Matrix Solid Phase Dispersion

Chemistry Data Sheet

Matrix Solid Phase Dispersion (MSPD) is a sample preparation technique for use with solid sample matrices. Conventional extraction of organic analytes from tissue usually begins with a homogenization step, followed by tedious liquid-liquid extraction procedures, sample clean-up and trace enrichment. A relatively large amount of sample matrix is required, and solvent consumption is high.

MSPD is a microscale extraction technique, typically using less that 1 g of sample and low volumes of solvents. It has been estimated to reduce solvent use by up to 98% and sample turnaround time by 90% [1].

The technique involves homogenization of a small amount of sample tissue with bulk bonded silica-based sorbent in a pestle and mortar. The mechanical shearing forces produced by the grinding process disrupt the structure of the tissue, dispersing the sample over the surface of the support sorbent by hydrophilic and hydrophobic interactions. The process causes the mixture to become semi-dry and free-flowing, and a homogenous blend of sample and sorbent is the result. This blend is then packed into a pre-fritted SPE column, and elution of interference compounds and analytes of interest can then take place using suitable solvents.

ISOLUTE® MSPD sorbents

Two MSPD grade sorbents, ISOLUTE MSPD C18 and MSPD C18(EC), have been developed to enhance this technique further allowing a more rapid and even blend of the sorbent/sample mixture to be produced. The characteristics of the MSPD grade sorbents make the blending process less operator dependant, and the homogenous nature of the blend produced allows a very even sorbent bed to be packed into the column. This in turn leads to even flow characteristics, and reduces the risk of channelling, so higher and more reproducible recoveries are possible.

ISOLUTE MSPD C18 and MSPD C18(EC) are both suitable for the extraction of a variety of organic analytes such as drugs, steroids and pesticides from complex matrices. The additional polar silanol interactions available with the non-endcapped C18 sorbent may give higher recoveries in the extraction of basic analytes. (See **Figure 1** for illustration at MSDS process).

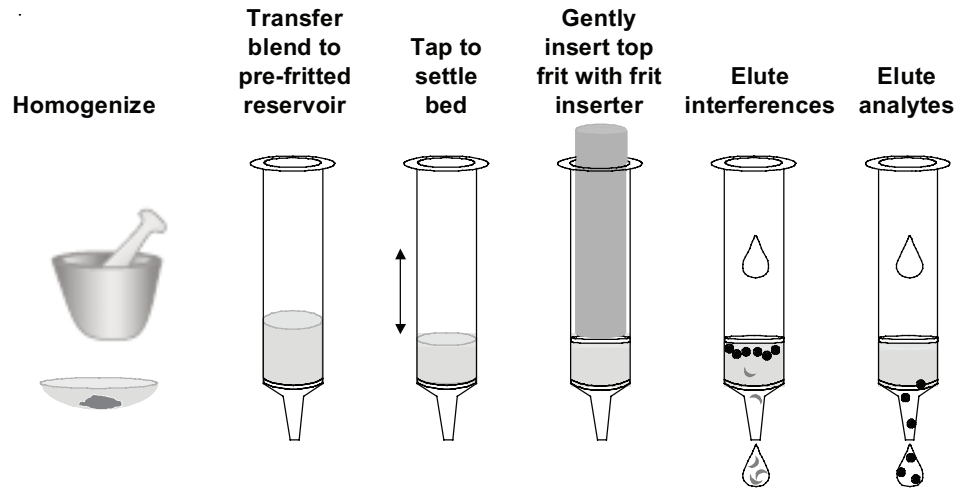

Figure 1. *The MSDS process*

Biotage

In method development using MSPD sorbents, the following points are important:

Sample Pre-treatment

A typical amount of sample used in MSPD is 0.5 g. However, the expected concentration of analyte, and the detection limits of the analytical technique should be considered during method development. If internal standard is used, it should be added prior to the sorbent-matrix blending process.

The sample should be placed in a glass mortar, and the ISOLUTE MSPD sorbent added. A typical ratio is 1 part sample to 4 parts sorbent. The optimum ratio will vary depending on the nature of the sample matrix e.g. a vegetable sample with a high water content may require higher proportion of sorbent to sample matrix.

The mixture should be gently blended in the pestle and mortar until a smooth, dry, homogenous, free-flowing powder is produced. This should take between 30 seconds and 1 minute.

Transfer the mixture into a pre-fritted reservoir containing a bottom frit. Tap the reservoir gently (vertically and horizontally) to settle the bed, and insert a top frit. Using a frit inserting tool, push the top frit to the top of the bed, and compress gently. Place the packed cartridge on a VacMaster™ Sample Processing Manifold.

