# Analytical Results for Municipal Biosolids Samples from a Monitoring Program near Deer Trail, Colorado (U.S.A.), 2007

By J.G. Crock, D.B. Smith, T.J.B. Yager, C.J. Berry, and M.G. Adams

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## Abstract

Since late 1993, the Metro Wastewater Reclamation District of Denver (Metro District), a large wastewater treatment plant in Denver, Colorado, has applied Grade I, Class B biosolids to about 52,000 acres of nonirrigated farmland and rangeland near Deer Trail, Colorado (U.S.A.). In cooperation with the Metro District in 1993, the U.S. Geological Survey (USGS) began monitoring ground water at part of this site. In 1999, the USGS began a more comprehensive monitoring study of the entire site to address stakeholder concerns about the potential chemical effects of biosolids applications to water, soil, and vegetation. This more comprehensive monitoring program recently has been extended through 2010. Monitoring components of the more comprehensive study include biosolids collected at the wastewater treatment plant, soil, crops, dust, alluvial and bedrock ground water, and streambed sediment. Streams at the site are dry most of the year, so samples of streambed sediment deposited after rain were used to indicate surface-water effects. This report will present only analytical results for the biosolids samples collected at the Metro District wastewater treatment plant in Denver and analyzed during 2007. We have presented earlier a compilation of analytical results for the biosolids samples collected and analyzed for 1999 through 2006. More information about the other monitoring components is presented elsewhere in the literature. Priority parameters for biosolids identified by the stakeholders and also regulated by Colorado when used as an agricultural soil amendment include the total concentrations of nine trace elements (arsenic, cadmium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc), plutonium isotopes, and gross alpha and beta activity. Nitrogen and chromium also were priority parameters for ground water and sediment components.

In general, the objective of each component of the study was to determine whether concentrations of priority parameters (1) were higher than regulatory limits, (2) were increasing with time, or (3) were significantly higher in biosolids-applied areas than in a similar farmed area where biosolids were not applied.

Previous analytical results indicate that the elemental composition of the biosolids from the Denver plant was consistent during 1999–2006 and this consistency continues with the samples for 2007; total concentrations of regulated trace elements remained consistently lower than the regulatory limits for the entire monitoring period.

Our previously reported data (1999–2006) and data presented in this report were used to compile an inorganic-chemical biosolids signature that can be contrasted with the geochemical signature for this site. The biosolids signature and an understanding of the geology and hydrology of the site can be used to separate biosolids effects from natural geochemical effects. Elements of particular interest for a biosolids signature include bismuth, copper, silver, mercury, phosphorus, and silver.

An alternative method of digestion of biosolids was also recently investigated, and the results are presented in this report. A microwave digestion using only nitric acid at controlled elevated temperature and pressure was tested to replace the much more time-consuming and labor-intensive, traditional four-acid, hotplate method for the preparation of solutions to be analyzed by inductively coupled plasma–mass spectrometry (ICP–MS). Elements of concern determined by ICP–MS following digestion include cadmium, copper, lead, molybdenum, nickel, and zinc. The microwave "digestion" proved to be a strong acid leach, and it was less efficient at digesting the biosolids samples with consistently lower recoveries (compared to the four-acid digestion value) for most elements, but especially for the elements of concern—copper, nickel, and zinc. Other elements traditionally associated with the silicate or oxide minerals demonstrated low recoveries, especially titanium (11 percent) and scandium (30 percent). Because of the lower recovery rates for the elements of concern, the microwave digestion will not replace the traditional four-acid, hotplate method.

# Introduction

Since 1993, the Metro Wastewater Reclamation District of Denver (Metro District) has been applying biosolids from the Denver metropolitan area to their property near Deer Trail, Colorado (fig. 1), as an agricultural soil amendment. The biosolids are applied to nonirrigated farmland according to agronomic loading rates. More information about the sewage-treatment process that results in the Metro District biosolids is available at *http://www.metrowastewater.com*. The biosolids-application areas, dates of application, and application rates provided by the Metro District for their properties near Deer Trail for 1999 through 2003 are detailed in Stevens and others (2003) and Yager and others (2004a, 2004b, 2004c). As more information becomes available, it will be posted at the USGS project Web page at *http://co.water.usgs.gov/projects/CO406/CO406.html*.

In 1999, the Metro District property, known as the METROGRO Farm, encompassed about 81 $mi^2$ (52,000 acres) of farmland in Arapahoe and Elbert Counties, Colorado. The Metro District property and surrounding private property are herein referred to as "the study area."

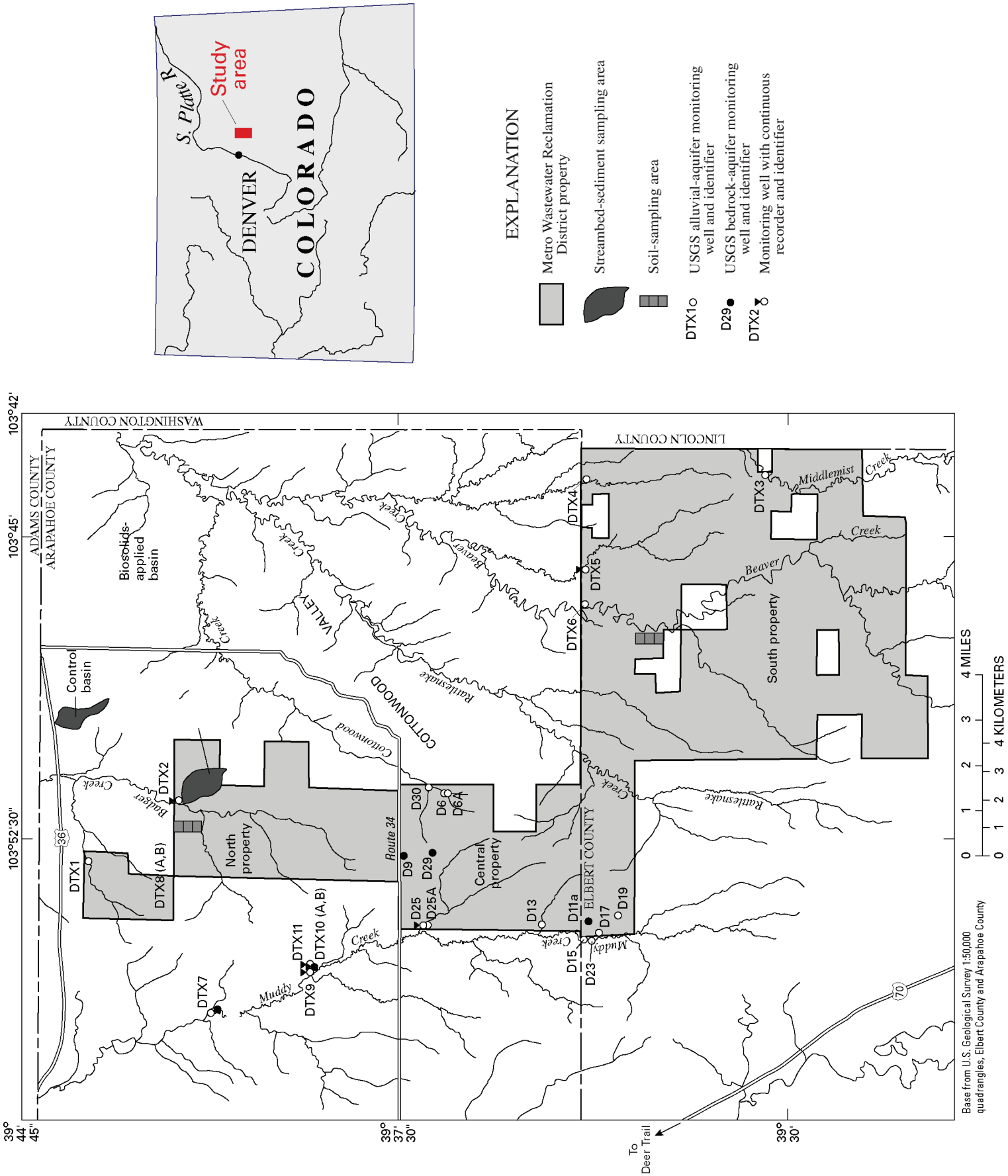


Figure 1. Metro Wastewater Reclamation District of Denver (Metro District) biosolids-application farm and study-area location.

