

## 7. ANALYTICAL METHODS

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

Analytical methods that determine nickel in biological materials are the same as those used for environmental samples. The most common methods determine the total nickel content of the sample instead of the particular nickel compound that may be present. Methodological differences are a function of the nickel level in the sample, digestion procedure required to solubilize the sample, and the level of potentially interfering substances that may be present. Either wet ashing with sulfuric acid or dry ashing through dissolution of the ash with dilute sulfuric or hydrochloric acid is generally a satisfactory method to detect nickel in tissue or food (Boyer and Horowitz 1986; Coleman et al. 1992). Another methodological approach utilizes digestion of biological samples with nitric acid (Custer et al. 2003; Odland et al. 2003) that can also be followed by treatment with hydrogen peroxide to remove residual biological material (USGS 2000). Digestion procedures for biological and environmental samples with particular reference to nickel determinations have been reviewed (Stoeppler 1980; Sunderman 1993; Versieck 1985). As the digestion procedures require the use of strong acids and substances with explosion hazards (e.g., perchloric acid), all safety procedures should be carefully reviewed before the analyses are completed.

Nickel is normally present at very low levels in biological samples. To determine trace nickel levels in these samples accurately, sensitive and selective methods are required. Atomic absorption spectrometry (AAS) and inductively coupled plasma-atomic emission spectroscopy (ICP-AES), with or without

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preconcentration or separation steps, are the most common methods. These methods have been adopted in standard procedures by EPA, NIOSH, IARC, and the International Union of Pure and Applied Chemistry (Brown et al. 1981). Direct aspiration into a flame and atomization in an electrically heated graphite furnace or carbon rod are the two variants of atomic absorption. The latter is sometimes referred to as electrothermal AAS (ETAAS). Typical detection limits for ETAAS are  $<0.4 \mu\text{g/L}$ , while the limit for flame AAS and ICP-AES is  $3.0 \mu\text{g/L}$  (Stoepler 1984; Sunderman 1993; Todorovska et al. 2002). The precision of analytical techniques for elemental determinations in blood, muscles, and various biological materials has been investigated (Iyengar 1989). Good precision was obtained with flame AAS after preconcentration and separation, electrothermal AAS, and ICP-AES. Inductively coupled plasma-mass spectrometry (ICP-MS) techniques have been used to quantify nickel in urine with detection sensitivities down to approximately  $1 \mu\text{g/L}$  (Sunderman 1993). The quantification of nickel in biological materials is hampered by the presence of calcium, sodium, and potassium and requires the use of isotope dilution techniques to validate the measurements of nickel in samples.

Voltammetric techniques are becoming increasingly important for nickel determinations since such techniques have extraordinary sensitivity as well as good precision and accuracy. Direct measurement of nickel in urine in the presence of other trace metals (e.g., cadmium, cobalt, and lead) was demonstrated using adsorption differential pulse cathodic stripping voltammetry at a detection limit of  $0.027 \mu\text{g/L}$  (Horng et al. 2003). The addition of dimethylglyoxime, a chelating agent, to the electrolyte significantly enhances the method's sensitivity (IARC 1990; Stoepler 1984). Detection limits of  $<0.001 \mu\text{g/L}$  have been achieved with differential pulse anodic stripping voltammetry (DPASV) using dimethylglyoxime chelation (Sunderman 1993).

Analytical methods and detection limits for nickel in biological materials are reported in Table 7-1. The presence of nickel in other biological materials such as hair and nails can be determined by the same analytical techniques used for blood and tissue after suitable procedures for dissolving the sample have been utilized (Stoepler 1980; Takagi et al. 1986, 1988). It should be noted that assays of metals in hair are difficult to interpret because of the likelihood of external contamination on the hair shaft, and due caution is advised.

Detailed reviews regarding the methodology used to determine nickel in environmental and biological samples are available (Stoepler 1980, 1984; Sunderman 1993).

