6. ANALYTICAL METHODS

The purpose of this chapter is to describe the analytical methods that are available for detecting and/or measuring and monitoring 2,4,6-trinitrotoluene in environmental media and in biological samples. The intent is not to provide an exhaustive list of analytical methods that could be used to detect and quantify 2,4,6-trinitrotoluene. Rather, the intention is to identify well-established methods that are used as the standard methods of analysis. Many of the analytical methods used to detect 2,4,6-trinitrotoluene in environmental samples are the methods approved by federal 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 refine previously used methods to obtain lower detection limits, and/or to improve accuracy and precision.

6.1 BIOLOGICAL MATERIALS

Only limited data were located regarding methods of analysis for 2,4,6-trinitrotoluene and metabolites in biological samples. Methods have been developed to quantify these substances in blood, urine, tissues, and handswab samples. Details for selected methods are shown in Table 6-l.

The primary method that has been used to analyze for 2,4,6-trinitrotoluene and/or its metabolites in blood and urine is high-performance liquid chromatography (HPLC)/mass spectrometry (MS) (Yinon and Hwang 1985b, 1986b, 1986c, 1987). Blood samples are prepared for analysis by centrifuging to obtain the serum. The serum is extracted with methylene chloride and the solvent is exchanged to acetonitrile (Yinon and Hwang 1986b, 1987). A second method, in which plasma is extracted with toluene followed by solvent exchange to acetonitrile and HPLC/ultraviolet (UV) detection, had a detection limit in the ppb range, with good precision (10% relative standard deviation [RSD]) and accuracy (Army 1981b). Since no information on sensitivity or reliability was provided for the MSbased method, and no other methods for blood were located, the adequacy of the available HPLC

TABLE 6-1. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Biological Materials

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Blood	Centrifuge sample; dilute serum with water; extract with methylene chloride; centrifuge and add sodium sulfate; filter and evaporate; redissolve in methylene chloride; evaporate and redissolve in acetonitrile	HPLC/MS	NR	NR	Yinon and Hwang 1986b
Urine	Acidify sample to hydrolyze; neutralize and extract with toluene; add sodium sulfate and filter; evaporate and redissolve in acetone or acetonitrile	HPLC/MS	0.1 µg/L	90% (TNT); 30% (metabolites)	Yinon and Hwang 1985b, 1986c
Urine	Acidify sample; neutralize and extract with toluene; add anhydrous sodium sulfate	GC/ECD	≈50 µg/L	90%	Almog et al. 1983
Urine	Acidify and heat sample; neutralize and extract with diethyl ether; evaporate and redissolve in acetone; develop silica gel plate with benzene/diethyl ether/methanol	TLC/densi-tometry	100 ng/spot	83–98%	Liu et al. 1991

TABLE 6-1. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Biological Materials (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Plasma, kidney	Add sodium chloride/acetic acid solution to sample; extract with toluene; add water and evaporate organic phase; add acetonitrile containing internal standard; filter	HPLC/UV	248 µg/L (plasma); 211 ng/g (kidney)	86% (plasma); 75% (kidney)	Army 1981b
Muscle, fat	Homogenize sample; extract with acetonitrile; concentrate; add internal standard and water; filter	HPLC/UV	62 ng/g	87%	Army 1981b
Liver	Homogenize sample; add acetic acid/sodium chloride solution; extract with toluene; evaporate and redissolve in acetonitrile containing internal standard; filter	HPLC/UV	50 ng/g	49%	Army 1981b
Handswabs	Wipe hand with swab soaked in MTBE and extract with MTBE in pentane; centrifuge to remove debris; clean up on Amberlite® XAD-7 eluting with ethyl acetate	HRGC/ECD	1 ng/swab	78%	Douse 1985, 1987; Douse and Smith 1986

TABLE 6-1. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Biological Materials (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Handswabs (standards)	Wipe hand with dry swab; extract with methanol/potassium phosphate; directly inject standards	HPLC/PMDE	24 pg/inj	NR	Lloyd 1983a, 1991
Handswabs	Wipe hand with swab soaked in acetone; squeeze out acetone and concentrate	HPLC/TEA; HRGC/TEA	Low pg	NR	Fine et al. 1984
Handswabs	Swab hand with swab soaked in ethanol; extract in water/buffer solution with vortexing; add aliquots to antibody-coated micro-titer plates	ELISA	15 ng/swab	NR	Fetterolf et al. 1991

