



Work-up of Basic Products from Organic Synthesis Mixtures using ISOLUTE® SCX-2

This Chemistry Data Sheet details the use of ISOLUTE® SCX-2, a strong cation exchange sorbent, for the work-up of basic products from organic synthesis mixtures containing non-basic reagents or by-products.

In medicinal chemistry reactions are often carried out in solvents such as DMSO. Once the reaction is complete, it is usually necessary to separate the products of the reaction from reagents and by-products. This can be performed using strong cation exchange sorbents (e.g. ISOLUTE SCX-2 or ISOLUTE Flash SCX-2) to selectively isolate the basic compounds from the reaction mixture. (see **Figure 1** for structure of ISOLUTE SCX-2 and ISOLUTE Flash SCX-2 sorbents)

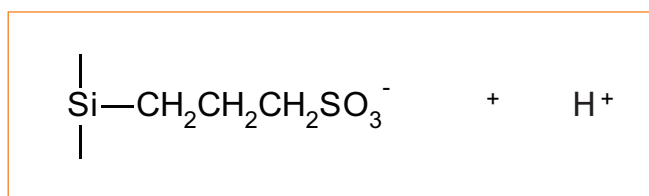


Figure 1. Structure of ISOLUTE SCX-2 and ISOLUTE Flash SCX-2 bonded phases

Chemical Data

Base Material: Silica, 50 µm

Functional Group: Propylsulphonic acid

Capacity: 0.6 mmol/g

Counter Ion: Proton

Protocol

This protocol is based on the use of an ISOLUTE SCX-2 500 mg/6 mL column (part number 532-0050-C). This column is suitable for isolation of approximately 10-50 mg of basic product with a molecular weight of 350 amu. The method can be scaled up as necessary using larger column sizes and increasing the volumes of solvents used accordingly.

N.B. If the basic compound to be removed is a reagent present in excess, a larger column would be required to work-up the same amount of product.

Crude Sample/Reaction Mixture Preparation

The sample (organic synthesis reaction mixture in a solvent such as DMSO) often requires dilution with solvents of lower polarity for optimum results. A suitable dilution solvent would be a mixture of dichloromethane (methylene chloride) and methanol. Choice of dilution solvent is influenced by the polarity of the compounds in the sample, as they must remain in solution. In general, the selectivity of the method will be enhanced by using the most non-polar solvent or solvent mixture possible.

SPE Column Conditioning

Wet the column with methanol or DCM (2 mL).

Sample Application

Apply the sample to the column under gravity. A typical volume of sample for the 500 mg/6 mL configuration would be 1-10 mL, however, this is influenced by the concentration of product in the sample. Sample size should be accurately determined by weight of product to be purified.

Non-basic Impurity Removal

Elute non-basic reagents and by-products with methanol/dichloromethane (2 x 2 mL).

Target Compound/Molecule Release

Elute basic products with methanol/1.0 M NH₃ (2 x 2 mL). For best results, compound elution solvent should be made up using 7.0 M NH₃, diluted 1:7 (v/v) with methanol. This solution contains no water, and is therefore easier to evaporate to dryness.

ISOLUTE SCX-2 Ordering Information

Part Number	Description	Quantity
532-0050-B	500 mg/3 mL	50
532-0050-C	500 mg/6 mL	30
532-0100-B	1 g/3 mL	50
532-0100-C	1 g/6 mL	30

ISOLUTE Flash SCX-2 Ordering Information

Part Number	Description	Quantity
456-0200-D	2 g/15 mL	20
456-0500-E	5 g/25 mL	20
456-1000-F	10 g/70 mL	16
456-2000-F	20 g/70 mL	16
456-5000-J	50 g/150 mL	8
456-7000-J	70 g/150 mL	8

ISOLUTE Tab-less Columns for High Throughput Work-up Ordering Information

Part Number	Description	Quantity
532-0050-B	ISOLUTE SCX-2 500 mg/3 mL ,tab-less	50
532-0050-C	ISOLUTE SCX-2 1g/3 mL, tab-less	50
532-0100-B	ISOLUTE SCX-2 500 mg/6 mL ,tab-less	30
532-0100-C	ISOLUTE SCX-2 1g/6 mL ,tab-less	30

ISOLUTE-96 SCX-2 Fixed-well Plate Ordering Information

Part Number	Description	Quantity
532-0100-P01	ISOLUTE-96 SCX-2 100 mg	1
532-0500-P01	ISOLUTE-96 SCX-2 500 mg	1

ISOLUTE Array SCX-2 Ordering Information

Part Number	Description	Quantity
532-0100-T	ISOLUTE- Array SCX-2 100 mg/2 mL	100



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