

Review

Matrix solid phase dispersion (MSPD) [☆]

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Abstract

A review of the many uses of matrix solid phase dispersion (MSPD) in the extraction and analysis of a variety of compounds from a range of samples is provided. Matrix solid phase dispersion (MSPD) has found particular application as a somewhat generic analytical process for the preparation, extraction and fractionation of solid, semi-solid and/or highly viscous biological samples. Its simplicity and flexibility contribute to it being chosen over more classical methods for these purposes. MSPD is based on several simple principles of chemistry and physics, involving forces applied to the sample by mechanical blending to produce complete sample disruption and the interactions of the sample matrix with a solid support bonded-phase (SPE) or the surface chemistry of other solid support materials. These principles are discussed as are the factors to be considered in conducting a MSPD extraction.

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Keywords: Matrix solid phase dispersion (MSPD); Solid phase extraction (SPE); Drug analysis; Tissue analysis; Food analysis

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1. Introduction

Since its introduction in 1989 [1], matrix solid phase dispersion (MSPD) has been cited as the extraction method employed in over 250 publications [1–262]. It has proven to be an efficient and somewhat generic technique for the isolation of a wide range of drugs, pesticides, naturally

occurring constituents, and other compounds from a wide variety of complex plant and animal samples (Table 1). MSPD was patented in 1993 [226] and its many uses have been extensively reviewed [52,76,110,133,139,157,159,161,163,190,192,199,200,215,222,228,230,231,236,237,245,246,251].

Matrix solid phase dispersion (MSPD) has found particular application as an analytical process for the preparation, extraction and fractionation of solid, semi-solid and/or highly viscous biological samples. Its simplicity and flexibility have been cited as contributing to it being chosen over more classical methods for these purposes. Indeed, MSPD is based on several simple principles of chemistry and physics, involving forces applied to the sample by mechanical blending to produce

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Table 1
Sample matrices that have been examined for the analytes noted

Matrix	Analyte(S)	Reference
Almonds	Ethylene bisdithiocarbamate	[72]
Animal ^a fats	Acetylgestagens	[213]
	DDT	[19]
	DDTs, aldrin, dieldrin	[77]
	Hexachlorobenzene	[116]
	Organochlorine pesticides	[204]
	PCBs	[55,132]
	Pesticides	[173,256,257]
Animal* feed	Chloramphenicol	[30]
	Dibenzo[a,l] pyrene	[143]
	Pesticides, PCBs	[37]
Animal* kidney	Aminoglycoside antibiotics	[223]
	Atrazine	[151]
	Avoparcin	[134]
Animal* liver	Benzimidazoles	[261]
	Beta-agonists	[205,219]
	Clenbuterol	[152,182,232,233]
	Ivermectin	[244]
	Mycotoxins	[122]
Animal* liver, kidney	Microcystins	[34]
	Sulfonamides	[107]
Animal* muscle	Aminoglycosides	[249]
	Antibiotics	[220]
	Benzimidazoles	[50,153,257]
	Cefquinome	[218]
	Chloramphenicol	[117]
	Diethylstilbestrol	[86]
	Furazolidone	[258]
	Moxidectin	[196]
	Nicarbazin	[243]
	Pesticides, anthelmintics and antibiotics	[1]
	Penicillin	[181]
	Sulfamethazine	[225,212,234]
	Sulfonamides	[90,140,217]
	Tetracyclines	[6]
	Thyreostats	[32]
	Veterinary drugs	[191]
Apples	Azoxystrobin, trifloxystrobin	[93]
	Pesticides	[3]
Apple juice	Pesticides	[9,13,48]
Beans	Pesticides	[78]
Biological samples	Beta-2-agonists	[160]
	Methomyl	[155]
	Pesticides	[184]
	Verapamil	[119]
Biota	Pcbs	[36,138]
	Surfactants	[91]
Bovine tissues and milk	Amoxicillin, ampicillin	[74]
Cattle and fish	Sulfonamides	[113]
Cheese	Biogenic amines	[35]
Chili peppers	Aflatoxins	[4]
Chocolate	Fatty acids	[64]
Confections	Coal-tar dyes	[197]
Corn	Carbofuran	[211]
Crayfish	Pesticides	[229]
Crustaceans and their parasites	Carotenoids	[61]
Cyanobacteria cultures	Microsystins	[56]
Dairy products	PCBs, dibenzodioxins, dibenzofurans	[169]
Eggs	Furazolidone	[75]

