6. ANALYTICAL METHODS

The purpose of this chapter is to describe the analytical methods that are available for detecting, measuring, and/or monitoring endosulfan, its metabolites, and other biomarkers of exposure and effect to endosulfan. The intent is not to provide an exhaustive list of analytical methods. Rather, the intention is to identify well-established methods that are used as the standard methods of analysis. Many of the analytical methods used for environmental samples are the methods approved by federal agencies and organizations such as EPA and the National Institute for Occupational Safety and Health (NIOSH). Other methods presented in this chapter are those that are approved by groups such as the Association of Official Analytical Chemists (AOAC) and the American Public Health Association (APHA). Additionally, analytical methods are included that modify previously used methods to obtain lower detection limits and/or to improve accuracy and precision.

6.1 BIOLOGICAL SAMPLES

Endosulfan in its pure form is a crystalline substance consisting of α- and β-isomers in the ratio of approximately 7:3. It is an organochlorine pesticide, and analysis of biological and environmental samples for endosulfan commonly results in the detection of other organochlorine pesticides and polychlorinated biphenyls. These can interfere with the determination of endosulfan unless adequate cleaning and separation techniques are used. Detection of low levels of endosulfan typically involves extraction of samples with organic solvents, a clean-up step to remove lipids and other materials that may interfere with analysis, high-resolution gas chromatography (HRGC) to separate endosulfan from other compounds in the extract, and confirmation of endosulfan by electron capture detector (ECD) or mass spectroscopy (MS). Method blanks and control samples should be used to verify method performance and to ensure that the reagents and glassware are not introducing contaminants that might interfere with the determination of endosulfan isomers or endosulfan sulfate.

The method of choice for the determination of α- and β-endosulfan in blood, urine, liver, kidney, brain, and adipose tissue is gas chromatography equipped with an electron capture detector (GC/ECD) (Coutselinis et al. 1976; Demeter and Heyndrickx 1979; Demeter et al. 1977; Le Bel and Williams 1986). This is because GC/ECD is relatively inexpensive, simple to operate, and offers a high sensitivity for halogens (Griffith and Blanke 1974). After fractionation of adipose tissue extracts using gel permeation chromatography, detection limits of low-ppb (1.2 ng/g) were achieved for endosulfan and other chlorinated pesticides using GC/ECD (Le Bel and Williams 1986).

A new technique has been developed to analyze α- and β-endosulfan concentrations in human urine (Vidal et al. 1998). Samples are mixed with a buffer solution and then passed through solid phase extraction cartridges for analysis using gas chromatography-tandem mass spectrometry (GC-MS-MS).

β-Endosulfan has also been measured in hand rinsings using GC/ECD (Kazen et al. 1974). Sample preparation involves hand rinses with hexane followed by concentration, fractionation, and clean-up with Florisil®. Sensitivity, recovery, and precision data were not reported.

Positive identification of low-ppb (μg/L) levels of endosulfan in human blood has been achieved by GC equipped with a microcoulometric detector (GC/MC) (Griffith and Blanke 1974). Although GC/MC is specific and nearly as sensitive as GC/ECD for detecting endosulfan in blood, GC/MC is more difficult to operate. Both isomers of endosulfan can be measured in blood using a method described by Guardino et al. (1996). According to the authors, endosulfan can be recovered and measured with an approximate limit of quantitation (LOQ) of 0.2 μg/L (sub-ppb).

GC/MS has been employed by Demeter et al. (1978) to quantitatively detect low-ppb levels of α- and β-endosulfan in human serum, urine, and liver. This technique could not separate α- and β-isomers, and limited sensitivity confined its use to toxicological analysis following exposures to high levels of endosulfan. More recently, Le Bel and Williams (1986) and Williams et al. (1988) employed GC/MS to confirm qualitatively the presence of α-endosulfan in adipose tissue previously analyzed quantitatively by GC/ECD. These studies indicate that GC/MS is not as sensitive as GC/ECD. Mariani et al. (1995) have used GC in conjunction with negative ion chemical ionization mass spectrometry to determine alpha- and beta-endosulfan in plasma and brain samples with limits of detection reported to be 5 ppb in each matrix. Details of commonly used analytical methods for several types of biological media are presented in Table 6-1.

