7. ANALYTICAL METHODS

The purpose of this chapter is to describe the analytical methods that are available for detecting, measuring, and/or monitoring parathion, its metabolites, and other biomarkers of exposure and effect to parathion. 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.

7.1 BIOLOGICAL MATERIALS

Parathion was widely used for agricultural purposes, which may have resulted in human exposure during its application, and residues on or in foods can result in exposure to humans by ingestion. All use of parathion has been cancelled in the United States to mitigate the risk of human exposure (EPA 2000). Methods for the determination of parathion in biological samples can be used to verify that exposure and absorption has occurred. Table 7-1 lists the applicable analytical methods for determining parathion in biological fluids and tissues.

The principal method used for the detection of parathion or its metabolites in biological samples is gas chromatography (GC) using a flame photometric detector (FPD), a mass spectroscopy (MS) detector, or an electron capture detector (ECD). The preparation of samples usually involves variations of solid-phase extraction (SPE), and/or liquid/liquid extraction with organic solvents.

García-Repetto et al. (2001) reported a method for parathion identification and quantification in human blood using SPE, GC-nitrogen phosphorus detection (NPD) analysis followed by GC-MS confirmation. The average recovery of parathion in blood is 96.1%, which is in the acceptable range established by the EPA. The LOD and LOQ reported in the study are 1.21 and 4.03 μg/L, respectively. This method has improved a previous method that involved liquid-liquid extraction with *n*-hexane and benzene resulting in more complex chromatograms. Not only is the method more precise, it also eliminates hazardous waste emissions and exposure of technicians to toxic solvents.

Table 7-1. Analytical Methods for Determining Parathion and Transformation Products in Biological Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Human blood	Addition of 1 mg/L azobenzene, 0.2 g ammonium sulfate and 2 mL 0.1 M sulfuric acid to a 0.5 mL sample of blood. Mixture is sealed and heated in a vial. Samples are collected by HS-SPME.	GC/MS	0.02 µg/g	Absolute recovery compared to a methanolic solution: 4.7%	Musshoff et al. 2002
Human blood	Extraction with methanol and triphenylphosphate followed by dilution. Silica gel SPE with C_{18} cartridges.	GC/NPD; GC/MS	1.21 µg/L	96.1% (1.29% RSD)	García-Repetto et al. 2001
Human urine (*p*-nitrophenol)[b]	Hydrolysis with β-glucuronidase, solid phase extraction, liquid/liquid extraction, and evaporation.	RP-HPLC-MS/MS	0.1 ng/mL urine	106% (1.4% RSD) low dose; 94% (2.2% RSD) high dose	Olsson et al. 2003
Rat urine (*p*-nitrophenol)	Acid hydrolysis followed by extraction with diethyl ether and redissolve in methanol.	HPLC/UV	12 ng/mL	89% (<11% CV)	Chang et al. 1997
Bovine liver, rumen content (partially digested grain and vegetation mixture)	Extraction of homogenized sample with methanol-dichloromethane (10–90, v/v) followed by gel permeation chromatography and silica gel solid phase extraction clean-up.	GC/FPD	0.01–0.05 µg/g using 5 g sample	Rumen content: 99% (2% RSD) at 0.1 µg/g; liver: 103 (6% RSD) at 0.05 µg/g	Holstege et al. 1991
Animal fat	Sweep codistillation, Florisil clean-up elution with methylene chloride-light petroleum-acetonitrite (50:48.5:1.5).	GC/FPD	No data	No data	Brown et al. 1987

[a]Parathion is the target analytes unless otherwise specified.
[b]Note that *p*-nitrophenol is also a metabolite of methyl parathion and nitrobenzene.

CV = coefficient of variation; FPD = flame photometric detector; GC = gas chromatography; HPLC = high-performance liquid chromatography, HS = head space, MS = mass spectrometry; MS/MS = tandem mass spectrometry, NPD = nitrogen phosphorus detector; RP-HPLC = reverse phase high-performance liquid chromatography, RSD = relative standard deviation; SPE = solid-phase extraction; SPME = solid-phase microextraction; UV = ultraviolet

A method for the rapid quantification of parathion metabolite, *p*-nitrophenol, in human urine using liquid chromatography/electrospray ionization-tandem mass spectrometry has been published (Olsson et al. 2003); however, this analyte is not unique to parathion since it is also a metabolite of methyl parathion and nitrobenzene.