Soils in the study area generally are sandy or loamy on flood plains and stream terraces, clayey to loamy on gently sloping to rolling uplands, and sandy and shaley on steeper uplands. About one-half of the Metro District property is farmed; the remaining is rangeland with some pasture. Land use within the rest of the study area during 1993 through 2007 mostly was rangeland or pasture with some cropland. Farmland in the study area was not irrigated. Biosolids were applied to the land surface of the Metro District property as an agricultural soil amendment, and the primary crop was wheat. Figure 2 shows a typical example of what fresh biosolids (the darker colored patches with the white arrows) look like on the landscape after a single broadcast application.

Figure 2. Biosolids as typically seen after broadcast application to agricultural land.

Public concern about applications of biosolids to farmland increased after the Metro District agreed to accept treated ground water from the Lowry Landfill Superfund site in Denver. The U.S. Geological Survey (USGS), in cooperation with the Metro District and (in 1999) the North Kiowa Bijou Groundwater Management District, studied natural geochemical conditions and the effects of biosolids applications to the Metro District properties near Deer Trail, Colorado, during 1999 through 2007. The study addressed the concerns about biosolids applications and other farming-related effects on the environment. The objectives of this USGS study were to (1) evaluate the combined effects of biosolids applications, land use, and natural processes on soil, crops, the bedrock aquifer, alluvial aquifers, and streambed sediments by comparing chemical data to regulatory standards, data from a site where biosolids are not applied (a control site), or earlier data from the same site (trends); (2) monitor biosolids for trace elements and radioactivity and compare trace-element concentrations and radioactivity with regulatory standards; and (3) characterize the hydrology of the study area. This report provides the 2007 analytical data for biosolids only. Analytical results for biosolids collected between 1999 and 2006 are in Crock and others (2008). A

complete discussion of findings (up to the end of 2003) for all matrices and the other study-area objectives is detailed in Yager and others (2004d).

## Methodology

Biosolids are solid organic matter recovered from a sewage-treatment process that meets State and Federal regulatory criteria for beneficial use, such as for a soil amendment. Figure 3 shows freshly collected biosolids from the Metro plant spread out in a plastic-lined box to dry. Biosolids are moist (usually ranging 75 to 85 percent moisture) and have a firm, puddinglike texture. The regulations state that land-applied biosolids must meet or exceed Table 1 Ceiling Concentration Limits and Class B pathogen criteria (Grade II, Class B criteria in the Colorado regulations until 2003) (Colorado Department of Public Health and Environment, 1998; U.S. Environmental Protection Agency, 1993). Table 3 and Grade I requirements are stricter than Table 1 and Grade II requirements. The Metro District applies Table 3 (Grade I) Class B biosolids to their properties near Deer Trail. The regulatory references for biosolids are available at the following Web sites.

*http://www.cdphe.state.co.us/wq/PermitsUnit/biosolids/index.html*
*http://www.epa.gov/owm/mtb/biosolids/503pe/index.htm*
*http://www.epa.gov/owm/mtb/biosolids/index.htm#awards*

The biosolids-application areas, dates of application, and application rates provided by the Metro District for their properties near Deer Trail are detailed in Stevens and others (2003) and Yager and others (2004a, 2004b, 2004c).

Priority parameters identified by stakeholders for biosolids (arsenic, cadmium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc; gross alpha and gross beta radioactivity, and plutonium isotopes; and later in the study, total sulfur) included the nine trace elements regulated by the State of Colorado for biosolids. No samples during 2007 were analyzed for gross alpha and gross beta radioactivity and plutonium isotopes. At this time, analyses are pending and therefore no discussion of these analytes is presented in this report. Consult table 1 for a complete list of the priority elements determined by the various analytical methods. Additional elements were determined by the multielement inductively coupled plasma–mass spectrometry (ICP–MS) method (Briggs and Meier, 1999; Taggart, 2002).

Figure 3. Biosolids sample, as received, prior to drying in the laboratory.

For 2007, biosolids samples were collected monthly. Biosolids samples were collected directly from the Metro District facility's processing line in Denver, rather than from individual trucks or fields near Deer Trail, to ensure a more representative sample. Each biosolids sample was a 24-hour composite of 12 subsamples collected about every 2 hours by Metro District personnel at the Metro District facility. The subsamples were collected from the conveyor belt that transfers the biosolids into the transport trucks. The samples were prepared and analyzed at the chemical laboratories of the USGS in Denver, Colorado. The biosolids material was air dried under forced air and an infrared lamp (surface temperature ~40°C) and then ground in an agate-lined shatter box to less than 150 µm prior to chemical analysis. Complete details of the analytical methods and the quality-assurance protocols used are described by Stevens and others (2003), Taggart (2002), and Yager and others (2004a, 2004b, 2004c). For quality control and quality assurance control, the National Institute of Standards and Technology (NIST) standard reference material (SRM) 2781, domestic sludge, was analyzed with the 2007 biosolids samples.

Because of the time-consuming, tedious nature of the four-acid digestion method prior to the elemental determinations by ICPMS (Briggs and Meier, 1999), an alternate digestion method was investigated. The alternate method was a closed-vessel, microwave-assisted nitric acid digestion controlled by both temperature and pressure monitoring. Closed vessels offer increased analytical accuracy due to minimal losses of analytes and reagents. Uniform results are seen also because of the simultaneous homogeneous heating of all 48 vessels in the microwave cavity.

The microwave digestion system used was the Multiwave 3000® microwave sample preparation platform system of Anton Paar®, Graz, Austria.

The following is taken directly from the manufacturer's brochure for this system:

**Multiwave® 3000 Specifications**

| Vessels: | MF50 |
|---|---|
| Liner material: | PFA (Teflon® PFA is a perfluoroalkoxy copolymer resin) |
| Pressure jacket: | PEEK (poly-ether-ether-ketone resin) |
| Volume: | 50 mL |
| Controlled pressure: | 20 bar (290 psi) |
| Maximum pressure | 30 bar (435 psi) |
| Maximum temperature: | 200°C |
| Test pressure | 140 bar |
| Typical applications: | Water, effluents, sewage sludge, plant material, soil, sediment, USEPA procedures (that is, USEPA Method 3051a), biological material |

For the biosolids samples, the procedure followed is presented below.

Weigh out 0.2000 gram of each sample into a PFA liner. Within the job there were three digestion blanks and one SRM NIST 2781 (domestic sewage sludge standard reference material). Four mL concentrated nitric acid ($HNO_3$) was added to each vessel; no water or hydrogen peroxide ($H_2O_2$) was used in this digestion. The liners were capped according to manufacturer's instructions and loaded into the rotor and the preset program run.