6.2 ENVIRONMENTAL SAMPLES

Reliable analysis of endosulfan residue concentrations in environmental samples usually involves detection of the α- and β-isomers plus endosulfan sulfate (a degradation product of endosulfan). GC/ECD has been the most widely used analytical technique for determining low-ppb to parts-per-trillion (ppt) levels of α- and β-endosulfan and endosulfan sulfate in air, water, waste water, sediment, soil, fish, and various foods (Bennett et al. 1997; Chopra and Mahfouz 1977a; EPA 1988a, 1997a, 1997b, 1997c, 1992a; FDA 1994; Fisk 1986; Fukuhara et al. 1977; Giabbai et al. 1983; Goebel et al. 1982; Kutz et al.

Table 6-1. Analytical Methods for Determining Endosulfan in Biological Samples

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Blood	Acidification of blood sample and extraction with ether; evaporation of extract to dryness and dissolution of residue in hexane	GC/ECD	No data	65–68	Coutselinis et al. 1976
Blood	Addition of H_2SO_4 to blood sample, extraction with hexane, acetone (9:1) and extract concentration	GC/MC	10–20 µg/L (ppb)	50	Griffith and Blanke 1974
Blood	Homogenization of sample followed by extraction with methanol and centrifugation; isolation of pesticides using SPE	GC/ECD	Approximately 0.2 µg/L (ppb)	No data	Guardino et al. 1996
Blood (serum) and urine	Extraction of sample with benzene and concentration; clean-up using HPLC	GC/MS	Low/µg/L (ppb) levels	99–103% (generally 1–14% RSD; worst for serum)	Demeter et al. 1978
Plasma, brain (alpha and beta)	Brain: homogenization with ethanol, centrifugation, phase separation and evaporation of ethanol and addition of internal stardard. Plasma: extraction with hexane and then as for brain samples	GC/NICI MS	5 ng/mL for plasma (ppb); 5 ng/g (ppb) for brain; 8–31% RSD	85–93	Mariani et al. 1995
Liver, kidney, and brain	Homogenization and addition of hexane to tissue sample; evaporation of extract to dryness; dissolution of residue in hexane	GC/ECD	No data	65–68	Coutselinis et al. 1976

Table 6-1. Analytical Methods for Determining Endosulfan in Biological Samples *(continued)*

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Liver	Addition of water to sample followed by homogenization; extraction with benzene, clean-up on silica column and HPLC	GC/ECD	No data	No data	Demeter and Heyndrickx 1979
Liver	Addition of water to sample followed by homogenization an dextraction with benzene; clean-up extract on HPLC	GC/MS	Low µg/L (ppb) levels	No data	Demeter et al. 1978
Adipose tissue	Addition of acetone: hexane (15:88) to tissue followed by homogenization; clean-up extract on gel permeation and Florisil® columns	GC/ECD and GC/MS	0.0012 µg/L (ppb)	96.5 (at 0.01 µg/L)	Le Bel and Williams 1986
Hand rinsings	Rinsing of hands twice with hexane; solvent volume reduction; fractionation and clean-up on Florisal®	GC/ECD	No data	No data	Kazen et al. 1974

ECD = electron capture detector; GC = gas chromatography; HPLC = high-performance liquid chromatography; MC = microcoulometric detector; MS = mass spectrometry; NICI = negative ion chemical ionization; RSD = relative standard deviation; SPE = solid phase extraction

1976; Marsden et al. 1986; Mitchell 1976; Musial et al. 1976; Noroozian et al. 1987; Pokharker and Dethe 1981; Woodrow et al. 1986; Zoun et al. 1987). Both GC and high performance liquid chromatography (HPLC) have been used to separate endosulfan and its major metabolites endosulfan ether, endosulfan sulfate, endosulfan lactone, and endosulfan diol (Kaur et al. 1997).

Solid phase micro extraction (SPME) is a techniques in which a silica fiber coated with a thin film of polymer is brought into contact with an aqueous matrix where the organics in solution partition onto the fiber. The fiber is subsequently placed into the injector of a GC where the heat causes the release of analyte onto the column. This has been applied to endosulfan (α- and β-) and endosulfan sulfate in water with limits of detection of less than 0.3 µg/L reported (Magdic and Pawliszyn 1996).