Parathion was determined in bovine liver and rumen content by GC/FPD by Holstege et al. (1991) using a method with an LOD reported to be 0.01–0.05 μg/g using a 5-g sample. Recoveries were reported to be 99% from rumen content and 103% from liver. Brown et al. (1987) used GC/FPD and sweep codistillation to determine parathion in animal fat. No recovery or LOD information was given.

7.2 ENVIRONMENTAL SAMPLES

Table 7-2 lists the methods used for determining parathion and its degradation products in environmental samples. The principal separation and detection methods of parathion and degradation products in environmental samples include GC or high performance liquid-chromatography (HPLC), in conjunction with MS, NPD, or FPD. Sample preparation methods vary depending on the sample matrix (Driss et al. 1993; OSHA 1986; USGS 2002). The method of Leoni et al. (1992) is applicable to both parathion and paraoxon. The NIOSH (1994) method has been fully validated for use in occupational settings where regulatory exposure limits are of concern.

Many methods were reported for the determination of parathion in water. Sample preparation methods include either some form of liquid/liquid extraction or the use of SPE, usually C_{18}-silica, for isolation of parathion residues. Mattern et al. (1991) reported an LOD for parathion in surface water of 0.005 ppb using GC in conjunction with chemical ionization ion trap MS. An LOD of 0.025 μg/L was reported for degradation product paraoxon in water with a recovery of 87% (2% relative standard deviation [RSD]) by Seiber et al. (1990). SPE provides an easy method to isolate residues and can greatly reduce the amounts of solvent used in sample preparation. Driss et al. (1993) preconcentrated parathion from drinking water onto C_{18}-silica or polystyrene-divinylbenzene co-polymer with a subsequent backflush onto an HPLC column (ultraviolet [UV] detection). LODs as low as 0.03 μg/L were reported. Kwakman et al. (1992) preconcentrated parathion from drinking water onto C_{18}-SPE disks and eluted the adsorbed compounds directly into a GC pre-column. Detection was by NPD and excellent LODs (20 pg/L) and recoveries (>95% with <4% RSD at 200 pg/L) were reported. Lebel et al. (1979) developed a method using macroreticular XAD-2 resin to isolate and concentrate parathion from drinking water at the ng/L level.

Table 7-2. Analytical Methods for Determining Parathion and Transformation Products in Environmental Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air	Preconcentration of pesticide onto OVS-2 tube (13-mm) quartz filter, XAD-2, 270 mg/140 mg. Elution with 90% toluene/10% acetone.	GC/FPD (NIOSH Method 5600)	0.0004 mg/m^3 (400 ng/m^3) for 240 L sample	92% (2.1% RSD at 1.2 µg) (0.005 µg/m^3, 240 L sample)	NIOSH 1994
Air	Air is drawn through a glass tube with a glass fiber filter and XAD-2 adsorbent. The samples are desorbed with toluene.	GC/FPD	3.1 µg/m^3	96.7% (2.9% CV)	OSHA 1986
Drinking water	Extraction with Amberlite XAD-2 resin from 100 L water. Elution with 15% acetone/85% hexane.	GC/NPD; GC/MS (SIM)	1 ng/L (ppt)	95% (±2% RSD at 100 ng/L); 102% (±1% RSD at 10 ng/L)	Lebel et al. 1979
Drinking water	Preconcentration onto 5 µm C_{18}-silica or 7 µm polystyrene-divinyl benzene co-polymer with subsequent backflush onto analytical HPLC column.	Reverse-phase-HPLC/UV (254 nm)	0.03–0.06 µg/L (ppb)	91% (±10% RSD) at sample volumes up to 300 mL	Driss et al. 1993
Drinking water	Preconcentration of 2.5 mL water onto C_{18} extraction disks, rinsing with additional 1 mL and purging disk with gas to remove residual water. Elution with ethyl acetate directly onto GC pre-column with solvent venting.	GC/NPD	20 pg/mL	>95% (<4% RSD at 200 ppt)	Kwakman et al. 1992
Surface water	Adsorption of pesticides from 2 L of water onto XAD-2 and XAD-7 resins. Elution with methylene chloride, water removal, and use of K-D to reduce volume.	GC/chemical ionization ion trap MS	0.005 µg/L	109.3% (3.4% CV) at 1 ppb level	Mattern et al. 1991
Water	Filtration using glass-fiber filters followed by SPE. Elution of dry SPE columns with ethyl acetate then evaporation.	GC/FPD (Method O-1402-01)	0.012 µg/L	81% (14% RSD at 0.02 ppb)	USGS 2002a
Water	Extraction with methylene chloride for 6 hours. Evaporation of solvent followed by solvent exchange to ethyl acetate.	cap. GC/FPD (Method O-3402-03)	0.015 µg/L	77% (15% RSD at 0.02 ppb)	USGS 2002b