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**Table 7-1. Analytical Methods for Determining Nickel in Biological Materials**

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Blood fluid, tissue, and excreta <sup>a</sup>	Acid digestion in mixture of nitric, sulfuric, and perchloric acid	Electrothermal AAS	0.2 µg Ni/L fluid; 0.49 µg Ni/kg of tissue	98% at 5 µg Ni/L; 97% at 8 µg Ni/L	IARC 1986 (Method 11)
Urine	Polydithiocarbamate resin extraction; ash filter and resins in a low temperature oxygen plasma asher or digest with HNO <sub>3</sub> :HClO <sub>4</sub>	ICP-AES; NIOSH 8310	0.1 µg/sample	80%	NIOSH 1994b
Urine	Diluted 1:1 in water	STPGFAA	0.56 µg/L	100.7%	Oliveira et al. 2000
Blood or tissue	Acid digestion in 3:1:1 (v/v/v) HNO <sub>3</sub> :HClO <sub>4</sub> :H <sub>2</sub> SO <sub>4</sub>	ICP-AES; NIOSH 8005	1 µg/100 g blood; 0.2 µg/g tissue	86% in blood	NIOSH 1994b
Serum or urine	Sample (10 µL) injected into graphite furnace with equal volume of 30% hydrogen peroxide; pyrolyzed at 1,200 °C; atomized at 2,100–2,200 °C	ETAAS	0.2 µg/L	95–97% at 1–20 µg/L	Todorovska et al. 2002
Lung tissue	Acid digestion in 4:2:1 (v/v/v) HNO <sub>3</sub> :HClO <sub>4</sub> :H <sub>2</sub> SO <sub>4</sub>	Electrothermal AAS	5 ng/g	No data	Svenes and Andersen 1998

<sup>a</sup>If substantial quantities of iron are present (e.g., whole blood, tissues), hydrochloric acid is added, and the resulting ferric chloride is extracted with methyl isobutyl ketone.

AAS = atomic absorption spectrometry; ETAAS = electrothermal atomic absorption spectrometry; HClO<sub>4</sub> = perchloric acid; HNO<sub>3</sub> = nitric acid; H<sub>2</sub>SO<sub>4</sub> = sulfuric acid; ICP-AES = inductively coupled plasma-atomic emission spectroscopy; Ni = nickel; NIOSH = National Institute for Occupational Safety and Health; STPGFAA = stabilized temperature graphite furnace atomic absorption; v = volume

## 7.2 ENVIRONMENTAL SAMPLES

Analytical methods that detect nickel in environmental samples generally determine the total nickel content of the sample; determining specific nickel compounds is difficult. Filtering a water sample through a 0.45- $\mu\text{m}$  membrane filter can distinguish between total and dissolved nickel (Martin et al. 1992). The most common methods used to detect nickel in environmental samples are AAS, either flame or graphite furnace, ICP-AES, or ICP-MS. Nickel in water and waste water samples can be analyzed using ASTM Test Methods D1976 (ICP-AES) (ASTM 2000) and D5673 (ICP-MS) (ASTM 2000) or EPA Test Methods 249.1 (atomic absorption, direct aspiration) (EPA 1983), 249.2 (atomic absorption, furnace technique) (EPA 1983), 200.7 (ICP-AES) (EPA 1983), 200.8 (ICP-MS) (EPA 1994), 1638 (ICP-MS) (EPA 1996e), and 200.12 (atomic absorption, graphite furnace technique) (EPA 1997b), or a direct current plasma atomic emission spectrophotometric method (EPA 1990b). Nickel can also be analyzed in ambient and marine water using stabilized temperature graphite furnace atomic absorption (STGFAA) detection techniques as described in EPA methods 1639 (EPA 1996d) and 200.12 (EPA 1997b), respectively, which give limits of detection for nickel concentrations ranging between 0.65 and 1.8  $\mu\text{g/L}$  and recoveries of >92%.