Table 1 (continued)

Matrix	Analyte(S)	Reference		
Fish	Alcohol ethoxylates	[175]		
	Alkylbenzene sulfonates	[120]		
	DDT	[135]		
	Ivermectin	[216]		
	Mycotoxins	[53,92]		
	Microcystins, nodularin	[27]		
	Oxolinic acid	[247]		
	Oxytetracycline	[260]		
	Pesticides	[227,255,256]		
	Pesticides and PCBs	[207]		
	PCBs	[29,224]		
	Sulfadimethoxine	[214,221,259]		
	Sulfonamides	[241,254]		
Fish and mussels	Surfactants	[170]		
	Triazenes	[158]		
Foods	Alkylphenol ethoxylates	[178]		
	Aflatoxins	[81]		
	Chloramphenicol	[201]		
	Dioxins and PCBs	[59]		
	Isoproc carb, deltamethrin	[31]		
	N-methylcarbamates	[46]		
	Fruit-green tea	Phenolics	[62]	
		Fruit juice	Carbendazim	[14]
	Pesticides		[101,130]	
	Fruits	Avermectin	[148]	
		Abamectin	[166]	
		Benzoyl ureas	[165]	
		Organophosphorous pesticides	[187]	
Pesticides		[26,63,149,176]		
Pesticides		[177]		
Fruits and nuts		Fruits and vegetables	Carbamates	[83,167]
			Carotenoids	[141]
		Fungicides	[127,128,131,137,189]	
		Organophosphorous pesticides	[111]	
		Pesticides	[40,41,144,202]	
		Pirimicarb, amitraz	[80,208]	
Fruits, vegetables and cereals		Fruits, vegetables and insects	Dithiocarbamates	[82]
	Pesticides		[124]	
	Oxamyl and methomyl	[248]		
Garlic	Pendimethalin	[10]		
	Insecticides	[136]		
	Honey	Organophosphorous pesticides	[114]	
		PAHs	[103]	
		Pesticides	[145]	
	Honeybees	Pesticides	[121]	
	Infant formula	Retinyl acetate	[174]	
		Tocopherol acetate, retinyl palmitate	[183,186]	
		Vitamin K1	[162,168]	
	Insects	Pesticides	[65]	
	Krill	Astaxanthin and esters	[22]	
	Leaves	Isoflavones	[58]	
	Marine Species	Antibacterials	[180]	
Medical foods	Beta-carotene	[172]		
	Tocopherol acetate, retinyl palmitate	[179]		
	Vitamin K1	[156,164]		
Medicinal plants	Isoflavanoids	[79]		
	Organophosphorous pesticides	[129]		
	Pesticides	[16,102]		
Milk	Phenolic acids	[118]		
	Aminoglycoside antibiotics	[45]		
	Carbamate insecticides	[57]		
	Clorsulon	[240,252]		

Table 1 (continued)