Table 7-2. Analytical Methods for Determining Parathion and Transformation Products in Environmental Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Water (paraoxon)	Filtration of 1 L of water followed by extraction 3 times with 100 mL methylene chloride after addition of 20 g sodium sulfate. Concentration using K-D and solvent exchange to benzene. Concentrations done under nitrogen. Fractionation by HPLC.	GC/ECD (HECD-N mode)	0.025 µg/L	87% (2% RSD)	Seiber et al. 1990
Water	SPME of filtered water sample; thermal desorption of diazinon from SPME fiber.	GC/AED	1 µg/L with carbon line (193 nm); 3 µg/L with S line (181 nm)	No data (precision 8–12 RSD)	Eisert et al. 1994
Water	Extraction of analytes from water using SPE; elution with ethyl acetate (108 µL) directly onto retention gap with solvent venting.	GC/AED	1 ng/L (100 mL sample) with P channel	92% (7% RSD) at 5 µg/L	Hankemeier et al. 1995
Water	UV activation of 1 mL water containing 5 µg of antiparathion polyclonal antibody (APA). UV-assisted absorption of APA onto QCM. Mix parathion solution with BSA solution to form a complex that will interact with the antibody.	QCM	4 ppb	No data	Funari et al. 2013
Industrial and municipal waste water	Extraction of 1 L of sample with 60 mL methylene chloride 3 times. Water removal from extract and solvent exchange to hexane during K-D concentration.	GC/FPD or thermionic detection (P-mode); GC/MS for qualitative identifications recom-mended. (Method 1657)	10 ng/L	61–121% (10% RSD)	EPA 1993a

Table 7-2. Analytical Methods for Determining Parathion and Transformation Products in Environmental Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Waste water	Extraction of 1 L of water with 15% methylene chloride in hexane using a separatory funnel. Concentration using K-D. Cleanup (if needed) by Florisil fractionation or acetonitrile partition.	GC/FPD (P-mode) or GC/ thermionic detection. GC/MS for qualitative compound identification recom-mended. (Method 614)	0.012 µg/L	102% (4.1% RSD)	EPA 1993b
Bed sediment (lake and stream), aqueous suspended sediment and soil	Extraction with Soxhlet apparatus of minimum 25-g equivalent dry-weight samples using 350 mL dichloromethane and 25 mL methanol (93:7). Concentration and filtration of extract. Elution with dichloro-methane through chromato-graphic column. Concentration and resolution in ethyl acetate.	GC/FPD	0.951 ppb	76% (5% RSD)	USGS 2002c
Cucumber, lettuce, grapes	Chopping of produce and extraction with acetone/ methylene chloride/petroleum ether (1:1:1). Evaporation to dryness and redissolution in acetone and concentration.	SFC/NPD	No data	No data	Zegers et al. 1994
Green beans, lettuce, carrot, bell pepper (parathion; paraoxon)	Homogenization of produce with acetonitrile. Addition of NaCl to affect phase separation, removal of acetonitrile, water removal volume reduction, addition of deuterated internal standards.	GC/MS	0.05 µg/g (parathion); 0.15 µg/g (paraoxon)	93% (21% RSD) (parathion); 91% (17% RSD) (paraoxon)	Liao et al. 1991
Kale, endive, carrots, lettuce, apples, potatoes, strawberries	Extraction of crops with ethyl acetate and granular sodium sulfate, filtration, concentration with K-D. Sweep co-distillation cleanup for GC.	GC thermionic detector	No data for GC	No data	AOAC 1990a