Measurements of endosulfan in air are made on samples forced through a collection device. The sample is extracted with an organic solvent, followed by clean-up using column chromatography. GC, GC/MS, and GC/ECD have been used to analyze endosulfan in air samples. Method TO-4 (EPA 1988a) uses the adsorption of the pesticides onto polyurethane foam in a high-volume sampler with subsequent extraction and analysis using GC/ECD. Sensitivities on the order of 1 ng/m^3 were reported. Kutz et al. (1976) used a polyethylene glycol impinger for sample collection of ambient air from several sites throughout the U.S. Extraction and clean-up were followed by quantitative analysis by GC/ECD. The lower limit of detection using this method was 0.001–0.01 µg/m^3.

GC/ECD or a halogen-specific detector (HSD) (Method 8080) is the technique recommended by EPA's Office of Solid Waste and Emergency Response for determining α- and β-endosulfan and endosulfan sulfate in water and waste water at low-ppb levels (EPA 1986a). At these low concentrations, identification of endosulfan residues can be hampered by the presence of a variety of other pesticides. Consequently, sample clean-up on a Florisil® column is usually required prior to analysis (EPA 1986a).

Methods 508, 508.1, and 525.2 (EPA 1997a, 1997b, 1997c) are applicable to drinking water and ground-water and can determine α- and β-endosulfan and endosulfan sulphate at concentrations as low as 7 ppt using liquid solid extraction (LSE) and GC/ECD.

GC/ECD and GC/MS (EPA Method 608) are the methods recommended for determining α-endosulfan, β-endosulfan, and endosulfan sulfate in municipal and industrial discharges (EPA 1991a). Sample clean-up on Florisil® column and an elemental sulfur removal procedure are used to reduce or eliminate interferences. Sensitivity is in the sub-ppb range. Recoveries and precision are good.

A procedure has been developed for the analysis of α- and β-endosulfan and endosulfan sulfate in fish, water, and sediments (Chau and Terry 1972; Musial et al. 1976). This procedure involves the acetylation of endosulfan residues into their diacetates and subsequent quantification by GC/ECD. Detection limits of low-ppb levels of endosulfan were reported. This approach is rapid and simple, and minimum sample preparation is required (Chau and Terry 1972; Musial et al. 1976).

Numerous methods have also been reported for foods, including milk (Bennett et al. 1997), chili fruits (Pokharkar and Dethe 1981), fruits and vegetables (Mitchell 1976), and the multiresidue methods for fatty and non-fatty foods (fruits, vegetables, seeds, dairy, eggs, meats) published by FDA (FDA 1994). Limits of detection are generally in the sub-ppm to ppb range.

Dreher and Podratzki (1988) developed an enzyme immunoassay technique for detecting endosulfan and its degradation products (i.e., endosulfan diol, endosulfan sulfate, endosulfan ether, and endosulfan lactone) in aqueous media. The enzyme immunoassay technique is based on detecting antibodies raised against the diol of endosulfan by immunizing rabbits with an endosulfan-hemocyanin conjugate. Minor problems were encountered with coupling of the detecting enzyme (peroxidase) to the conjugate and with cross-reactivity with the pesticide endrin. Although the enzyme immunoassay technique does not require sample extraction, and it is rapid and inexpensive, it is not yet in common use in environmental residue analysis. A detection limit of 3 µg/endosulfan/L of sample was achieved (Dreher and Podratzki 1988; Frevert et al. 1988). Immunoassays have also been reported for endosulfan (both isomers), endosulfan sulfate, and endosulfan diol in water and soil (Lee et al. 1997a, 1997b) with limits of detection reported to be 0.2 µg/L for water and 20 µg/kg in soil. Details of commonly used analytical methods for various environmental media are presented in Table 6-2.

6.3 ADEQUACY OF THE DATABASE

Section 104(i)(5) of CERCLA, as amended, directs the Administrator of ATSDR (in consultation with the Administrator of EPA and agencies and programs of the Public Health Service) to assess whether adequate information on the health effects of endosulfan available. Where adequate information is not available, ATSDR, in conjunction with the National Toxicology Program (NTP), is required to assure the initiation of a program of research designed to determine the health effects (and techniques for developing methods to determine such health effects) of endosulfan.