This table below gives the microwave settings/parameters for this digestion.

[W, watt; –, not applicable]

| ***Step*** | ***Power*** | ***Ramp time*** | ***Hold time*** | ***Fan speed*** |
|---|---|---|---|---|
| 1 | 900 W | 10 minutes | 5 minutes | 1 |
| 2 | 1,400 W | 5 minutes | 10 minutes | 1 |
| 3 | 900 W | - | 15 minutes | 2 |
| 4 | 0 | - | 30 minutes | 3 |

The pressure rate was set at 0.4 bar/second with the upper pressure limit set at 20 bar. The temperature maximum for the vessel exterior was set at 140˚C. Total digestion time was 45 minutes and 30 minutes cooling time in the Multiwave® 3000.

After the samples had cooled to room temperature, the samples were quantitatively transferred to 50-mL centrifuge tubes and brought up to 20 mL with 18 Ω DI (deionized water) water. The solutions were diluted an additional 1:10 with 18 Ω DI for analysis by ICP–MS.

## Discussion and Results

Biosolids exceeding the standards for trace elements could adversely affect the quality of soil on which the biosolids are applied and could alter Metro District plans for the application of biosolids in Arapahoe and Elbert Counties. The composition of biosolids was monitored to provide an independently determined data set against which the Metro District chemical analyses and the regulatory standards for biosolids can be compared. The 2007 data will also augment the chemical baseline, against which any future change in the concentration of constituents analyzed for in this study may be recognized, measured, and compared, that has been established earlier by Crock and others (2008). This data set will also enhance the "geochemical signature" for biosolids that will potentially enable scientists to recognize when biosolids have affected soils or stream sediments.

All data for the 1999–2006 biosolids samples are presented in Crock and others (2008) and in figures 4–13, supplemented with the 2007 data. The concentration of all nine trace elements remained consistent throughout the study (1999–2007) and below the Grade 1 biosolids requirements. Reference material NIST SRM 2781 results are also presented in tables 2–4. The certificate of analysis for NIST SRM 2781 is available at *https://srmors.nist.gov/certificates/2781.pdf?CFID=14170104&CFTOKEN=d06e643b27500f42-A419A2E6-D5FA-93A1-CD37130CDA7B75FF&jsessionid=b43051d8258c3f293473* or at *https://srmors.nist.gov/certificates/view_cert2gif.cfm?certificate=2781*. Figures 4–13 show the temporal variation of the priority parameters and total sulfur. Arsenic (fig. 4) showed the most variability with its high and low concentration differing by a factor of 6. The other eight elements

varied by a factor of 3 or less. All trace-element concentrations were less than the maximum allowable concentrations established for Table 3 (Grade I) biosolids. (Note that molybdenum does not have a maximum allowable concentration established for Table 3 biosolids. The value used is that for Table 1 biosolids.)

In conclusion, chemical data for biosolids samples collected from the Metro District plant over a 9-year period (1999–2007) show that all nine of the trace elements for which regulatory limits are established maintained relatively uniform concentrations and never exceeded the maximum allowable levels for Table 3 (Grade I) biosolids.

In addition to the nine trace elements that have regulatory standards established, the USGS analyzed the samples for many other elements. Of the regulated elements, mercury and copper had the highest concentrations in biosolids compared to concentrations in soil. Of the nonregulated elements, silver, phosphorus, and bismuth have the highest concentrations in biosolids compared to soils (Yager and others, 2004a, 2004b, 2004c). For this reason, these five elements would be the most likely "geochemical signature" to indicate that soils or stream sediments may have been affected by biosolids.

Table 4 lists the analytical results for the microwave digestion followed by ICP–MS analysis. Table 5 lists the average difference in concentration units between the microwave digestion method and the four-acid digestion for all 27 samples. This difference is the microwave value minus the four-acid value. The next column in table 5 lists this difference as a relative percentage of the average four-acid value for each element. This indicates the ability of the microwave to recover analytes as compared to the four-acid digestion. Negative values indicate incomplete recovery of a given analyte. Averages of all the samples for each of the elements were used as an indication of similarity since the values for a given element ranges so small, as indicated in figures 4–9. A regression analysis would be difficult to interpret. The next two columns of table 5 list the absolute maximum and minimum difference values between the two digestion methods in analyte concentration units.

For the elements reported by the ICP–MS method (cadmium, copper, lead, nickel, molybdenum, and zinc), the microwave digestion does give reasonable and acceptable recovery for cadmium (102.3 percent, relative), lead (98.7 percent, relative), and molybdenum (94.8 percent, relative). However, the microwave method does not give sufficient recovery for copper (91.3 percent, relative), nickel (91.5 percent, relative), and zinc (90.2 percent, relative). These differences are too large to change methods midstudy. There would be a very noticeable shift in the data as presented in figures 4–12. There are also substantial differences (low recovery rates) for elements not regulated, but of interest. These would include iron (89.4 percent, relative), phosphorus (92.6 percent, relative), silver (88.0 percent, relative), sodium (37.2 percent, relative), and titanium (11.3 percent, relative). Many others, including the rare earths and alkali and alkaline earth elements, have low recovery by the microwave digestion method for biosolids. As a result of these lower recovery rates, the four-acid digestion will be continued for this study. However, for different studies, the microwave method may prove to be a feasible alternative to the four-acid method.

## Acknowledgments

This study was done in cooperation with and funding from the Metro Wastewater Reclamation District and the North Kiowa-Bijou Groundwater Management District. The authors

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## References Cited

Briggs, P.H., and Meier, A.L., 1999, The determination of forty-two elements in geological materials by inductively coupled plasma-mass spectrometry: U.S. Geological Survey Open-File Report 99–166, 15 p.

Brown, Z.A., and Curry, K.J., 2002, Total sulfur by combustion, Chapter Q, *in* Taggart, J.E., Jr., ed., 2002, Analytical methods for chemical analysis of geological and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 2002–0223, available at *http://pubs.usgs.gov/of/2002/ofr-02-0223/.*

Colorado Department of Public Health and Environment, 1998, Biosolids regulation: 5CCR 1002–64, January 12, 1998 (and subsequent revisions), 53 p.

Crock, J.G., Smith, D.B., Yager, T.J.B., Brown, Z.A., and Adams, M.G., 2008, Analytical results for municipal biosolids samples from a monitoring program near Deer Trail, Colorado (USA), 1999 through 2006: U.S. Geological Survey Open-File Report 2008–1172, available at *http://pubs.usgs.gov/ofr/2008/1172/.*

Hageman, P.L., 2007, TM 5-D2: Determination of mercury in aqueous and geologic materials by continuous flow–cold vapor–atomic fluorescence spectrometry (CVAFS), available at *http://pubs.usgs.gov/tm/2007/05D02/.*

Hageman, P.L., and Welsch, Eric, 1996, Arsenic, antimony, and selenium by flow injection or continuous flow-hydride generation-atomic absorption spectrometry, *in* Arbogast, B.F., ed., 1996, Analytical methods manual for the Mineral Resource Surveys Program: U.S. Geological Survey Open-File Report 96–525, p. 24–30.

Stevens, M.R., Yager, T.J.B., Smith, D.B., and Crock, J.G., 2003, Biosolids, soils, ground water, and streambed-sediment data for a biosolids-application area near Deer Trail, Colorado: U.S. Geological Survey Open-File Report 2002–51, 118 p.