Matrix	Analyte(S)	Reference	
Milk	Cypermethrin	[73]	
	Doxycycline, fumequine	[67]	
	Enrofloxacin, ciprofloxacin	[20]	
	Fenbendazole	[193]	
	Ivermectin	[210]	
	Organochlorine and phosphorous pesticides	[206]	
	PCBs and organochlorine pesticides	[142]	
	Tetracyclines	[262]	
	Milk, muscle, organ tissues	Methomyl	[198]
		Pesticides	[185]
Sulfonamides		[25]	
Milk, cheese, meat	Tetracyclines	[188]	
Milk and eggs	Alkylphenols, bisphenol A	[24]	
	Sulfonamides	[106]	
Milk, eggs, avacados	Pesticides	[38]	
Milk, eggs, tissues	Sulfonamides	[242]	
Milk, infant formula	Vitamins A And E	[60]	
Milk and meat	Sulfonamides	[253]	
Milk and urine	Thyrestats	[23]	
Mycobacteria	Biologic compounds	[97]	
	Natural products	[235,239,250]	
Okra	Pesticides	[51]	
Olive oil	Benzo(a) pyrene	[84]	
	Organophosphorous pesticides	[87]	
	Terbutylazine	[11]	
Olives, olive oil	Pesticides	[44]	
Oranges	Pesticides	[66,125,147,203]	
	Thiabendazole	[69]	
Oysters	Pesticides	[238]	
Pistachio nuts	diazinon, ethion	[94]	
Plants	Aflatoxins	[96]	
	Alkylphenols	[115]	
	Carbendazin	[85]	
	Natural products	[112]	
	Pesticides	[195]	
Rat feces	Bilastine	[146]	
Rice	Carbofuran	[105]	
	Isoprocarb	[39]	
	Pesticides	[54]	
Root bark	Flavanones, xanthones	[49]	
Sewer sludge, sediments	Phenolics	[2]	
Soil	Fenprothrin	[99]	
	Pesticides	[12,104]	
	Phenthoate	[123]	
	Uniconazole	[109]	
Spinach	Carotenoids	[25,108]	
Spinach and retina	Carotenoids	[171]	
	Lutein and zeaxanthin	[154]	
Sugar cane	Simazine	[71]	
Starfish	Saponins	[150]	
Tea	Caffeine	[33]	
	Pesticides	[15]	
Tobacco	Pesticides	[18]	
Tomato	Glyphosate, aminomethylphosphonic acid	[21]	
	Pesticides	[100]	
Tomato juice	Tocopherols	[126]	
Toothpaste, gingival tissues	Tocopherols	[126]	
Vegetables	Carotenoids	[47]	
	Pesticides	[194]	
	Pyrethroids	[209]	
	Tebufenpyrad	[42]	

Table 1 (continued)

Matrix	Analyte(S)	Reference
Vegetable and fruit juices	Herbicides	[70]
Wheat grain	Carbindazim	[88]
	Carbofuran	[89]
	Fungicides	[7]

DDT, 1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane; PCBs, polychlorinated biphenyls; PAHs, polycyclic aromatic hydrocarbons.

^a Excluding marine species.

complete sample disruption and the interactions of the sample matrix with a solid support bonded-phase (SPE) or the surface chemistry of other solid support materials.

In its original conception, the blending of a bonded-phase [such as octadecylsilyl (ODS)-derivatized silica (C_{18})] solid support material with a biological sample is seen as acting as both an abrasive, producing shearing and grinding forces that induce disruption of the sample architecture, and as a “bound” solvent that assist in accomplishing complete sample disruption and dispersion. In this manner, the sample is dispersed over the surface of the bonded-phase support material, producing, through hydrophobic and hydrophilic interactions of the various components, a unique mixed-character phase for conducting target analyte isolation. Indeed, blended samples (muscle tissue and ODS-silica support) have been examined by scanning electron microscopy (SEM) and show that sample architecture is completely disrupted and that sample matrix components are, apparently, evenly distributed over the surface of the bonded-phase/support, forming an observable layer of approximately 100 μm in thickness, similar to that of some micelle or membrane bilayers [1]. More recently, many applications of MSPD have involved the blending of samples with underivatized silicates (silica gel, sand, etc.) [for example, see 49,85,74,113,121,130] or other organic (graphitic fibers) [19,82] or inorganic (Florisil, alumina, etc.) [10,77,138,140] solids which cause sample disruption but do not, apparently, possess the same dispersive properties [159,237].