Table 7-2. Analytical Methods for Determining Parathion and Transformation Products in Environmental Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Numerous non-fatty crops	Extraction with acetonitrile and partition into petroleum ether. Concentration using K-D and purification using Florisil column chromatography.	GC/KCl thermionic detector; identifications by combin-ations of gas, thin layer, and paper chroma-tography; polarographic confirmatory method	Polarographic method: 0.2 ppm based on 1 g crop in 1 mL cell	>80%	AOAC 1990a, 1990b, 1990c
Soybeans and rice	Grinding of 25-g samples and extraction with 150 mL of 2:1 acetone: methanol; filtration and reduction of volume to 100 mL. Addition of water, NaCl followed by extraction with methylene chloride (2 times); solvent evaporation and redissolution in methylene chloride:cyclohexane (1:1) and fractionation on Bio-Bead S-X3. Evaporation under N_2 stream and redissolution in 2 mL hexane.	GC/NPD or GC/MS (SIM)	Rice: 0.007 ppm soybeans: 0.04 ppm	Rice: 86.8% (1.2% RSD) at 1 ppm soybeans: 91.3% (1.2% RSD) at 1 ppm	Hong et al. 1993
Strawberries and cherries	Spike samples were sliced and homogenized.	HS-SPME	8.9 ppb in strawberries; 12.3 ppb in cherries	Strawberries: 81–86% (9–12% RSD); cherries: 77–79% (9–10% RSD)	Lambropoulou and Albanis 2003
Various fruits and vegetables	Homogenization of sample (adding water if needed) and adsorption on activated Florisil to produce a free-flowing powder. Elution with ethyl acetate or methylene chloride.	GC/NPD or FPD	5 ppb	96–103% at 0.05 mg/kg	Kadenczki et al. 1992
Various produce	Homogenization of sample and extraction with acetonitrile, filtration, addition of salt and solvent evaporation. Redissolution of residue in acetone for analysis.	GC/FPD or alkali FID	0.1 ppm	No data	Hsu et al. 1991

Table 7-2. Analytical Methods for Determining Parathion and Transformation Products in Environmental Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Various prepared foods	Blending of sample with acetone, filtration and transfer to Hydromatrix column. Elution with methylene chloride and concentration.	GC/FPD	No data	94% at 300 ppb	Hopper 1988
Apples, whole milk, olive oil, eggs	Blending of samples with acetone and extraction with dichloromethane and acetone, water removal and volume reduction. Cleanup using carbon-celite (apples), Extrelut-3 minicolumns with hexane (whole milk; olive oil), or C_{18} SPE (eggs).	GC/FPD	0.26 ng	Apples: 71%; whole milk: 85%; olive oil: 98%; eggs: 80%	Leoni et al. 1992
Apples, whole milk, olive oil (paraoxon)	Blending of samples with acetone and extraction with dichloromethane and acetone, water removal and volume reduction. Cleanup using carbon-celite (apples), or Extrelut-3 minicolumns with hexane (whole milk; olive oil).	GC/FPD	0.15 ng	Apples: 97%; whole milk: 89%; olive oil: 90%	Leoni et al. 1992
Cow's milk	Extraction of milk 3 times with 70% acetonitrile in water, filtration, removal of fat by zinc acetate addition, and partitioning with methylene chloride. Reduction of volume after drying.	GC/FPD (P-mode)	10 ppb	92.9% (2.9% RSD) at 100 ppb	Toyoda et al. 1990

Table 7-2. Analytical Methods for Determining Parathion and Transformation Products in Environmental Samples