Taggart, J.E., Jr., ed., 2002, Analytical methods for chemical analysis of geological and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 2002–0223, available at *http://pubs.usgs.gov/of/2002/ofr-02-0223/.*

U.S. Environmental Protection Agency, 1993, (revised July 1, 2003), Part 503—Standards for the use or disposal of sewage sludge: Code of Federal Regulations Title 40, v. 27, 40CFR503, p. 820–852.

Yager, T.J.B., Smith, D.B., Crock, J.G., and Stevens, M.R., 2004a, Biosolids, soil, crop, ground water, and streambed-sediment data for a biosolids-application area near Deer Trail, Colorado, 2000: U.S. Geological Survey Open-File Report 2003–400, 90 p.

Yager, T.J.B., Smith, D.B., and Crock, J.G., 2004b, Biosolids, soil, crop, ground water, and streambed-sediment data for a biosolids-application area near Deer Trail, Colorado, 2001: U.S. Geological Survey Open-File Report 2004–1388, 69 p.

Yager, T.J.B., Smith, D.B., and Crock, J.G., 2004c, Biosolids, soil, crop, ground water, and streambed-sediment data for a biosolids-application area near Deer Trail, Colorado, 2002–2003: U.S. Geological Survey Open-File Report 2004–1404, 90 p.

Yager, T.J.B., Smith, D.B., and Crock, J.G., 2004d, Effects of surface applications of biosolids on soil, crops, ground water, and streambed sediment near Deer Trail, Colorado, 1999–2003: U.S. Geological Survey Scientific Investigations Report 2004–5289, 93 p.

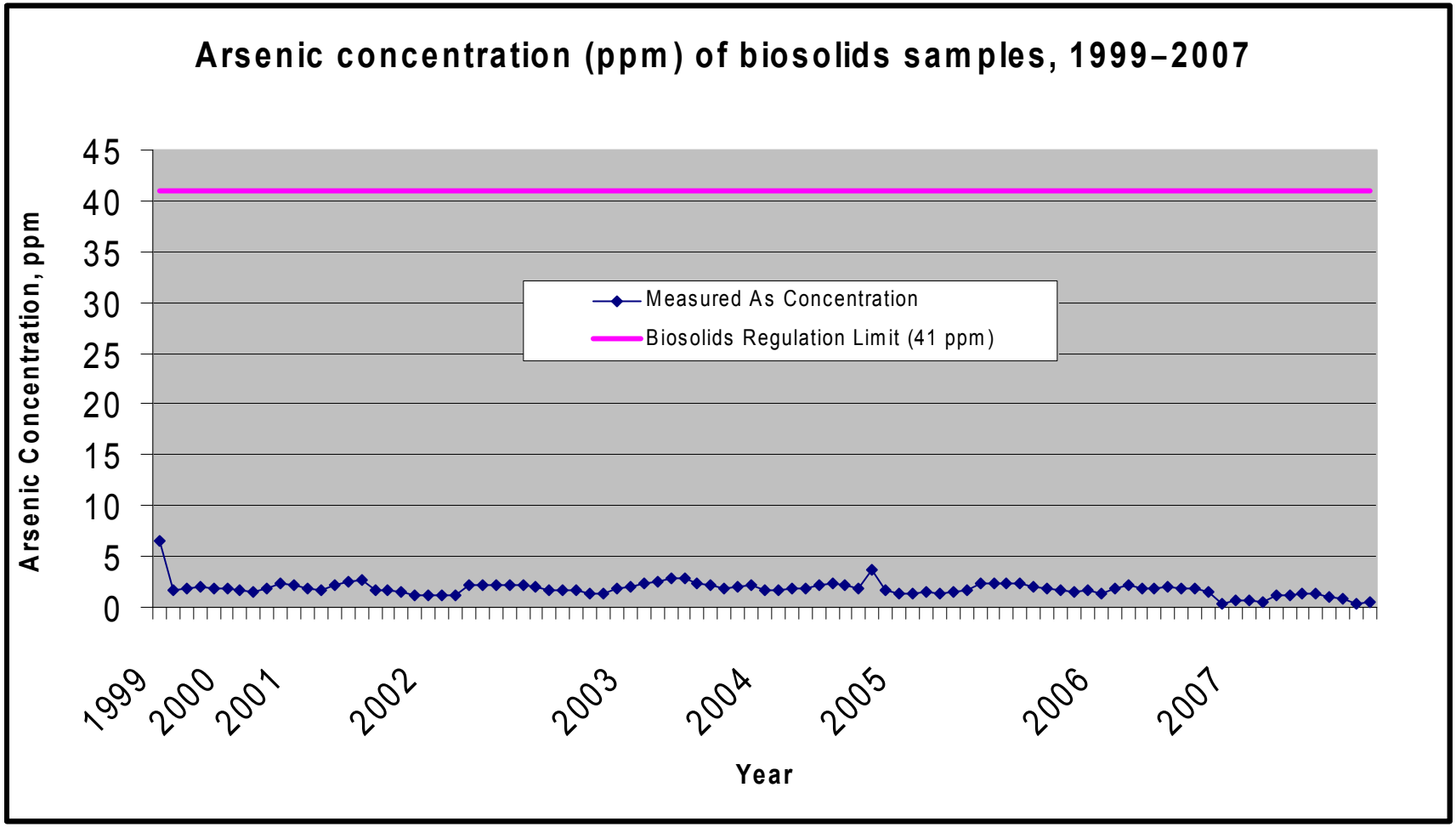


Figure 4. Arsenic concentrations of biosolids samples, 1999–2007.