MSPD has found favor in its many applications because it eliminates most of the complications of performing classical liquid–liquid and/or solid phase extractions of solid and semi-solid samples, particularly complex biological samples. Indeed, all classical forms of liquid chromatography require that the sample be applied in a solubilized state to the head of the column. To accomplish this, the overall method must include steps to render the sample and its components into a non-viscous, particulate-free and relatively homogeneous liquid state. While many biological fluids, such as urine or blood plasma or serum, can be applied to columns directly, most other samples are not directly applicable to SPE, particularly the solids and semi-solids which are derived from biological origins. Such samples may be obtained from animal tissues or vegetable material and consist of a non-homogeneous array of fat and/or other tissues, such as fiber and pulp in the case of plants.

Thus, classical approaches for the preparation of solid or semi-solid samples for chromatography usually consist of

various combinations of methods of mincing, shredding, grinding, pulverizing and/or pressurizing of the sample to disrupt sample architecture. This step is usually followed by the addition of solvents, acids, bases, buffers, abrasives, salts, detergents and/or chelators in an effort to more completely disrupt cellular and architectural composition and initiate the extraction and fractionation of various sample components from the analyte(s) of choice. At this point in the preparation the sample may need to be filtered, centrifuged or otherwise treated to separate sample “debris” prior to further processing. In some of these steps, the analyst may encounter the creation of often intractable emulsions as a consequence of these actions. In many cases repeated centrifugation, re-extraction and sample manipulation may be required to render the sample suitable for application to an SPE column.

MSPD has been found to eliminate these complications in dealing with solid and semi-solid samples. This is achieved by literally combining the sample directly with the bonded-phase or other solid support, simultaneously accomplishing several steps in the more classical approach to sample preparation described above while producing a unique SPE extraction/fractionation column for isolation of target analytes.

2. MSPD extraction

Thus, in the MSPD process, a sample (liver, fruit, etc.) is placed in a glass or agate mortar containing an appropriate bonded-phase or other solid support material, such as octadecylsilyl (ODS)-derivatized silica (C_{18}) or other suitable support. The solid support and sample are manually blended together using a glass or agate pestle, a step that takes about 30 s. Internal standards or spikes may be added prior to this step. The blended material is then transferred and packed into a column suitable for conducting sequential elution with solvents.

The entire length of the column consists of blended sample components and their distribution in the bonded-phase and support, producing a new phase that exhibits unique character for sample fractionation. In this manner, an appropriate solvent or a sequence of solvents may be used to clean the column or directly isolate the compound(s) of choice. Co-columns, consisting of other solid phase or chromatographic supports, may also be incorporated into the column to assist in analyte isolation or further clean-up (Fig. 1).

2.1. Factors to consider in performing a MSPD extraction

Several factors that have been examined for their effect in conducting MSPD extractions include 1) the effect of average particle size diameter. As expected, very small particle sizes (3–10 μm) lead to extended solvent elution times and the need for excessive pressures or vacuum to obtain adequate flow. A blend of silicas possessing a range of particle sizes (40–100 μm) works quite well, and such materials also tend to be less expensive. 2) Non-end-capped vs. end-capped materials or materials having a range of carbon loading (8–18%), 3) the character of the bonded-phase. Depending on the polarity of the phase chosen, rather dramatic effects on the results may be