Although these methods are suitable for groundwater and surface water samples and domestic and industrial effluents, the nickel concentration in some groundwater, surface water, marine water, and drinking water is often below the method detection limits. Therefore, the sample must be preconcentrated or other test methods must be used. One EPA standardized test method, 1640, uses a chelation preconcentration step to increase the detection sensitivity of the ICP-MS based assay (EPA 1996c). Two other EPA standard test methods, 200.10 and 200.13, also use preconcentration techniques in conjunction with ICP-MS (EPA 1997c) or graphite furnace AAS (EPA 1997d) detection techniques, respectively, for analysis of nickel in marine water. One method uses activated charcoal to preconcentrate nickel in natural waters, followed by elution with 20% nitric acid and analysis by inductively coupled plasma-optical emission spectrometry (ICP-OES) (Yunes et al. 2003). This method achieved a detection limit of 82 ng/L. Measurement of trace metals, including nickel, in waste water, surface runoff, and seawater can be completed using an in-line system with stripping voltammetry or chronopotentiometry (Sedlak et al. 1997; van den Berg and Achterberg 1994). These methods provide rapid analysis (1–15 minutes) with little sample preparation. The detection limit of these methods for nickel was not stated. Recommended EPA methods for soil sediment, sludge, and solid waste are Methods 7520 (AAS) and 6010B (ICP-AES). Before the widespread use of AAS, colorimetric methods were employed, and a number of colorimetric reagents have been used (Stoeppler 1980).

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With analytical methods such as x-ray fluorescence (XRF), proton-induced x-ray emission (PIXE), and instrumental neutron activation analysis (INAA), many metals can be simultaneously analyzed without destroying the sample matrix. Of these, XRF and PIXE have good sensitivity and are frequently used to analyze nickel in environmental samples containing low levels of nickel such as air, rain, snow, and soil (Adamo et al. 1996; EPA 1999; Hansson et al. 1988; Landsberger et al. 1983; Nygren 2002; Schroeder et al. 1987; Sweet et al. 1993; Wiersema et al. 1984). The Texas Air Control Board, which uses XRF in its network of air monitors, reported a mean minimum detectable value of 6 ng nickel/m<sup>3</sup> (Wiersema et al. 1984). In the EPA method IO-3.3, detection limits of 0.18 and 1.89 ng/m<sup>3</sup> are reported in the analysis of nickel contained in fine (ca. 2.5 µm) and coarse (>10 µm) particulate matter (PM), respectively, collected on Teflon filters (EPA 1999). A detection limit of 30 ng/L was obtained using PIXE with a nonselective preconcentration step (Hansson et al. 1988). Lower detection limits of 2.37 ng/m<sup>3</sup> are reported for the EPA method IO-3.6 based on dichotomous sampling for 24 hours using a Teflon filter at a sampling rate of 0.9 m<sup>3</sup>/hour (EPA 1999). Energy dispersive x-ray analysis, in conjunction with a four-step metal extraction technique, has been used to measure the speciation of nickel in soils (Adamo et al. 1996). In these techniques, the sample (e.g., air particulates collected on a filter) is irradiated with a source of x-ray photons or protons. The excited atoms emit their own characteristic energy spectrum, which is detected with an x-ray detector and multichannel analyzer. INAA and neutron activation analysis (NAA) with prior nickel separation and concentration have poor sensitivity and are rarely used (Schroeder et al. 1987; Stoepler 1984).

There are other standardized analytical methods for quantifying airborne nickel. These techniques utilize an extraction procedure to isolate nickel and other trace metals from PMs collected on air sampler filters. The extraction methods typically involve the use of hot nitric acid or microwave digestion techniques, for example as described in EPA Method IO-3.1 (EPA 1999). The extracted metals are commonly analyzed using instrumental techniques as described in EPA test methods IO-3.2 (atomic absorption, furnace technique), IO-3.4 (ICP-AES), and IO-3.5 (ICP-MS) (EPA 1999), providing limits of detection for concentrations of nickel in air ranging between 0.02 and 0.10 ng/m<sup>3</sup> (Table 7-2; Vousta and Samara 2002). Use of trace-metal-free acids and sample extraction methods that are designed to exclude contamination of samples from adventitious metals can yield detection limits for determining airborne nickel concentrations down to 0.013–0.02 ng/m<sup>3</sup> when using ICP-MS techniques (EPA 1999; Magari et al. 2002).