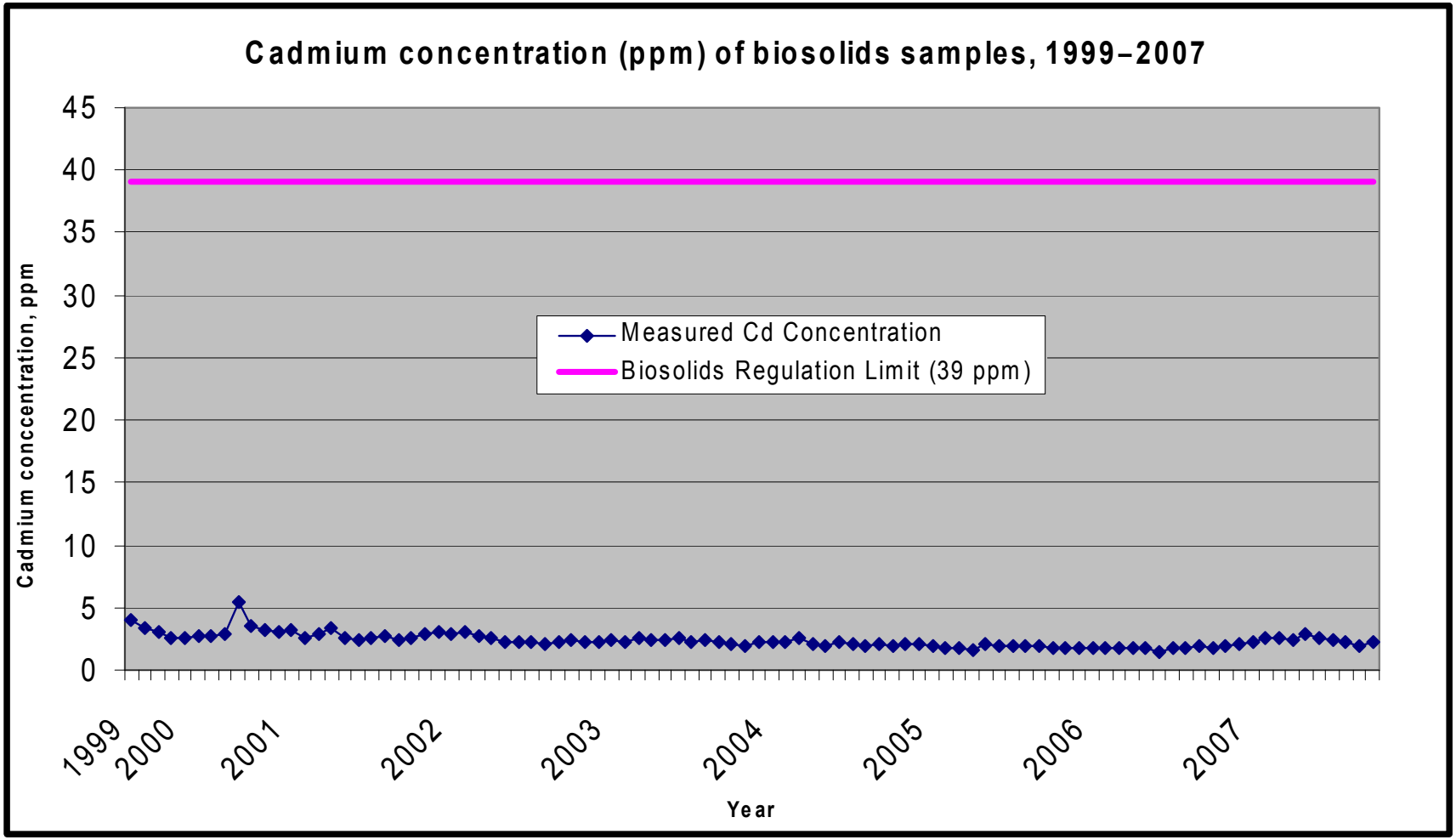


Figure 5. Cadmium concentrations of biosolids samples, 1999–2007.

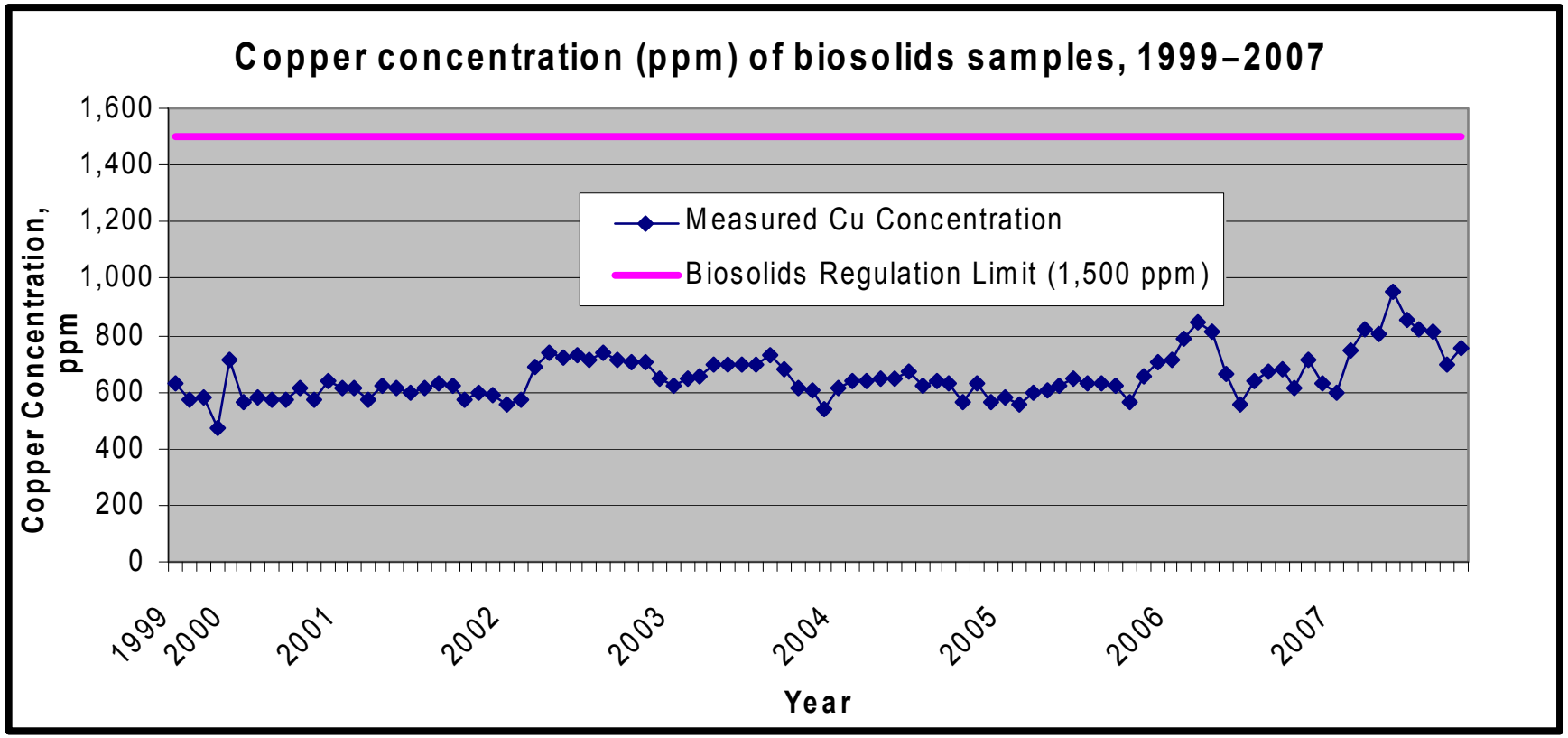


Figure 6. Copper concentrations of biosolids samples, 1999–2007.