ECD = electron capture detection; ELISA = enzyme-linked immunosorbent assay; GC = gas chromatography; HPLC = high-performance liquid chromatography; HRGC = high-resolution gas chromatography; inj = injection; MS = mass spectrometry; MTBE = methyl-tert-butyl ether; NR = not reported; PMDE = pendant mercury drop electrode; TEA = thermal energy analyzer; TLC = thin-layer chromatography; TNT = 2,4,6-trinitrotoluene; UV = ultraviolet detection

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methods for determining 2,4,6-trinitrotoluene and/or its metabolites in blood could not be completely evaluated. However, HPLC/MS is known to be a highly selective and sensitive method and results with urine samples indicate that it should also be a good method for determining 2,4,6-trinitrotoluene in blood. Hydrolyzed urine samples are extracted with toluene and the solvent is exchanged to acetone prior to separation by HPLC (Yinon and Hwang 1985b, 1986c, 1987). The limited data on this method of analyzing urine show a high recovery for 2,4,6-trinitrotoluene but much lower recovery for its metabolites. The high sensitivity (the limit of detection is in the sub-ppb range) and selectivity of the MS detector compensate for the low recovery of the metabolites. Gas chromatography (GC)/electron capture detection (ECD) and thin-layer chromatography (TLC)/densitometry have also been used to detect 2,4,6-trinitrotoluene and/or its metabolites in urine. GC/ECD accurately determined the 2,4,6-trinitrotoluene metabolite, 4-ADNT, in a toluene extract of hydrolyzed human urine (Almog et al. 1983). The limit of detection for this method was in the low-ppb range with high recovery of the analyte. A modification of TLC that employed a computer-linked densitometer for detection and quantitation reliably measured 2,4,6-trinitrotoluene and its metabolites in hydrolyzed human urine (Liu et al. 1991). Advantages of this method were its rapidity and low cost, However, it is about 3-4 orders of magnitude less sensitive than HPLC/MS and requires substantially more sample.

An HPLC/UV method has been developed for determining 2,4,6-trinitrotoluene in animal kidney, muscle/fat, and liver (Army 1981b). Detection limits for these matrices were in the low-to-mid ppb, and the analyses were reproducible with RSDs of 15% or better. There were some problems with recovery of the analytes, especially in liver samples. No other methods were available for comparison.

High-resolution gas chromatography (HRGC) with ECD or thermal energy analysis (TEA) and HPLC with electrochemical detection (EC) using a pendant mercury drop electrode (PMDE) or TEA have been proposed for the detection of 2,4,6-trinitrotoluene in handswabs (Douse 1985, 1987; Douse and Smith 1986; Fine et al. 1984; Lloyd 1983a, 1991). Limited data available indicate that both HPLC and HRGC are good separation methods for this analysis. Sensitivities for all three detectors are in the pg-to-low-ng range. TEA is slightly more selective for nitroaromatics, but the PDME has the advantage of being easily renewable which reduces contamination problems. An additional method based on monoclonal antibody technology was also located (Fetterolf et al. 1991). The enzyme-linked immunosorbent assay (ELISA) had a detection limit of about 15 ng/swab and showed no

cross-reactivity with other explosives or common contaminants. Electron spin resonance spectrometry has also been tested for handswab analysis and was found to be selective and specific (Bums et al. 1987). Sensitivity was comparable to other methods and precision was high (1.8% RSD).

6.2 ENVIRONMENTAL SAMPLES

Methods have been developed to detect 2,4,6-trinitrotoluene and some of its breakdown products in air, water, soil, plant tissue, explosives, explosives residues, and postblast debris. Methods include semiquantitative screening methods that can be used in the field and quantitative laboratory-based methods. Selected methods for analysis of environmental samples are presented in Table 6-2.