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**Table 7-2. Analytical Methods for Determining Nickel in Environmental Samples**

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air, airborne particulates	Collection on cellulose acetate filter; digestion with concentrated nitrated and perchloric acids	ICP-AES; NIOSH 7300	1 µg/sample	105% at 2.5 µg; 97% at 1 mg	NIOSH 1994b
Air, airborne particulates	Collection on glass or quartz fiber filter; microwave or hot acid digestion Method IO-3.1	AAS, graphite furnace; Method IO-3.2	0.10 ng/m <sup>3</sup>	No data	EPA 1999
Air, airborne particulates	Collection on Teflon (fine PM) and Nucleopore (coarse PM) membrane filter	XRF; Method IO-3.3	0.18 ng/m <sup>3</sup> (fine PM); 1.89 ng/m <sup>3</sup> (coarse PM)	No data	EPA 1999
Air, airborne particulates	Collection on glass or quartz fiber filter; microwave or hot acid digestion Method IO-3.1	ICP-AES; Method IO-3.4	3.1 ng/m <sup>3</sup>	96.4%	EPA 1999
Air, airborne particulates	Collection on glass or quartz fiber filter; microwave or hot acid digestion Method IO-3.1	ICP-MS; Method IO-3.5	0.02 ng/m <sup>3</sup>	101.7% at 20 µg/L; 102.3% at 100 µg/L	EPA 1999
Air, airborne particulates	Collection on PCTE or Teflon filters, or Kapton impaction surface	PIXE; Method IO-3.6	2.37 ng/m <sup>3</sup>	No data	EPA 1999
Air, airborne Ni(CO) <sub>4</sub>	Collection on low-Ni charcoal sorbent tube; ultrasonic digestion with nitric acid	Graphite furnace AAS; NIOSH 6007	0.01 µg/sample	93% at 5 to 121 µg/m <sup>3</sup>	NIOSH 1994b
Water	Acid digestion in mixture of nitric, sulfuric, and perchloric acids	Electro-thermal AAS; Method 11	0.2 µg Ni/L fluids	98% at 5 µg Ni/L; 97% at 8 µg Ni/L	IARC 1986
Water	Preconcentrated on activated charcoal; eluted with 20% nitric acid	ICP-OES	82 ng/L	96.0% at 2.0 µg/L	Yunes et al. 2003
Drinking, domestic, surface water; industrial waste water	Filter and acidify sample	ICP-AES; Method D1976	15 µg/L	92%	ASTM 2000
Drinking water, surface water, groundwater	Filter and acidify sample	ICP-MS; Method D5673	4 µg/L	104%	ASTM 2000

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**Table 7-2. Analytical Methods for Determining Nickel in Environmental Samples**

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Water, waste water	Acid digestion	AAS, direct aspiration; Method 249.1	0.04 mg/L	100% at 0.20 mg Ni/L; 97% at 1.0 mg Ni/L; 93% at 5.0 mg Ni/L	EPA 1983
	Acid digestion; sample solutions should contain 0.5% HNO <sub>3</sub>	AAS, direct aspiration; Method 249.2	1 µg/L	100%	EPA 1983
	Filter and acidify sample (dissolved Ni); digest in nitric acid (total recoverable Ni)	ICP-AES; Method 200.7	5 µg/L	Accuracy: 6.7% at 30 µg/L; 8.3% at 60 µg/L; 2.0% at 120 µg/L	EPA 1983, 1994; Martin et al. 1992
Water, waste water	Filter and acidify sample (dissolved Ni); digest in nitric acid (total recoverable Ni)	ICP-MS; Method 200.8	0.5 µg/L	100.1% at 100 µg/L	EPA 1994
Water, waste water	Acid digestion	AAS, graphite furnace; Method 7521	1 µg/L	No data	EPA 2002
Water, waste water	Digestion with nitric and hydrochloric acids	ICP-AES; Method 6010C	10 µg/L	98% at 250 µg/L; 92% at 60 µg/L; 93% at 30 µg/L	EPA 2002
Marine water	Acidified with nitric acid, undissolved material removed	STPGFAA; Method 200.12	1.8 µg/L	92% at 15 µg/L; 93% at 37.5 µg/L	EPA 1997b
Snow	Samples acidified with nitric acid	ICP-MS	0.7 pg/L	95%	Barbante et al. 2002
Soil, sediment, sludge, solid waste	Digestion with nitric and hydrochloric acids; Method 3050	ICP-AES; Method 6010B	10 µg/L	98% at 250 µg/L; 93% at 50 µg/L	EPA 1986b; EPA 2002
Soil, sediment, sludge, solid waste	Digestion with nitric and hydrochloric acids; Method 3050	AAS, direct aspiration; Method 7520	0.04 mg/L	100% at 0.2 mg/L; 97% at 1.0 mg/L; 93% at 5.0 mg/L	EPA 1986b