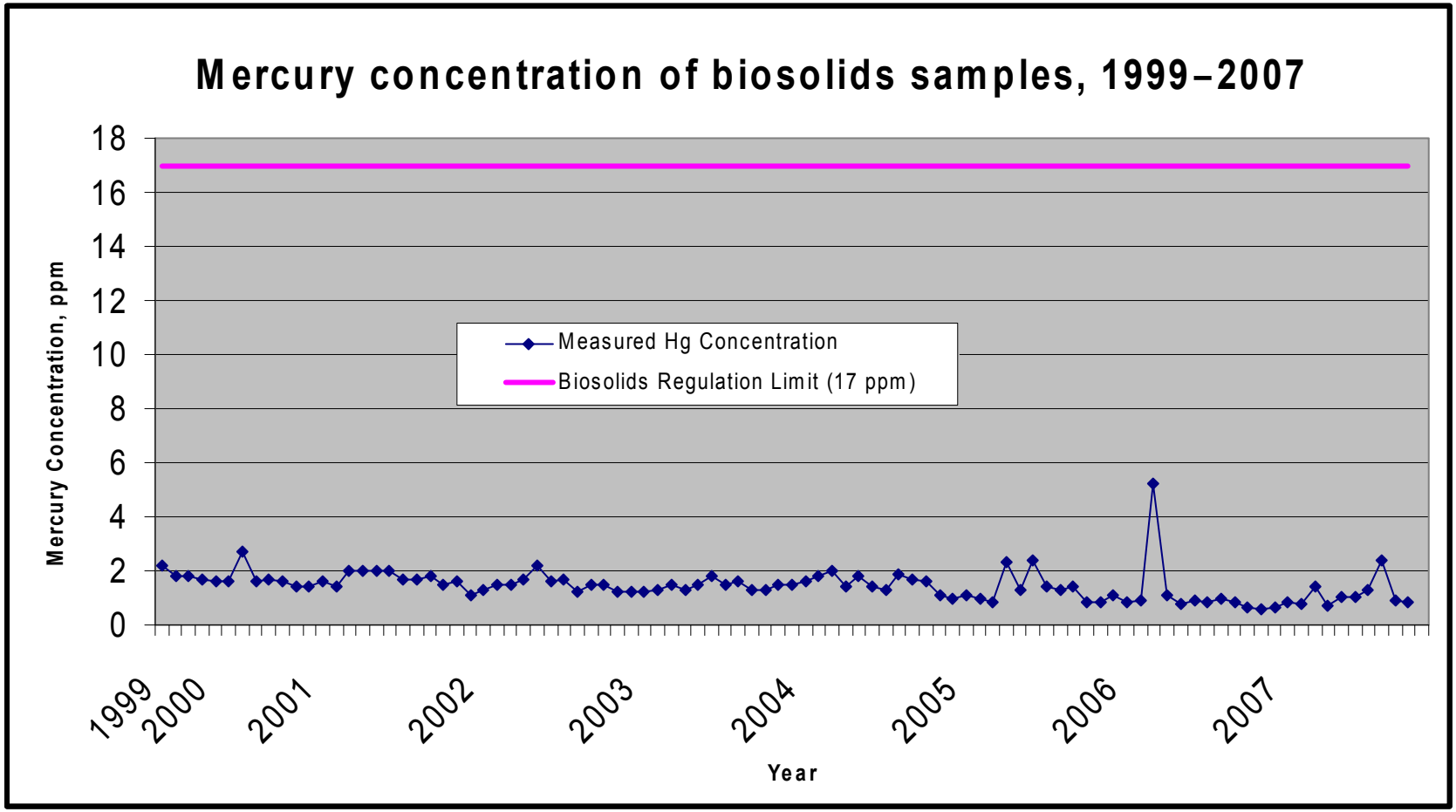


Figure 7. Mercury concentrations of biosolids samples, 1999–2007.

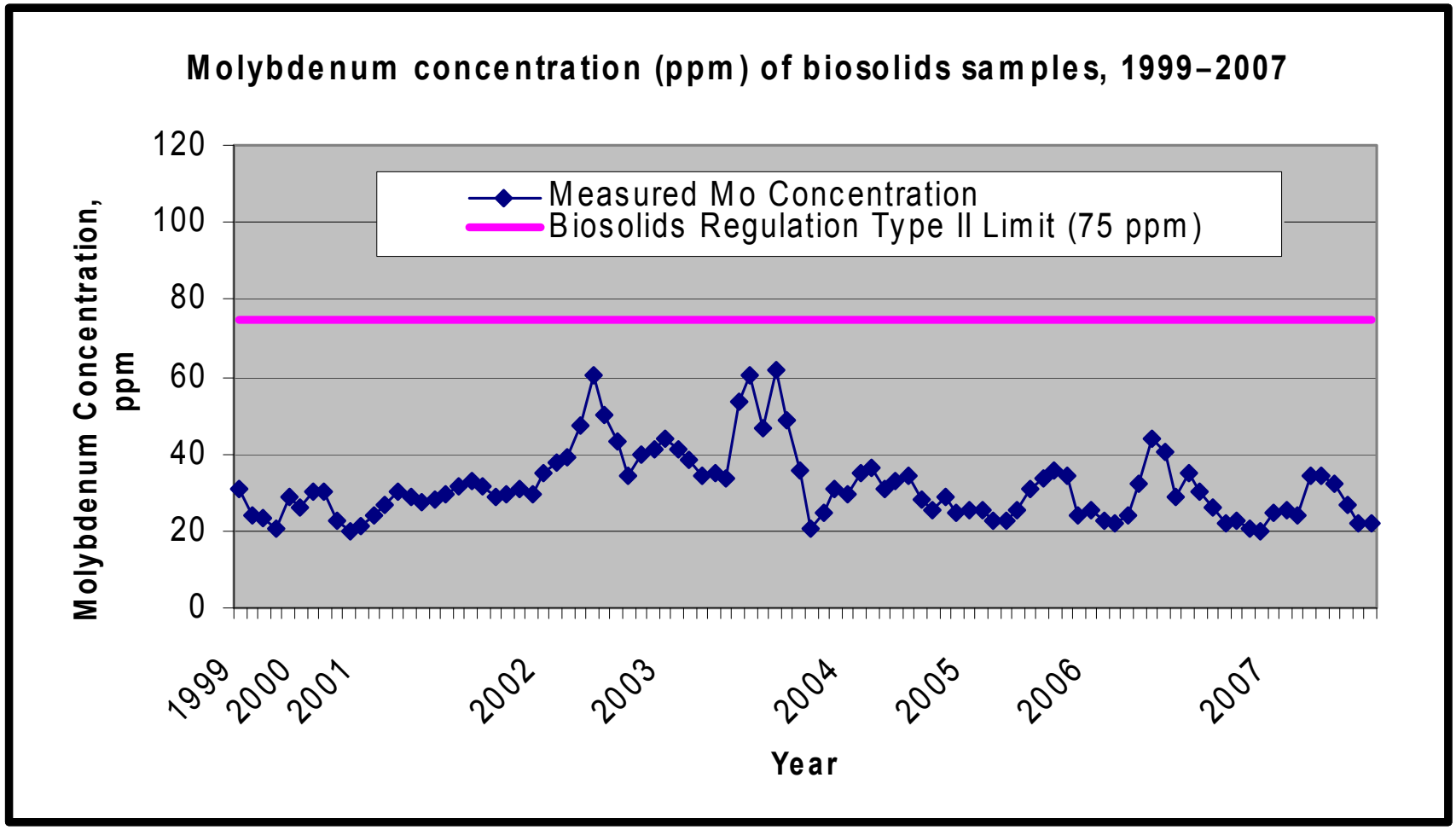


Figure 8. Molybdenum concentrations of biosolids samples, 1999–2007.