The primary method of analyzing for 2,4,6-trinitrotoluene in air is by GC, usually with ECD (Andersson et al. 1983; Bishop et al. 1981, 1988; Pella 1976, 1977; Van Slyke et al. 1985). Methods based on MS, including ion dilution MS (IDMS) (St. John et al. 1975) and glow discharge MS (GDMS) (McLuckey et al. 1988), have also been used successfully to measure 2,4,6-trinitrotoluene vapor in air. For most methods, the sample is collected in a tube containing a solid sorbent and desorbed with an organic solvent. A few methods have been developed that permit direct entry of the sample into the detecting instrument. These include GDMS (McLuckey et al. 1988) and ion mobility spectrometry (IMS) (Karasek and Denney 1974; Spangler et al. 1983). The latter can be adapted to a portable instrument for field use. A field method based on TLC has also been used (Chrostowski et al. 1976), but is more time consuming and much less sensitive than IMS. The limited data on sensitivity, accuracy, and precision make it difficult to compare these parameters for the different methods. However, most of the methods will detect 2,4,6-trinitrotoluene in air at the ppb level or less. While GC/ECD is the most commonly used method, several of the other methods have distinct advantages, such as increased sensitivity (GDMS and IMS), simplicity of sample collection/preparation (GDMS and IMS), or portability (IMS).

Methods have been developed to measure 2,4,6-trinitrotoluene and/or it breakdown products in drinking water, surface water, groundwater, waste water effluents, and sea water. The two methods most frequently used to analyze water for the presence of 2,4,6-trinitrotoluene and other polynitroaromatic hydrocarbons are HPLC/UV and HRGC/ECD. In addition, methods based on

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air	Collect sample in glass wool-charcoal absorbent tubes; desorb with benzene	GC/ECD	<0.05 ppb	74–108%	Pella 1976
Air	Collect sample in Tenax®-GC or Florisil® adsorbent tubes; desorb with acetonitrile	GC/ECD	NR	96–101% (Tenax); 85–94% (Florisil)	Bishop et al. 1981
Air	Collect on Amberlite® XAD-2; desorb with toluene	GC/ECD	NR	77–87%	Andersson et al. 1983
Air	Collect sample in sampling bulb containing known amount of d_6-TNT in benzene; equilibrate for 1 hour; remove sample and wash bulb with benzene; evaporate in sample capillary	IDMS	≈0.1 ppb	NR	St. John et al. 1975
Air	Direct incorporation of sample into glow discharge chamber	GDMS	≈1.4 ppt	NR	McLuckey et al. 1988
Air	Collect sample on Chromosorb® 102 adsorbent column; desorb with acetone; visualize developed plate with diphenyl-amine in ethanol and expose to ultraviolet light	TLC	NR	NR	Chrostowski et al. 1976

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air	Direct incorporation of sample into reaction chamber	IMS	0.01 ppb	NR	Spangler et al. 1983
Incinerator emission	Collect sample on sampling train containing Amberlite® XAD-2 resin; desorb with toluene	GC/ECD	0.025 µg/mL	69–100%	Van Slyke et al. 1985
Water	Add internal standard to sample; collect sample on Amberlite® XAD-2 or XAD-4; dry column and extract with dichloromethane; dry on anhydrous Na_2SO_4; concentrate and exchange solvent to methanol	HRGC/ECD	NR	95%	Feltes et al. 1990
Water	Collect on SEP-PAK® cartridges and elute with methanol; concentrate; elute from reverse-phase column with methanol/ water	HPLC/UV	0.05–0.1 µg/L	29–79%	Army 1981a
Tap water, groundwater	Collect sample on Amberlite® XAD-4 resin; elute with ethyl acetate; concentrate	HRGC/ECD	<0.1 µg/L	95–97%	Richard and Junk 1986

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Drinking water, groundwater	Extract sample with toluene	HRGC/ECD	0.06 µg/L	102-105%	Hable et al. 1991
Waste water effluents	Add internal standard to sample; cleanup on SEP-PAK® cartridge, eluting with acetonitrile/water; centrifuge; elute from reverse-phase HPLC column with methanol/water	HPLC/UV	0.2 mg/L	75–95%	Army 1983b
Waste water, groundwater	Form membrane by dissolving polyvinyl chloride, deoctyl-phthalate, and polyoxyethylamine in tetrahydrofuran; react membrane with water sample in sealed chamber; remove membrane and analyze	Spectrophoto meter	10 µg/L	95–105%	Zhang et al. 1989
Waste water effluents, groundwater	Dilute sample with methanol/acetonitrile; filter; elute from reverse-phase column with methanol/aceto-nitrile/water	HPLC/UV	14 µg/L	101%	Army 1985b; Jenkins et al. 1986
Sea water	Add internal standard to sample; extract with benzene; evaporate; redissolve in benzene	GC/ECD	≈20 ng/L	70%	Hoffsommer and Rosen 1972