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**Table 7-2. Analytical Methods for Determining Nickel in Environmental Samples**

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Soil (total nickel)	Digest with nitric acid; oxidize with hydrogen peroxide at 450 °C to destroy organic matter; digest with sulfuric and hydrofluoric acids, followed by digestion with nitric, sulfuric, and perchloric acids	AAS	0.1 µg Ni/g soil	No data	Baker and Amacher 1982
Soil (DPTA extractable)	Shake soil with 0.005 M DPTA extraction solution for 2 hours	AAS	No data	No data	Baker and Amacher 1982
Soil (acid extractable)	Shake soil with 0.1 N hydrochloric acid for 5 minutes; complete 3 times	AAS	No data	No data	Baker and Amacher 1982
Soil and sediment	Sample is heated to 110 °C in a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids and evaporated to dryness, and then treated with aqua regia	ICP-AES	3 ppm	92–114%	USGS 2002
	Sample is heated to 110 °C in a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids and evaporated to dryness, and then treated with aqua regia	ICP-MS	0.16 ppm	91–104%	USGS 2002
Food	Wet oxidation with sulfuric acid, complexation with ammonium tetramethylenedithiocarbamate followed by extraction with methyl butyl ketone <sup>a</sup>	AAS; Method 17	20 µg/kg	No data	IARC 1986
Edible tissues	Samples were homogenized, mixed with magnesium nitrate solution (6.67%), lyophilized, dry ashed twice, and dissolved in hydrochloric acid	AAS	0.15 ppm	101%	Coleman et al. 1992

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**Table 7-2. Analytical Methods for Determining Nickel in Environmental Samples**

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Food	Samples were homogenized then solubilized using atmospheric pressure microwave digestion in nitric acid	ICP-MS	7.0 ng/g	52–96% <sup>b</sup>	Melnyk et al. 2003

<sup>a</sup>The digestion procedure is not satisfactory for fats and oils. For these substances, sulfuric acid and 50% hydrogen peroxide should be used.

<sup>b</sup>Percent recoveries of nickel in food samples spiked at 2 times the limit of detection (LOD) of nickel were given as: rice cereal, 94%; fatty food, 95%; beverage, 93%; duplicate diet 1, 52%; and duplicate diet 2, 90%. In food samples spiked with nickel at 5 times the LOD, the percent recoveries were given as: fatty food, 96%; beverage, 94%; duplicate diet 1, 81%; and duplicate diet 2, 81%.

AAS = atomic absorption spectrometry; DPTA = diethylenetriamine pentaacetic acid; HNO<sub>3</sub> = nitric acid; ICP-AES = inductively coupled plasma-atomic emission spectroscopy; ICP-OES = inductively coupled plasma-optical emission spectrometry; Ni = nickel; Ni(CO)<sub>4</sub> = nickel carbonyl; NIOSH = National Institute for Occupational Safety and Health; PCTE = polycarbonate track etched; PIXE = proton induced x-ray emission spectroscopy; PM = particulate matter; STPGFAA = stabilized temperature graphite furnace atomic absorption; XRF = x-ray fluorescence

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Contamination and loss are the main concerns when determining trace metals (Christensen 1995). Nickel-containing knives and needles should be avoided when collecting specimens. A study that compared the effects of using different dissecting tools on trace metal analysis did not report significant differences in the nickel content of fish or mussel samples dissected with stainless steel, lexan, titanium, or Teflon-coated instruments (Iyengar 1986). Contamination can result from impurities in reagents or laboratory apparatus and laboratory dust. Losses may also occur when the analyte adsorbs onto container walls. When collecting air samples on filters, one should be aware that filter material can contain high and variable trace metal concentrations. Glass fiber filters may contain  $<80$  ng/cm<sup>2</sup> of nickel. Silver membrane, cellulose, and polystyrene filters may contain  $\approx 100$  ng/cm<sup>2</sup> of nickel (Schroeder et al. 1987). Trace metals in blanks of different filter types and in different filters of the same type may vary from 5 to 20% (Brzezinska-Paudyn et al. 1986).