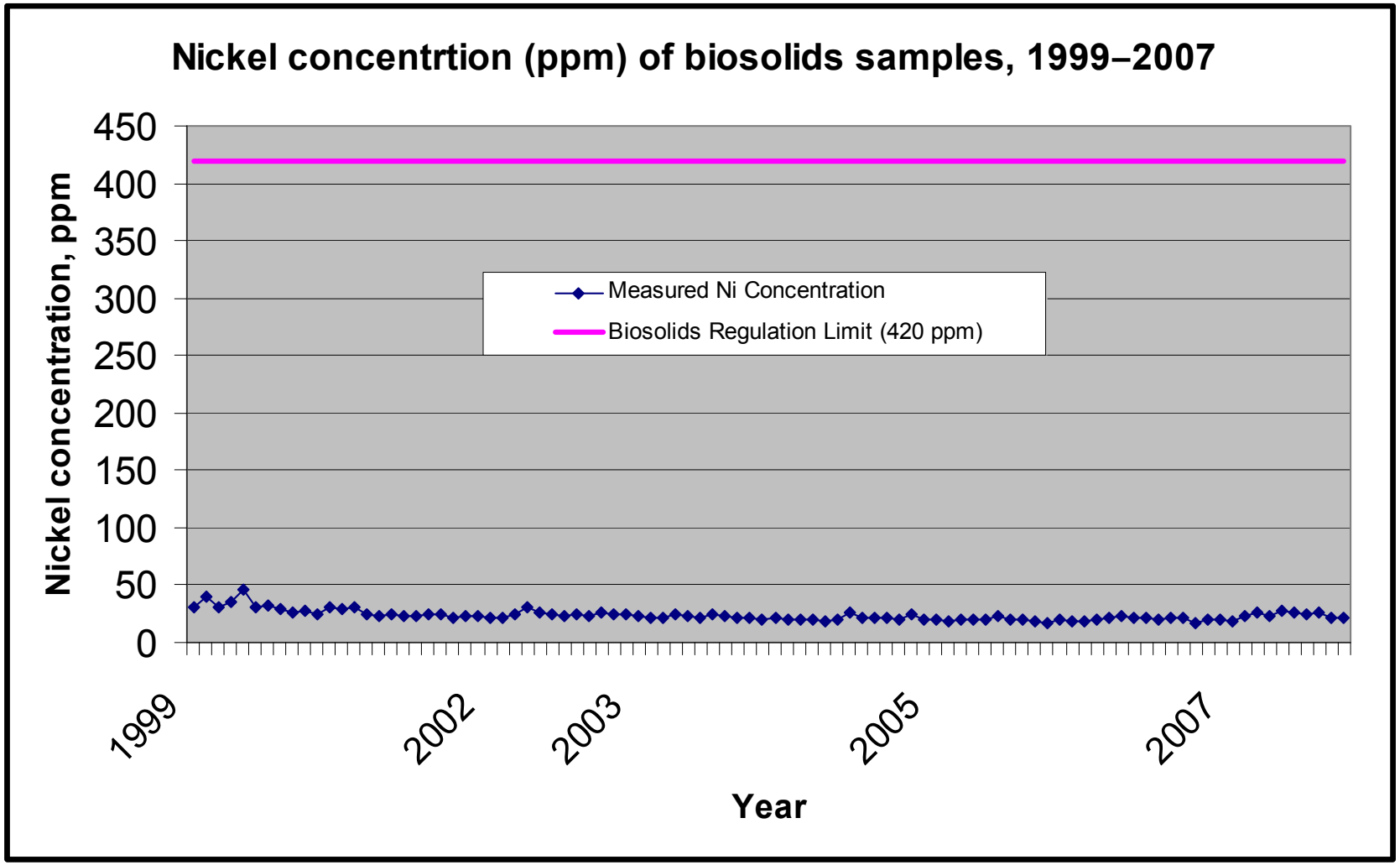


Figure 9. Nickel concentrations of biosolids samples, 1999–2007.

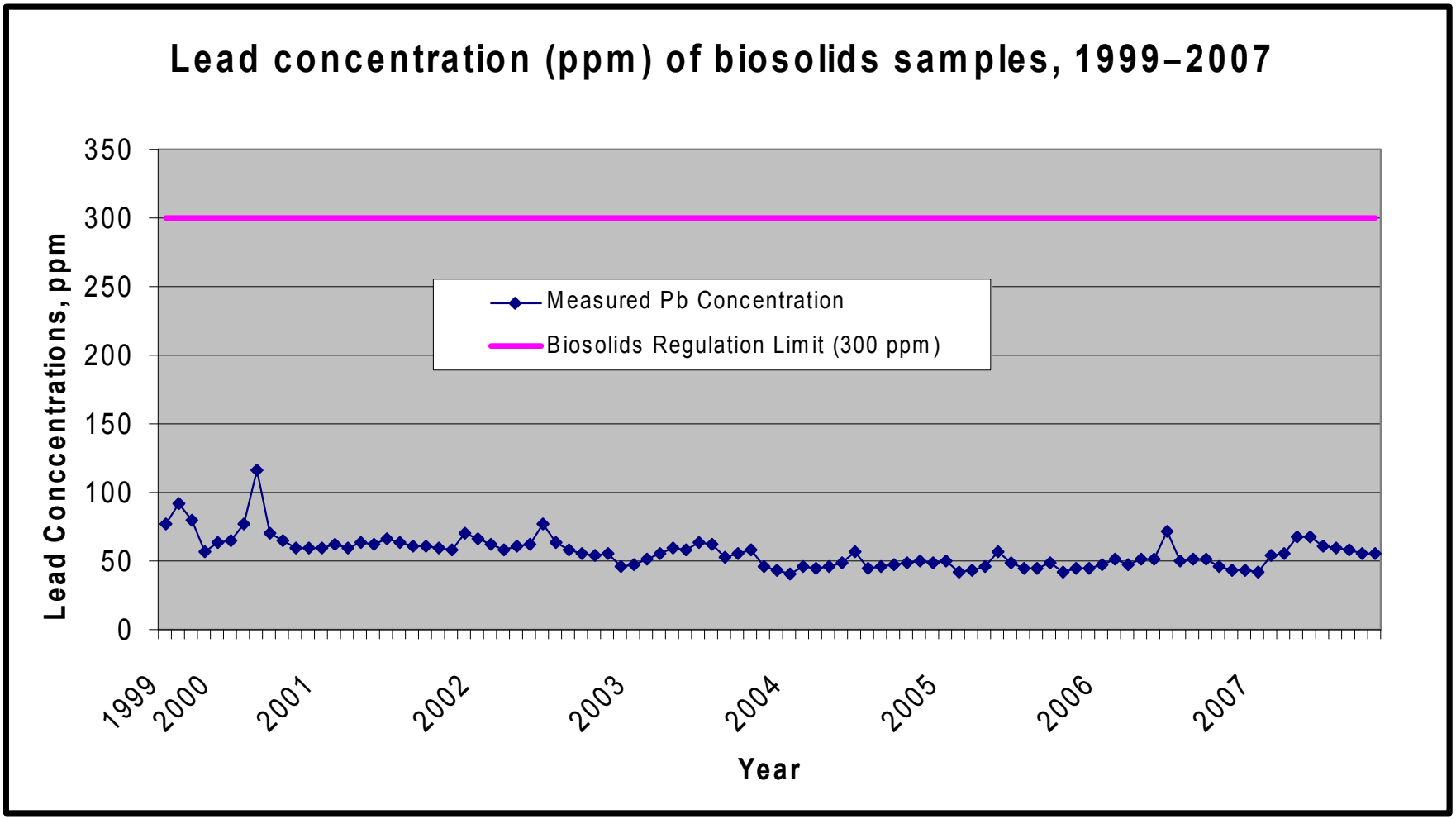


Figure 10. Lead concentrations of biosolids samples, 1999–2007.