Interference Elution

The purpose of interference elution is to selectively remove undesired compounds from the sorbent without eluting the analytes. A typical volume of interference elution solvent is 1-2 mL/100 mg of sorbent-matrix blend. The flow rate should be adjusted such that the solvent is in contact with the sorbent for 1-2 minutes.

Typical interference elution solvents are deionized water, or 10-20 mM buffers. A buffer containing 10-30% methanol or acetonitrile is often suitable for removing lipophilic interferences. Lipids can be eluted using hexane. Care should be taken when eluting interferences with organic solvents that analytes are not eluted. For basic analytes, an interference rinse with 50 mM acetic acid may be suitable. Cartridge drying may be necessary to remove water if the elution solvent is water immiscible.

Drying can be performed by vacuum aspiration, N_2 or CO_2 flow, or centrifugation (useful if the analytes are volatile). Drying times depend on factors such as sorbent, bed dimensions, and drying method. A typical range, depending on the degree of dryness required, is 30 seconds to 30 minutes. If a water miscible elution solvent is selected, the time required for cartridge drying can be reduced or eliminated. If the sample is to be concentrated to a smaller volume after elution, and the drying step was reduced or eliminated, care should be taken not to reduce the volume of solvent to where phase separation occurs, or the analytes precipitate.

Analyte Elution

The elution solvent should be one in which the analytes are soluble. It must often overcome primary and secondary retention mechanisms, and so a solvent or mixture of solvents offering multiple interactions is usually most effective. The elution solvent should also be compatible with the final analysis technique. A water miscible elution solvent may be used to elute analytes and minimize cartridge drying times (see interference elution in previous step).

For example, for an HPLC analysis, a solvent similar to the mobile phase is a good choice of elution solvent. A volatile solvent is generally selected for subsequent GC analysis. Other factors to consider include whether there will be a derivatization step, as well as volatility of the solvent if further concentration is required.

A minimum volume of elution solvent allows maximum concentration of the analytes. A typical minimum elution volume is 250 µL /100 mg of sorbent-matrix blend. Flow control is important to ensure reproducibility. The use of two small aliquots of solvent with a 1-4 minute soak step between elution volumes is often more efficient than one large aliquot. If a single elution is required, the flow rate of the elution solvent should be such that contact time between solvent and sorbent is 1-4 minutes.

Typical elution solvents are methanol, acetone, dichloromethane or other solvents or solvent mixtures; solvent containing 1-5% formic or acetic acid, or solvent containing a volatile amine such as TEA or TMA (these will help overcome secondary silanol interactions).

Sample Clean-up

Due to the very complex nature of the sample matrix, it may be necessary to perform a separate clean-up of the eluate. This is best performed when the analytes have been eluted in a relatively non-polar solvent.

The eluate should be applied to a pre-conditioned polar column such as ISOLUTE SAX/PSA, or Florisil. Polar interferences will be retained on the column, while the analytes can be selectively eluted with the correct choice of solvent.

For more detailed information on the use of polar ISOLUTE SPE columns for removal of polar interferences from non-aqueous samples, please refer to Chemistry Data Sheet TN102 'Method Development in Solid Phase Extraction using Polar ISOLUTE SPE Sorbents for the Extraction of Non-aqueous Samples'.

A more recent development in MSPD is particularly applicable to the extraction of basic veterinary drugs from liver and other biological matrices. This involves the addition of ISOLUTE SCX-2 cation exchange sorbent to the C18-matrix blend. This step enables additional interference elution washes to be used in the clean-up step, producing exceptionally clean samples[2]. Request Application Note 1045 for more information.

References

1. S. Barker and R. Hawley, *Int-Lab. Sep* **1992**, *22(8)*, pp46-48
2. S. Collins, M. O'Keeffe, R. Calverley and M.R. Smyth, *Proceedings of Euroresidue III*, Conference on Residues of Veterinary Drugs in Food, Veldhoven, The Netherlands, 6th-8th May **1996,** pp340-345

MSPD Sorbent Ordering Information

Part Number	Description	Quantity
9370-0100	ISOLUTE MSPD C18	100g
9371-0100	ISOLUTE MSPD C18(EC)	100g

MSPD Accessories Ordering Information

Part Number	Description	Quantity
120-1151-C	Frit inserter for 6 mL (C) columns	5
120-1151-D	Frit inserter for 15 mL (D) columns	5
120-1142-C	6 mL (C) reservoirs fitted with single bottom frit, 100 additional frits included	100
120-1143-D	15 mL (D) reservoirs fitted with single bottom frit, 100 additional frits included	100
120-1148-L	6 mL (L) glass reservoirs fitted with single PTFE frit, 30 additional PTFE frits included	30

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