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Well water, surface water	Collect sample on Amberlite XAD-4® resin; rinse sorbent with distilled water and elute with acetone; concentrate; add methanol/water	HPLC/ED	2 µg/L	30–120%	Army 1986c
Surface water (brooks, ponds)	Collect sample on Amberlite XAD-2/4/8® resin; dry; desorb with dichloromethane; dry over anhydrous sodium sulfate; solvent exchange to methanol; concentrate; elute from reverse-phase column with methanol/water	HPLC/UV	50 ng/L	85–105%	Feltes and Levsen 1989
Surface water (lakes)	Extract with methylene chloride; cleanup on silica gel SEP-PAK® if needed; concentrate and exchange solvent to acetonitrile	HPLC/UV	6–11 µg/L	NR	Powell et al. 1983
Drinking water	Extract with toluene; inject into instrument	HRGC/ECD	NR	100–102%	Belkin et al. 1985

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Groundwater	Collect sample on Hayesep-R® solid sorbent cartridge; elute with acetone; concentrate; add internal standard; solvent exchange to methanol/water	HPLC/UV/PC	5–14 µg/L	62–82%	Army 1989b
Soil	Air dry, grind, homogenize sample; extract with acetonitrile in ultrasonic bath; dilute with aqueous $CaCl_2$; filter; elute from reverse-phase column with water/methanol	HPLC/UV	0.08 µg/g	102%	Jenkins et al. 1989
Soil	Extract with acetonitrile in ultrasonic bath; centrifuge and filter; elute from reverse-phase column with acetonitrile/water/methanol	HPLC/UV	0.1 µg/g	95–106%	Jenkins and Grant 1987
Soil	Air dry, grind; extract with acetonitrile in ultrasonic bath; add $CaCl_2$; filter	HPLC/UV	NR	70%	Bauer et al. 1990

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Soil	Stabilize soil samples at 20–30% moisture; homogenize; extract by sonication with acetonitrile; centrifuge and filter; elute from reverse-phase column with methanol/water	HPLC/UV	0.76 µg/g	99%	Bongiovanni et al. 1984
Soil	Air dry, grind, homogenize; extract with acetonitrile in ultrasonic bath; centrifuge and filter extract; elute from reverse-phase column with methanol/water	HPLC/UV	0.8 µg/g	98.2%	Army 1987c
Soil	Extract soil with methanol; filter extract; add calcium chloride and refilter; pump through indicator tube	Indicator tube	0.5 µg/g	58–70%	Army 1990b
Soil	Extract soil with acetone; filter; read background absorbance; add potassium hydroxide and sodium sulfite; filter; read absorbance and apply background correction	Spectrophoto meter	1.1 µg/g	63–96%	Army 1990b

TABLE 6-2. Analytical Methods for Determining 2,4,6-Trinitrotoluene in Environmental Samples (continued)

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Plant stems	Add sodium chloride and water to ground sample; extract with hexane/isopropanol; evaporate and add acetonitrile containing internal standard and water; filter	HPLC/UV	90 ng/g	52%	Army 1981b

$CaCl_2$ = calcium chloride; ECD = electron capture detection; ED = electrochemical detection; GC = gas chromatography; GDMS = glow-discharge mass spectrometry; HPLC = high-performance liquid chromatography; HRGC = high-resolution gas chromatography; IDMS = isotope-dilution mass spectrometry; IMS = ion-mobilization spectrometry; NA_2SO_4 = sodium sulfate; NR = not reported; PC = photoconductivity detector; TLC = thin-layer chromatography; d_6-TNT = fully deuterated TNT; UV = ultraviolet detection

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colorimetry and spectrophotometry have been used to screen field samples for 2,4,6-trinitrotoluene and other polyaromatic hydrocarbons. A method involving direct injection of water samples into a mass spectrometer proved to be fast and simple, but the high detection limit makes it useful only for screening (Yinon and Laschever 1982). In addition, MS is expensive and requires more technical training than some of the other methods used for analysis of 2,4,6-trinitrotoluene in water.

