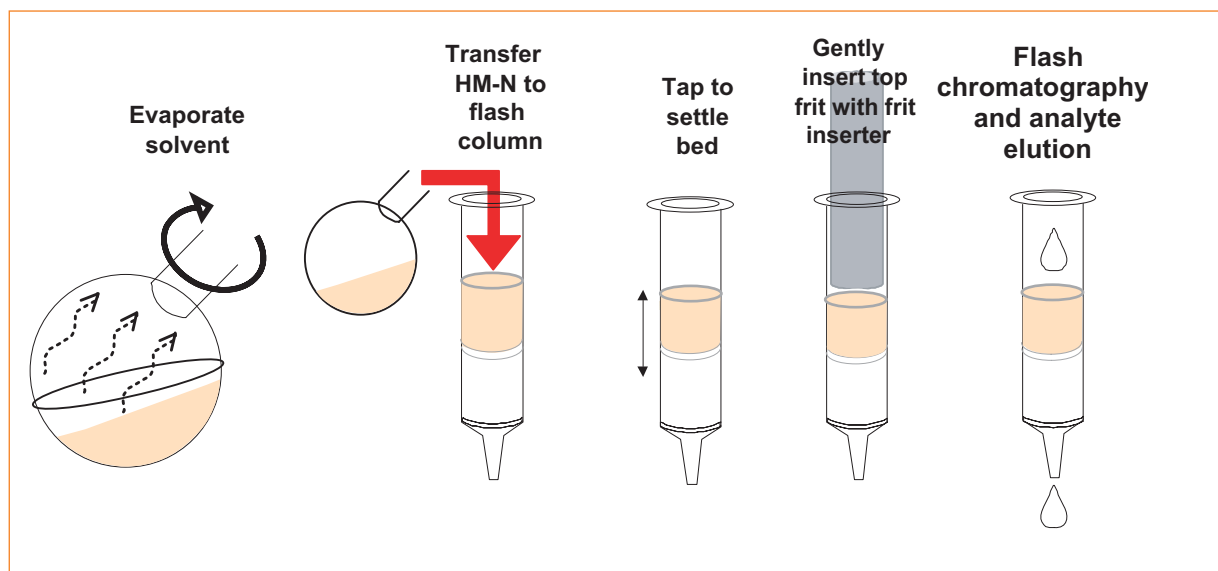


Figure 1. Procedure for pre-absorption in flash chromatography using ISOLUTE HM-N

Bulk ISOLUTE HM-N Ordering Information

Part Number	Description	Quantity
9800-1000	Bulk ISOLUTE HM-N	1 Kg
9800-5000	Bulk ISOLUTE HM-N	1 Kg

Polyethylene Frit Ordering Information

Part Number	Description	Quantity
120-1036-D	20 μ Polyethylene frit, 16 mm	100
120-1037-E	20 μ Polyethylene frit, 20 mm	100
120-1038-F	20 μ Polyethylene frit, 27 mm	100
120-1039-J	20 μ Polyethylene frit, 37 mm	50
120-1049-X	20 μ Polyethylene frit, 40 mm	50

Frit Inserter Ordering Information

Part Number	Description	Quantity
120-1151-D	Frit inserter for 16 mm (D) column	5
120-1151-E	Frit inserter for 20 mm (E) column	1
120-1151-F	Frit inserter for 27 mm (F) column	1
120-1151-J	Frit inserter for 37 mm and 40 mm (J & X) columns	1



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