Sample matrix[a]	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Cow's milk	Homogenization of milk, acetonitrile and ethanol followed by equilibration with a mixture of light petroleum-acetonitrile-ethanol and separation of the upper phase and elution through a solid matrix cartridge. Concentration and drying of the eluates to a residue that is dissolved.	GC/FPD	No data (0.016 MDL)	92.7% (5% RSD) at 0.8 µg/mL	Di Muccio et al. 1996
Milk	5 g of homogenized sample extracted using acetone and methylene chloride (1+1, v/v), dried, reconstituted with cyclohexane + ethyl acetate (1+1, v/v) and cleanup using GPC	GC/FPD	0.002 mg/kg	56.8–69.3%	Yang et al. 2012
Eggs	2 g of homogenized sample extracted using acetone and methylene chloride (1+1, v/v), dried, reconstituted with cyclohexane + ethyl acetate (1+1, v/v) and cleanup using GPC	GC/FPD	0.002 mg/kg	67.1–95%	Yang et al. 2012
Fish	5 g of homogenized sample extracted using acetone and methylene chloride (1+1, v/v), dried, reconstituted with cyclohexane + ethyl acetate (1+1, v/v) and cleanup using GPC	GC/FPD	0.002 mg/kg	70–89.2%	Yang et al. 2012

[a]Unless otherwise stated, parathion was determined.

AED = atomic emission detection; AOAC = Association of Official Analytical Chemists; BSA = bovine serum albumin; CV= coefficient of variation; ECD = Ni electron capture detector; EPA = U.S. Environmental Protection Agency; FID = flame ionization detector; FPD = flame photometric detector; GC = gas chromatography; GPC = gel permeation chromatography; HECD = Hall Electrolytic Conductivity Detector; HPLC = high-performance liquid chromatography; HS = head space, KCl = potassium chloride; K-D = Kuderna-Danish; MDL = method detection limit; MS = mass spectrometry; NaCl = sodium chloride; NIOSH = National Institute for Occupational Safety and Health; NPD = nitrogen phosphorus detector; OSHA = Occupational Safety and Health Administration; QCM = quartz crystal microbalance; RSD = relative standard deviation; SFC = supercritical fluid chromatography; SIM = selected ion monitoring; SPE = solid phase extraction; SPME = solid-phase microextraction; USGS = U.S. Geological Survey; UV = ultraviolet absorbance detection

An LOD of 1 ng/L was reported using GC with a nitrogen-phosphorus selective detector and by GC/MS using selected ion monitoring (SIM). Funari et al. (2013) describe the use of a photonic immobilization technique (PIT) to produce UV-activated antibodies that interact with quartz crystal microbalance (QCM) electrodes to develop an immunosensor for the detection of parathion in water, with a LOD of <4 ppb. Anti-parathion polyclonal antibodies are adsorbed to a gold electrode and activated with UV light using a custom-built femtosecond laser having a highly tunable pulse rate. The ultrashort UV (258 nm) pulses disrupt disulfide bridges of the antibody solution allowing free thiol groups to adsorb to the gold surface, causing the antibody to orient in a manner that increases antigen-antibody specific binding, thus increasing sensitivity of the immunosensor (Funari et al. 2013).

Supercritical fluid extraction (SFE) is also used in sample preparation methods. A supercritical fluid chromatography (SFC)-based method for cucumber, lettuce, and grapes (Zegers et al. 1994) was published, but did not specify the LOD or recovery.

Three standardized methods were found in the *Official Methods of Analysis of the Association of Official Analytical Chemists* (AOAC 1990a, 1990b, 1990c). The first of these methods is based on the extraction of crops (kale, endive, carrots, lettuce, apples, potatoes, and strawberries) with ethyl acetate and isolation of the residue followed by a sweep codistillation cleanup prior to GC/thermionic detection (Method 968.24). In the second method (Method 970.52), the sample is extracted with acetonitrile and the residue is partitioned into petroleum ether followed by Florisil clean-up and GC/potassium chloride (KCl) thermionic detection. Chemical identifications are based on combinations of gas, thin-layer, and paper chromatography. The recovery for parathion in this method is stated to be >80%; no data on LODs were given. The third method utilizes the same extraction and clean-up techniques as the second and then GC/FPD for detection (Method 970.53).

Some methods employ the homogenization of the plant material with aqueous acetonitrile (Hsu et al. 1991) or other polar organic solvents such as acetone/methanol mixtures (Hong et al. 1993). Phase separation is brought about with the addition of a salt. The acetonitrile approach is preferred by the California Department of Food and Agriculture because of the possible higher recoveries (see Table 7-2) (Lee et al. 1991). The advantage of acetonitrile is found in its ability to more readily solvate residues and in the ease with which the phase separation can be accomplished through the addition of salt (Lee et al. 1991). Reported LODs for parathion were typically 10–50 ppb. One of the methods eliminated any clean-up steps after the initial extraction (Hsu et al. 1991) to provide a method with a faster turnaround time with some loss in sensitivity (LOD approximately 0.1 ppm) relative to the purified samples.