Table 6-2. Analytical Methods for Determining Endosulfan in Environmental Samples

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air	Pumping of air through glass fiber filter and polyurethane foam plugs for collection; extraction of plugs with petroleum ether and filters with dichloromethane followed by hexane reflux; clean-up on either Florisil/ alumina or silicic acid column	GC and GC/MS	No data	No data	Bidleman 1981
Air	Collection on polyethylene glycol impinger and extraction with hexane; clean-up on Florisil column	GC/ECD	0.001–0.01 μg/m^3	No data	Kutz et al. 1976
Air	Collection of pesticide onto polyurethane foam using high volume sampler; extraction of PUF using 5% ether in hexane; extract volume reduction; column clean-up	GC/ECD	Approximately 1 ng/m^3	>75%	EPA 1988a (Method TO-4)
Drinking water	Extraction of water with methylene chloride, removal of water from extract, volume reduction to 5 mL after solvent exchange to methyl-*t*-butyl ether	GC/ECD	α-endosulfan: 0.015 μg/L (ppb); β-endosulfan: 0.024 μg/L; endosulfan sulfate: 0.015 μg/L	α: 87 (10% RSD) β: 92 (11% RSD) sulfate: 102 (15% RSD)	EPA 1997d (Method 508)
Drinking water	Extraction of water using C_{18} extraction disks (LSE); elution using ethyl acetate and methylene chloride; volume reduction	GC/ECD	< 0.007 μg/L (α, β, and sulfate)	88–106, (12–29% RSD) at 0.03 μg/L	EPA 1997e (Method 508.1)
Drinking water	Extraction of sample using LSE; solvent elution; volume reduction	GC/MS	0.07 to 0.11 μg/L using ITMS (α, β, and sulfate)	116–128	EPA 1997f (Method 525.2)
Water	Passage of samples through XAD-4 resin column and extraction with methylene chloride; clean-up extract with HPLC	GC/ECD	0.00001 μg/L	65.5 (at 0.01 μg/L	Woodrow et al. 1986
Water	Extraction of sample with methylene chloride	GC/MS	10 μg/L	87 (α-endosulfan); 107 (β-endosulfan II); 71 (endosulfan sulfate)	Eichelberger et al. 1983

Table 6-2. Analytical Methods for Determining Endosulfan in Environmental Samples *(continued)*

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Water	Extract ion of sample with toluene	GC/ECD	0.5 µg/L (α-endosulfan); 0.5 µg/L (β-endosulfan); 25 µg/L (endosulfan sulfate)	99 (α-endosulfan); 99 (β-endosulfan); no data (endosulfan sulfate)	Zoun et al. 1987
Water	Adjustment of pH of sample to near neutral and extraction with methylene chloride; volume reduction; clean-up sample on Florisil column	GC/ECD	0.5 µg/L (α-endosulfan); 0.1 µg/L (β-endosulfan); 0.1 µg/L (endosulfan sulfate)	No data	Marsden et al. 1986
Water	Development of antibodies against the diol of endosulfan and its degradation products	EIA	3 µg/L	No data	Dreher and Podratzki 1988; Frevert et al. 1988
Water	SPME of water; thermal transfer to GC	GC/ECD	α: 0.3 µg/L (ppb) β: 0.4 µg/L (ppb) Sulfate: 0.05 µg/L (ppb)	No data	Magdic and Pawliszyn 1996
Water, soil	Analysis of water directly; extraction of soil with methanol followed by dilution of extract with water	Immunoassay (Total endosulfan including endosulfan sulfate and endosulfan diol)	water: 0.2 µg/L (ppb) soil: 20 µg/kg (ppb)	No data	Lee et al. 1997a, 1997b
Water; waste water	Extraction of sample with methylene chloride and clean-up on Florisil column	GC/ECD	0.49 µg/L (α-endosulfan); 6.1 µg/L (β-endosulfan); 2.7 µg/l (endosulfan sulfate)	No data	EPA 1986c (Method 8080)
Municipal and industrial discharge	Extraction of sample with methylene chloride; water removal; exchange to hexane; volume reduction; clean-up on Florisil column and removal of elemental sulfur	GC/ECD; GC/MS	0.014 µg/L (α-endosulfan); 0.004 µg/L (β-endosulfan); 0.066 µg/L (endosulfan sulfate)	97 (α-endosulfan); 93 (β-endosulfan); 89 (endosulfan sulfate)	EPA 1991b (Method 608)

Table 6-2. Analytical Methods for Determining Endosulfan in Environmental Samples *(continued)*