With the HPLC- and GC-based methods, sample preparation usually involves direct solvent extraction of the sample (Army 1985b; Bauer et al. 1986; Belkin et al. 1985; Hable et al. 1991; Hoffsommer and Rosen 1972; Jenkins et al. 1986; Leggett et al. 1990; Maskarinec et al. 1984; Powell et al. 1983) or concentration on a solid sorbent (Army 1981a, 1983b; Feltes et al. 1989, 1990; Maskarinec et al. 1984; Richard and Junk 1986).

HPLC/UV is the method usually employed by the Army to measure 2,4,6-trinitrotoluene in waste water effluents from munitions plants (Army 1981a, 1983b, 1985b; Bauer et al. 1986; Jenkins et al. 1986; Leggett et al. 1990). HPLC methods are relatively simple and fast, accurate, and selective. In explosives analysis, where many of the analytes are thermally-labile, they also have the advantage of not requiring heat. Sensitivity is generally in the low-to-mid ppb range. Substitution of a photodiode array detector (PAD) for the usual ultraviolet detector allowed detection of ppt in water (Feltes and Levsen 1989). HPLC with EC at a gold-mercury electrode increased sensitivity relative to UV and also improved selectivity (Maskarinec et al. 1984).

HRGC/ECD is also a sensitive and selective method for determination of 2,4,6-trinitrotoluene in water. Detection limits in the sub-ppb range are obtainable and recoveries are high (Belkin et al. 1985; Feltes et al. 1990; Hable et al. 1991; Hoffsommer and Rosen 1972; Richard and Junk 1986). In addition, precision is excellent with the RSD usually less than 10%. Detection by TEA or MS has also been used with GC with good results (Feltes et al. 1990). A comparison of these detection methods with ECD showed that while TEA was more selective than ECD for nitro compounds and had a larger linear concentration range, ECD was more sensitive than TEA by about three orders of magnitude (Feltes et al. 1990). MS was the most selective of the three detection methods, and was determined to be useful for confirmatory analysis. One disadvantage of GC methods in explosives analysis is that thermally-labile analytes may be destroyed during analysis.

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The available optical methods rely on the conversion of 2,4,6-trinitrotoluene to a fluorescent or colored complex (Army 1990b; Heller et al. 1977, 1982; Jian and Seitz 1990; Zhang et al. 1989). In general, these methods are not as sensitive or selective as the more commonly used HPLC and GC methods and they are primarily useful for simple and rapid screening of samples at field sites to determine which samples should be subjected to more intensive quantitative analysis. A recently proposed spectrophotometric method reacts a polynitroaromatic hydrocarbon-contaminated water sample with a membrane containing polyoxyethylamine. The reaction produces a colored product that can be analyzed with a spectrophotometer (Zhang et al. 1989). Since different polynitroaromatic hydrocarbons produce different absorption spectra, the method is selective as well as relatively sensitive (detection of low ppb). The method is used to screen samples in the field and, by employing fiber optics, may be useful for remote monitoring (Zhang and Seitz 1989).

An HPLC/UV method developed by the Army is the method most commonly used to analyze soils and sediments for 2,4,6-trinitrotoluene and its breakdown products (Army 1985c, 1987c; Bauer et al. 1990; Bongiovanni et al. 1984; Jenkins and Grant 1987; Jenkins et al. 1989). Sample extraction consists of homogenizing the soil or sediment, extraction with an organic solvent, and separation on a reverse-phase HPLC column. The method is sensitive and reliable and has been used to determine 2,4,6-trinitrotoluene and some of its metabolites at levels in the low ppb range. GC/ECD has also been used to analyze soil samples for 2,4,6-trinitrotoluene (Army 1985c). Sample preparation is similar to that used for HPLC. Results from GC analysis were mixed, with some samples producing results comparable to HPLC, but others (those samples high in organic matter) subject to substantial interference. More recent efforts by the Army have focused on the development of simple, rapid methods that can be used to screen samples in the field. Two semiquantitative methods have been tested, one based on an indicator tube originally designed for testing water samples (Army 1990b; Heller 1982) and the other based on spectrophotometry (Army 1990a). Both methods could detect 2,4,6-trinitrotoluene in the sub- to low-ppm range but were not specific for 2,4,6-trinitrotoluene. The indicator tube was inexpensive, simple, rapid, and easy to use. However, it was found to have poor accuracy and precision. The spectrophotometric method had better accuracy and precision and estimated concentrations correlated well with laboratory analyses of the same samples. It is not as convenient as the indicator tube, however, because a battery-operated spectrophotometer must also be carried into the field.