Some investigators have characterized the forms of nickel in an environmental sample by using successively stronger solvents. Each fraction solubilized is subsequently analyzed for nickel by atomic absorption or other procedures. In air, where the speciation of nickel is less complex, a method of sequential selective leaching has been developed to determine the amount of nickel in four phase categories of a dust sample, namely, soluble nickel, sulfidic nickel, metallic nickel, and refractory nickel oxides (Zatka et al. 1992). Soluble nickel salts, mostly nickel sulfates, are leached at pH 4; sulfidic nickel is next solubilized with a peroxide-citrate solution; and metallic nickel is oxidized with bromine. The residue consists of refractory nickel oxides. Wong and Wu (1991) used an adsorptive stripping voltammetry method to determine different forms of nickel in air at a nickel manufacturing facility. The method distinguished between metallic nickel ions and nickel oxides. The results showed that speciation of nickel from several samples taken at the same location were highly variable. Although it is important to characterize the nickel contained in an environmental sample, methods that determine nickel speciation are difficult and not in widespread use.

Analytical methods and detection limits for standard methods of determining nickel in environmental media are reported in Table 7-2. If the determination of dissolved nickel is required, samples should be filtered with a 0.45- $\mu$ m membrane filter.

### 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

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adequate information on the health effects of nickel is 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 nickel.

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.** Nickel concentrations in hair, nails, blood, or urine are elevated in exposed individuals. A correlation has been established between nickel levels in urine, plasma, and feces in occupationally exposed workers and nickel levels in air (Angerer and Lehnert 1990; Bernacki et al. 1978; Hassler et al. 1983). If the identity of the nickel compounds to which workers are exposed is known, nickel levels in urine and plasma can be used as a biomarker for nickel exposure (Sunderman 1993). Available analytical methods can determine the nickel levels in these media in both unexposed and occupationally exposed persons. Also, reference values for nickel measured in urine and blood in individuals exposed to low levels of nickel are needed to establish norms for the general population (Christensen 1995).

Methods for determining exposure of individuals through the assessment of plasma or urine levels of nickel are adequate, but further method development is needed to determine nickel speciation in biological media. Also, development of assays that make use of biological markers, such as changes in gene expression in blood cells or protein levels in serum, as measured with gene or protein arrays would be useful not only in providing an alternative method for assessing nickel exposure in occupational and public populations, but also in providing information on biological effects to nickel exposures.

**Effect.** There are no unique biomarkers of effect for nickel.

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**Methods for Determining Parent Compounds and Degradation Products in Environmental**

**Media.** Methods for determining total nickel in environmental media are well developed and adequate. Standardized methods are available from several sources including EPA (EPA 1983, 1986b, 1999, 2002). Most analytical methods measure total nickel content. Sequential extraction techniques are sometimes used to determine the nature of nickel in particles, e.g., they are exchangeable, adsorbed, easily reducible, or organically bound (Adamo et al. 1995; Lottermoser 2002; Rudd et al. 1988; Rybicka 1989). There is a need for more development in this area and the adoption of standard methods for determining nickel species or forms of nickel in various media.

**7.3.2 Ongoing Studies**

Information on ongoing research studies involving sample collection and the characterization and quantification of nickel was derived from a search of Federal Research in Progress (FEDRIP 2004) and are summarized in Table 7-3.

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**Table 7-3. Ongoing Studies Involving Sample Collection and the Characterization and Quantification of Nickel<sup>a</sup>**

Investigator	Affiliation	Research description	Sponsor
Barker, A	University of Massachusetts	Validate commonly used analytical methods for measuring trace elements in a variety of soils, water, and plants	Hatch
Odom, JW	Auburn University	Develop analytical techniques for determining total and extractable heavy metals in Alabama soils and plant materials and assess the normal occurrence of metals in select soil profiles	Hatch

<sup>a</sup>FEDRIP 2004