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Municipal and industrial wastewater; sludge	Extraction with methylene chloride or acetonitrile and methylene chloride (depending on solids content); volume reduction and clean-up using GPC, column chromatography, or SPE; sulfur removal if needed	GC/ECD	α: 11 ng/L β: 8 ng/L sulfate: 7 ng/L	18–158	EPA 1992a (Method 1656)
Water; fish	Extraction of sample with organic solvents; derivatize to the diacetate and analyze	GC/ECD	0.005 µg/L	60–65	Chau and Terry 1972
Fish	Grinding of sample and extraction with toluene; clean-up extract on alumina column	GC/ECD	0.005 µg/L (α-endosulfan); 0.005 µg/L (β-endosulfan); 0.025 µg/L (endosulfan sulfate)	84 (α-endosulfan); 79 (β-endosulfan); 86 (endosulfan sulfate)	Zoun et al. 1987
Sediment	Extraction of sample with organic solvent; derivatize extract to the diacetate	GC/ECD	0.005–0.01 µg/g (ppm)	No data	Musial et al. 1976
Sediment; soil	Extract of sample with methylene chloride: acetone (1:1); clean-up extract using Florisil column	GC/ECD	0.002 µg/g (ppm) (α-endosulfan); 0.004 µg/g (β-endosulfan); 0.004 µg/g (endosulfan sulfate)	No data	Marsden et al. 1986
Non-fatty foods (<2% fat, > 75% water)	Extraction with acetone and removal of water with Hydromatrix; cleanup using Florisil	GC/ECD	No data	>85% (α, β, sulfate)	FDA 1994 (PAM Method 302)
Non-fatty foods (<2% fat, <75% water)	Extraction with acetonitrile, partition into petroleum ether; cleanup using Florisil	GC/ECD	No data	>85% (α, β, sulfate)	FDA 1994 (PAM Method 303)
Fatty foods (>2% fat)	Extraction of fat using sodium sulfate, petroleum ether, by filtering, or by solvents; cleanup using solvent partitioning, Florisil	GC/ECD	No data	>85% (α, β, sulfate)	FDA 1994 (PAM Method 304)

Table 6-2. Analytical Methods for Determining Endosulfan in Environmental Samples *(continued)*

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Milk	Extraction of milk with ethanol-ethyl acetate (9: 95, v/v) with sodium sulfate; centrifugation and volume reduction	GC/ELCD	α: 0.9 µg/kg (ppb) β: 0.9 µg/kg Sulfate: 1.8 µg/kg	α: 90 (5% RSD) β: 91 (11% RSD) Sulfate: 88 (11% RSD)	Bennett et al. 1997
Chili fruits	Homogenization of sample with benzene:isopropanol (3:1); clean-up extract using carbon:celite (1:1)	GC/ECD	0.005 µg per sample	96.4 (α-endosulfan); 97 (β-endosulfan); 96.2 (endosulfan sulfate)	Pokharkar and Dethe 1981
Fruits; vegetables	Homogenization of sample with acetonitrile and clean-up on Florisil column	GC/ECD	<1 ppm (mg/kg)	101.5–103.6 (α-endo-sulfan); 100–102 (β-endo-sulfan); 92.9 (endosulfan sulfate)	Mitchell 1976

GC = gas chromatography; ECD = electron capture detector; EIA = enzyme-immunoassay; GPC = gel permeation chromatography; HPLC = high-performance liquid chromatography; ITMS = ion trap mass spectrometer; LSE = liquid solid extraction; MS = mass spectrometry; RSD = relative standard deviation; SPE = solid phase extraction

The following categories of possible data needs have been identified by a joint team of scientists from ATSDR, NTP, and EPA. They are defined as substance-specific informational needs that if met would reduce the uncertainties of human health assessment. This definition should not be interpreted to mean that all data needs discussed in this section must be filled. In the future, the identified data needs will be evaluated and prioritized, and a substance-specific research agenda will be proposed.