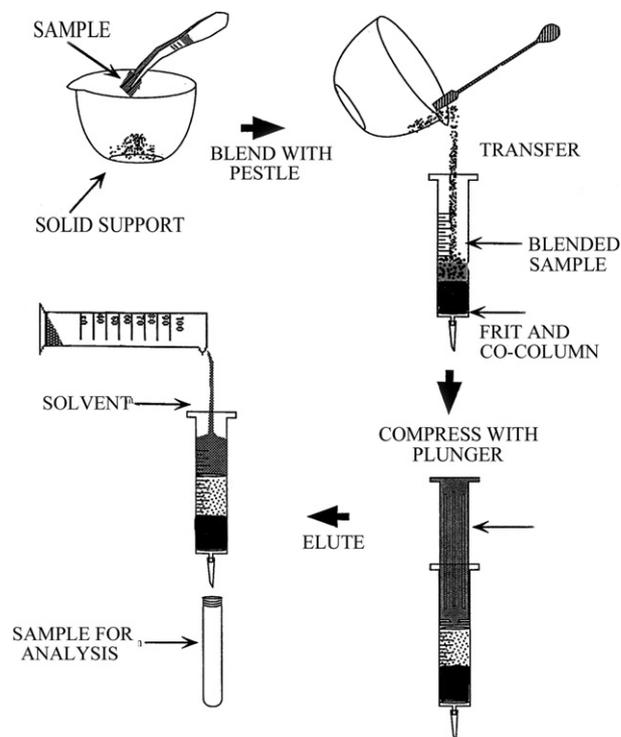


Fig. 1. Steps in a typical MSPD extraction.

observed. Applications requiring a lipophilic bonded-phase may use C_{18} and C_8 materials interchangeably. 4) The use of underivatized silica or other solid supports. Use of non-modified or underivatized solids, such as sand, to blend samples do not work in exactly the same manner as originally described for bonded-phase solid supports, such as ODS. However, the same basic principles will apply; abrasion and sample disruption will occur during the blending process. However, the further disruption of the sample and component dispersion will only occur to the degree that the components interact with the chemical characteristics of the particulate surface and each other. All surfaces have a definable chemistry and many substances, including a variety of minerals, may well serve to enhance isolation of specific compounds or classes of compounds and may even be blended together to form unique interactions to accomplish desired results. To date, silica-based support materials (derivatized silica, silica gel, sand, Florisil) have been almost exclusively reported for use in MSPD. One recent report has demonstrated the use of an activated carbon fiber for the isolation of dithiocarbamates from fruits, vegetables and cereals [82] while another has reported the isolation of DDT [1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)] from fat samples [19]. Two others have reported the use of polymeric materials (XAD-7) for sample dispersion and extraction of atrazine [151] or the antibiotic avoparcin [134]. The blending of samples with alumina has also been used in several extraction methods [10,77,140]. The further use and effect of synthetic polymer-based solid supports and of granular minerals is a subject for further study, particularly of supports that possess unique surface and/or pore chemistries, such as hydrophobic interaction supports. For silica-based materials, however,

studies have shown that the pore size is of minor importance in MSPD. This effect could vary with the sample and should, nonetheless, be considered. 5) the best ratio of sample to solid support material. The most often applied is 1 to 4, respectively, but has varied from application to application. Most protocols use lipophilic bonded-phase (C_{18} , C_8) materials, blending 2.0 g of solid support with 0.5 g of sample. This ratio is dependent on the application and must be examined as a major variable during method development. Both smaller and greater ratios have been used successfully. 6) Chemical modification of the matrix or matrix solid support blend. Addition of chelating agents, acids, bases, etc. at the time of blending affect the distribution and elution of target analytes from the sample. The elution profile of matrix components is likewise affected. 7) The optimum choice of elution solvents and the sequence of their application to a column. Elution solvent sequence attempts to isolate the analyte or further clean the column of interfering substances with each solvent step. MSPD columns permit isolation of different polarity analytes or entire chemical classes of compounds in a single solvent or in differing polarity solvents passed through the column, making MSPD amenable to conducting multi-residue isolation and analysis on a single sample. Several recent studies have reported the use of hot water as an eluting solvent as well as the addition of pressure, a process known as pressurized-liquid extraction (PLE) or accelerated solvent extraction (ASE) [12,27,45,57,83,106,107,134]. Such applications demonstrate the potential to make extraction methods based on MSPD free of hazardous solvents and even less expensive to perform. Preconditioning of the support materials used for any MSPD application enhances analyte recovery and speeds the process of sample blending and dispersal. This is due to the breaking of surface tension differences that may exist between the sample and bonded-phase solid support. As with SPE, washing or rinsing the solid support materials also eliminates contaminants from the final eluates. 8) The elution volume. It has been observed that for an 8 ml elution of a 2 g MSPD column blended with 0.5 g of sample that target analytes usually elute in the first 4 ml, approximately one column volume. This will vary for each application and should be examined to reduce the use of solvent and the unintended co-elution of potential interferences. Miniaturization of the MSPD technique, using smaller sample sizes and proportionately less support or solvent [56,65,146,149], as well as the potential for on-line LC methods, using valve switching to accomplish elution and concentration of the sample, may permit the overall use of less solvent and the opportunity for automation and 9) the effect of the sample matrix itself. All of the components of the sample are dispersed throughout the column, covering much of the bonded-phase solid support surface, creating a new phase that can have dramatic effects on isolation in going from one matrix to another [1,159,161,163,199,226,231,236,237,246].