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A method for determining 2,4,6-trinitrotoluene in plant stems using HPLC/UV was located (Army 1981b). The limit of detection for the method was in the ppb, but both recovery (52%) and precision (33% RDS) were poor. No other methods in similar matrices were available for comparison,

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 2,4,6-trinitrotoluene 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 2,4,6-trinitrotoluene.

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 or eliminate 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 may be proposed.

6.3.1   Identification of Data Needs

Methods for Determining Biomarkers of Exposure and Effect. Only a few methods for monitoring exposure to 2,4,6-trinitrotoluene were located. These included HPLC/MS, GC/ECD, and TLC/densitometry for determining 2,4,6-trinitrotoluene or its metabolites in blood and/or urine (Almog et al. 1983; Liu et al. 1991; Yinon and Hwang 1985b, 1986c) and HPLC/UV for determining 2,4,6-trinitrotoluene in tissues (Army 1981). In addition, there are methods for analyzing for the chemical and its metabolites in handswab samples (Douse 1987; Fetterolf et al. 1991; Fine et al. 1984; Lloyd 1991). While these methods appear to be useful and reliable for measuring 2,4,6-trinitrotoluene in biological samples, the limited information on sensitivity, selectivity, accuracy, and precision make

it difficult to compare and fully evaluate the reliability of the methods. More information is needed on these parameters for the different methods in order to assess their usefulness for monitoring exposure to 2,4,6-trinitrotoluene.

Possible biomarkers of effect for 2,4,6-trinitrotoluene exposure include changes in hematological and blood chemistry parameters, such as decreased hemoglobin and hematocrit levels, increased mononuclear leukocyte and lymphocyte counts, and changes in levels of SGOT and LDH (Army 1976, 1978a; Morton et al. 1976). Reliable standard clinical laboratory methods exist to measure these parameters; however, they are not specific to 2,4,6-trinitrotoluene exposure and have only limited use as biomarkers of effect for this chemical. Urine discoloration and cataracts are also observed in workers exposed to 2,4,6-trinitrotoluene, but no methods are available to quantitate these nonspecific biomarkers.

Methods for Determining Parent Compounds and Degradation Products in Environmental Media. Methods exist to detect and quantify 2,4,6-trinitrotoluene in air, fresh water, sea water, waste-water effluents, soil, and plant material (Army 1981, 1985b; 1986c, 1988, 1990a, 1990b; Bauer et al. 1990; Feltes and Levsen 1989; Feltes et al. 1990; Hable et al. 1991; Hoffsommer and Rosen 1972; Jenkins et al. 1989; McLuckey et al. 1988; Spangler et al. 1983; Van Slyke et al. 1985; Zhang et al. 1989). The HPLC- and GC-based methods are generally considered to be sensitive and reliable, but in some cases (e.g., air samples) better characterization is needed. Some of the newer methods (e.g., IMS, GDMS) and those proposed for field use (e.g., IMS, membrane-based spectrophotometry) need continued research and characterization in order to be useful quantitative methods for analysis of environmental samples (Army 1990b; McLuckey et al. 1988; Spangler et al. 1983; Zhang et al. 1989).

6.3.2 On-going Studies

No on-going analytical methods studies were located. However, it is likely that research is continuing on some of the more recently introduced methods for analyzing biological and environmental samples,

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such as the use of ELISA (Fetterolf et al. 1991). GDMS (McLuckey et al. 1988), IMS (Spangler et al. 1983), and spectrophotometric field methods (Army 1990b; Zhang et al. 1989).