6.3.1 Identification of Data Needs

Methods for Determining Biomarkers of Exposure and Effect. GC/ECD, GC/MS, and GC/MC are analytical techniques used for measuring endosulfan in blood, urine, hand rinses, and various biological tissues and excreta at low- and sub-ppb levels (Coutselinis et al. 1976; Demeter and Heyndrickx 1978; Demeter et al. 1977; Griffith and Blanke 1974; Guardino et al. 1996; Kazen et al. 1974; Le Bel and Williams 1986; Mariani et al. 1995; Williams et al. 1988). These techniques are sensitive for measuring background levels of endosulfan in the population and levels of endosulfan at which health effects might begin to occur. However, it should be noted that because endosulfan is used in tobacco farming, background levels of endosulfan in the population may vary considerably, especially between smokers and nonsmokers (Coleman and Dolinger 1982; WHO 1984). Although accurate and reliable methods are available for analysis of endosulfan in biological tissues and fluids, insufficient data have been collected using these techniques to correlate the concentrations of endosulfan in biological materials with environmental exposure and health effects (see Chapter 2).

Sensitive, reliable biochemical assays have been used for measuring changes in enzyme activities (e.g., aminopyrine-*N*-demethylase, aniline hydroxylase) as an indication of exposure to endosulfan in animals (Agarwal et al. 1978). Decreased red blood cells, hemoglobin, and IgG and IgM levels have also been detected in animals following exposure to endosulfan (Banerjee and Hussain 1986, 1987; Das and Garg 1981; Hoechst 1985a; Siddiqui et al. 1987b). While well documented methods exist to monitor these parameters, they are not specific for endosulfan exposure (see Chapter 2). However, if used in combination with measurements of endosulfan and its metabolites in biological tissues and excreta, one or more of these enzymatic and blood changes may prove to be useful biomarkers of exposure and effect. There is a need for further research to correlate specific levels of endosulfan in biological media with known biochemical changes that occur on exposure to endosulfan.

Methods for Determining Parent Compounds and Degradation Products in Environmental Media. GC/ECD is the most prevalent analytical method for measuring low levels of α- and

β-endosulfan and endosulfan sulfate in water, waste water, soil, sediment, and foods (Bennett et al. 1997; Chopra and Mahfouz 1977a; EPA 1986a, 1988a, 1991a, 1992; FDA 1994; Fisk 1986; Fukuhara et al. 1977; Giabbai et al. 1983; Goebel et al. 1982; Kutz et al. 1976; Marsden et al. 1986; Mitchell 1976; Noroozian et al. 1987; Pokharker and Dethe 1981; Woodrow et al. 1986; Zoun et al. 1987). This technique is sensitive for measuring background levels of endosulfan in foods and water (media of most concern for potential human exposure to endosulfan) and levels of endosulfan at which health effects might begin to occur. The chronic oral MRL is 0.002 mg/kg/day, which translates to a required LOD of 0.07 mg/L, and these methods easily meet that need. GC/ECD or HSD is the method (Method 8080) recommended by EPA (1986a) for detecting α- and β-endosulfan and endosulfan sulfate in water and waste water at low-ppb levels. GC/ECD has also been used to detect low-ppb levels of α- and β-endosulfan and endosulfan sulfate in foodstuffs, soil, and sediment.

An enzyme immunoassay technique has been employed for measuring endosulfan and its degradation products (i.e., endosulfan diol, endosulfan sulfate, endosulfan ether, and endosulfan lactone) in water at 3 ppb (Chau and Terry 1972; Musial et al. 1976). However, this technique is not currently in use in environmental residue analysis. Further research into this technique could produce a rapid, reliable, and sensitive method for identifying contaminated areas posing a risk to human health. No additional methods for detecting endosulfan in environmental media appear to be necessary at this time. However, methods for the determination of endosulfan degradation products are needed.

6.3.2 Ongoing Studies

Researchers at the University of Florida Department of Food Science and Nutrition are evaluating the use of liquid solid extraction for the determination of endosulfan and other pesticides in seawater and fish (FEDRIP 1999). At the U.S. Department of Agriculture in Beltsville, Maryland, scientists are studying the transport, deposition, and degradation of pesticides, including endosulfan. They stress the importance of measuring both the α-and β-isomers because of the transformations (FEDRIP 1999). A technique using solid phase extraction to sample water for endosulfan concentrations is being developed by the University of Florida (FEDRIP 1999). The storage stability and transportability of endosulfan absorbed by the disks will be studied to determine analytical efficiency. The Eastern Regional Research Center in Wyndmoor Pennsylvania is developing advanced technologies for the analysis of endosulfan in meat, poultry and eggs (FEDRIP 1999). This technique will include the use of a supercritical fluid extractor in order to reduce the amount of organic solvent use and to speed up extraction times.