Methods found for the determination of parathion in animal products also used homogenization with a polar organic solvent as the first step in residue recovery. Toyoda et al. (1990) isolated parathion from cow's milk via partition into methylene chloride after extraction of the milk with 70% acetonitrile in water. Based on GC/FPD, an LOD of 10 ppb and a recovery of 92.9% (2.9% RSD) at 100 ppb were reported. Parathion residues in eggs were studied (Leoni et al. 1992) after blending the eggs with acetone and partitioning into dichloromethane and acetone followed by C_{18}-silica SPE. Based on GC/FPD analysis, an LOD of 0.26 ng and a recovery of 80% at 13 ppb were reported.

7.3 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 parathion is available. Where adequate information is not available, ATSDR, in conjunction with 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 parathion.

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.

7.3.1 Identification of Data Needs

Methods for Determining Biomarkers of Exposure and Effect.

Exposure. Section 3.8.1 provided information on biomarkers used to identify or quantify exposure to parathion. Some methods for the detection of the parent compound in biological samples were described above. The parent chemical is quickly metabolized so the determination of metabolites can also serve as biomarkers of exposure. The use of GC coupled with MS has been reported for the elucidation and confirmation of parathion in biological samples (Musshoff et al. 2002). The most specific biomarkers will be those metabolites related to *p*-nitrophenol. Methods for the detection of this compound in human urine have been reported (Olsson et al. 2003). A method for *p*-nitrophenol in rat urine has been described

by Chang et al. (1997) with reported sensitivities in the sub-ppm range. Further studies designed to refine the identification of metabolites specific to parathion and provide dosimetric data would be useful in the search for a more dependable biomarker of parathion exposure.

Effect. Significant decreases in plasma cholinesterase and erythrocyte (red blood cell) activities indicate possible exposure to insecticidal organophosphorus compounds (see Chapter 3). Rapid, simple, and specific methods should be sought to make assays readily available to the clinician. Nonspecific biomarkers of effect exist, but future studies to determine specific biomarkers of effect would be useful.

Methods for Determining Parent Compounds and Degradation Products in Environmental Media. Human exposure to parathion may have occurred via inhalation of ambient air; ingestion of contaminated food and water; and dermal uptake through occupational and non-occupational contact with contaminated soils, surface water, and commercial preparations. Methods have been reported for the measurement of parathion in various foods, soils, sediment, waste water, drinking water, and air. The method of OSHA (1986) (LOD 3.1 $\mu g/m^3$) and NIOSH (1994) (LOD 400 ng/m^3) are adequate for the determination of parathion in air. If a 70-kg individual is assumed, method LODs of 0.007 mg/L (7 ppb) and 0.007 mg/kg (7 ppb) in water and foods, respectively, are required for the method to be adequate at the oral intermediate MRL. All of the methods for detection of parathion in water shown in Table 7-2 are adequate. With regard to foods, the methods of Kadenczki et al. (1992) and Leoni et al. (1992) for detection of parathion are adequate. Methods for other non-fatty crops would need to be validated or developed if routine use were desired. Di Muccio et al. (1996) describe a quick and simple method for the determination of parathion in cow's milk; however, no data were provided on LODs. Additional methods for detection of parathion in fatty foods are needed to permit the evaluation of the residues in those fatty media.

There are also methods for the analysis of parathion degradation products in water and food. Seiber et al. (1990) reported a method for parathion and its oxon in water. Several methods were reported for the determination of parathion and paraoxon in various food products, including produce, whole milk, olive oil, and eggs (Leoni et al. 1993; Liao et al. 1991). Additional methods are needed for the quantitative analysis of parathion transformation products in environmental matrices. It would also be important to establish MRLs for the transformation products to put the analytical requirements into perspective.

7.3.2 Ongoing Studies

No ongoing studies regarding parathion detection by analytical methods were located.