The eluates obtained in MSPD may be taken directly to instrumental analysis, being adequately “clean” for direct injection. However, in some cases additional steps are required to remove co-eluting matrix components. This may involve a more classical SPE approach, using a second solid phase material, co-column or a separate column technique. For

example, bonded-phase or other support materials of varying character may be packed at the bottom of the MSPD column (co-column). Alternatively, the MSPD column may be eluted directly onto a standard SPE column or disc material. Some studies have applied a variety of such approaches, including liquid–liquid extraction. However, the advent and availability of LC/MS and MS/MS instrumentation make many of these extra steps unnecessary by simultaneously enhancing sensitivity and selectivity.

3. MSPD and SPE

MSPD has been found to be physically and functionally different from classical SPE in several ways: 1) it accomplishes complete sample disruption and dispersal onto particles of very small size, providing an enhanced surface area for subsequent extraction of the sample. In SPE sample disruption must be conducted as a separate step in preparing samples for SPE and many of the sample components must be discarded in the process of making the sample suitable for addition to an SPE column. 2) In SPE the sample is usually absorbed onto the top of the column packing material, not throughout the column as in MSPD. 3) The physical and chemical interactions of the components of the system are greater in MSPD and different, in many respects, from those seen in classical SPE or other forms of liquid chromatography. These have been previously reviewed [159,161,163,199,226,231,236,237,246] and encompass the interaction of a) the sample components with the solid support, b) the sample components with the bonded-phase, c) the analyte with the solid support, d) the analyte with the bonded-phase, e) the analyte with the dispersed sample components, f) all of the above interacting with the elution solvent(s) and their sequence of addition and g) the dynamic interactions of all of the above occurring simultaneously. Nonetheless, general chemical principles involved in conducting SPE and other forms of chromatography are also operable in applying MSPD. Thus, the chemical composition and characteristics of the solid support and bonded-phase are expected to affect the retention and elution of the analytes. These same properties will also apply to the dispersed sample components and the unique phase that is created.

4. Conclusions

The list of representative MSPD applications for the isolation of an assortment of compounds from a variety of matrices (Table 1) illustrates the rather generic character of MSPD. Where examined, MSPD has been found, in many cases, to provide equivalent or superior results to older official methods conducted by more classical countercurrent extraction and/or SPE techniques. Further, it has been rather consistently observed that MSPD requires approximately 95% less solvent and can be performed in 90% less time when compared to such classical methods. The use of smaller sample sizes, combined with lower solvent consumption, purchase and disposal, make MSPD competitive with such methods on several levels and should be considered as an alternative when developing new

analytical protocols. This is especially the case for solid or semi-solid biological materials. The continuing development of new supports and bonded-phases and the potential for miniaturization and automation of the MSPD process suggest several areas for future uses and research.

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