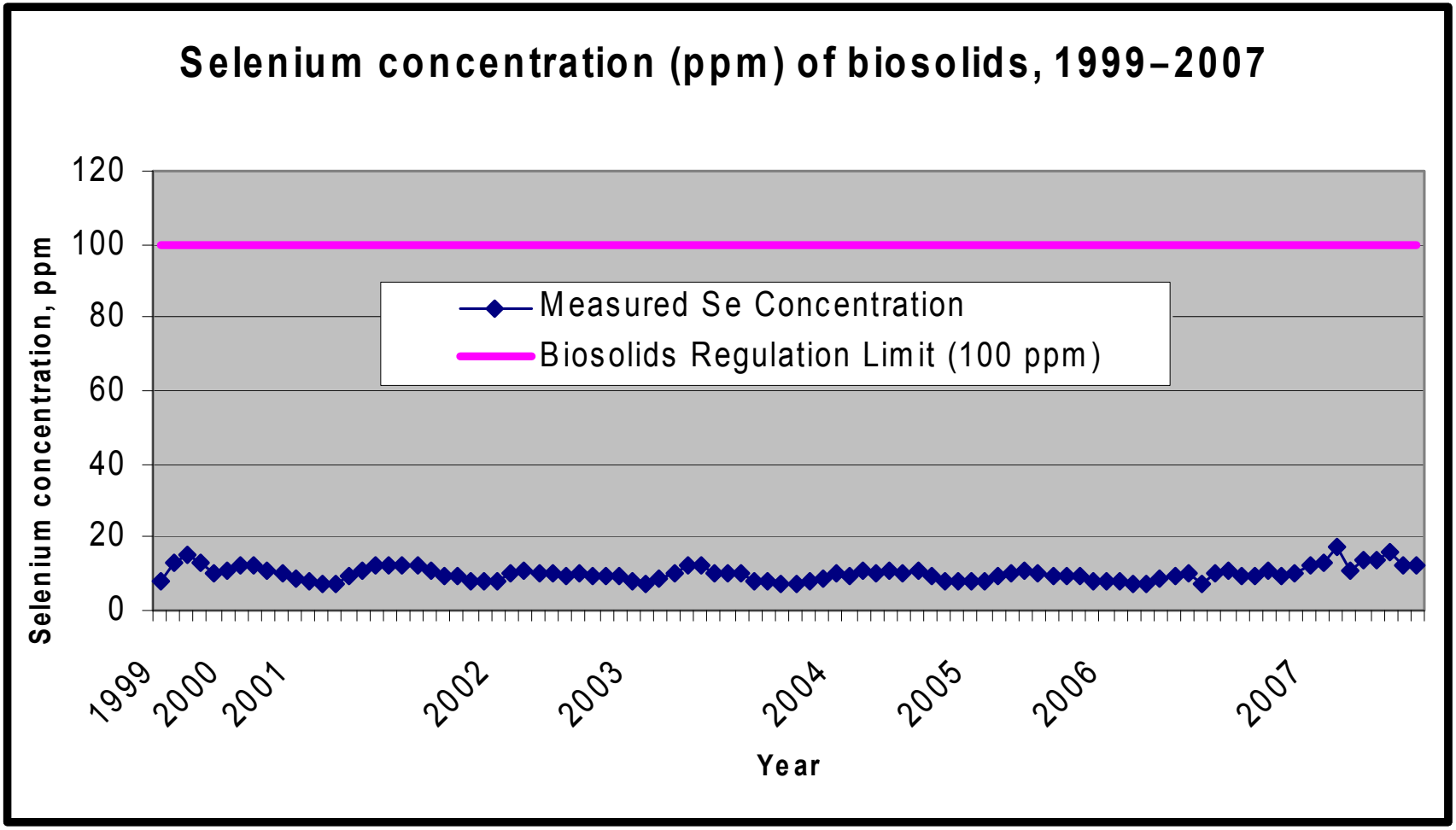


Figure 11. Selenium concentrations of biosolids samples, 1999–2007.

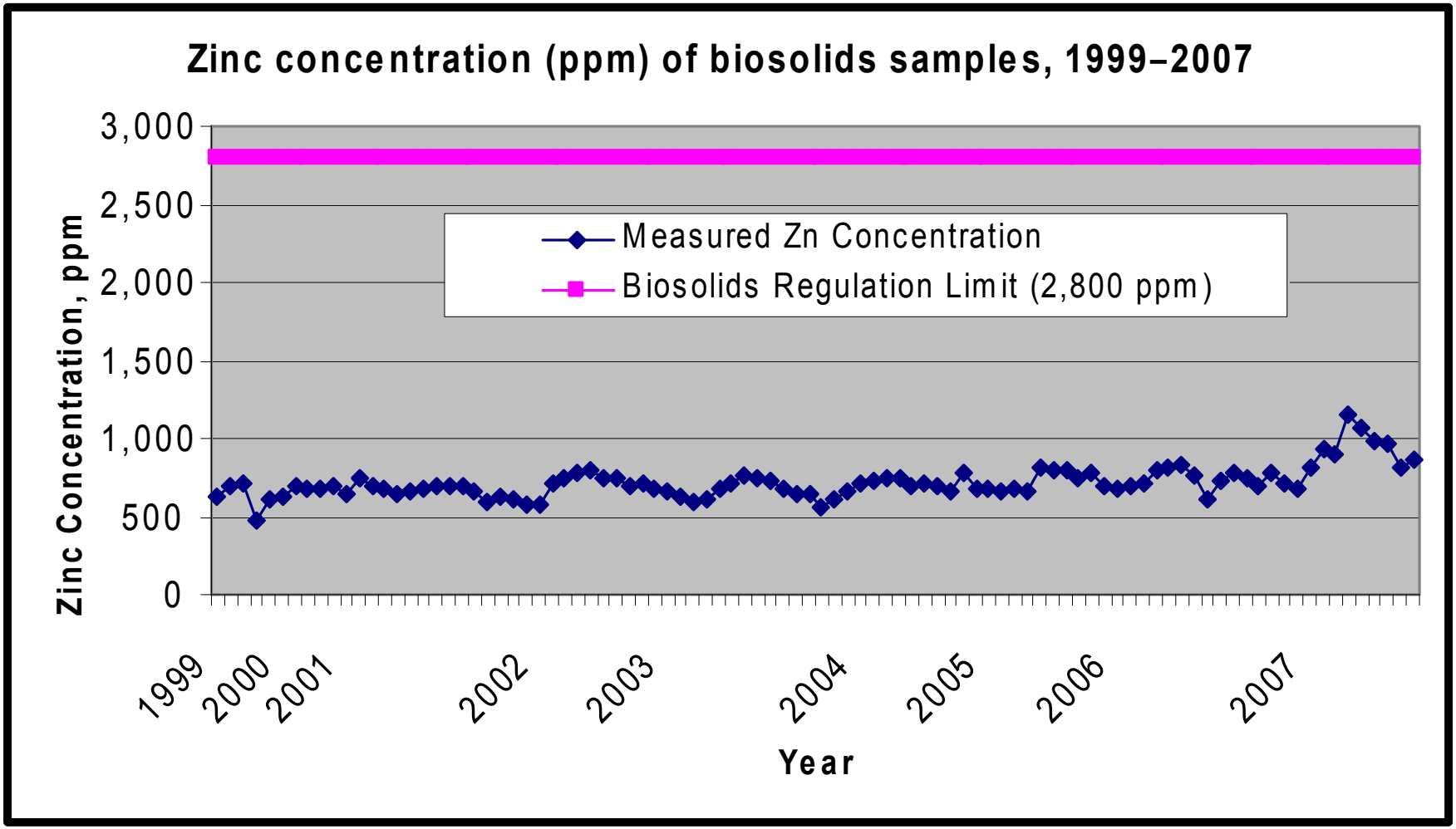


Figure 12. Zinc concentrations of biosolids samples, 1999–2007.

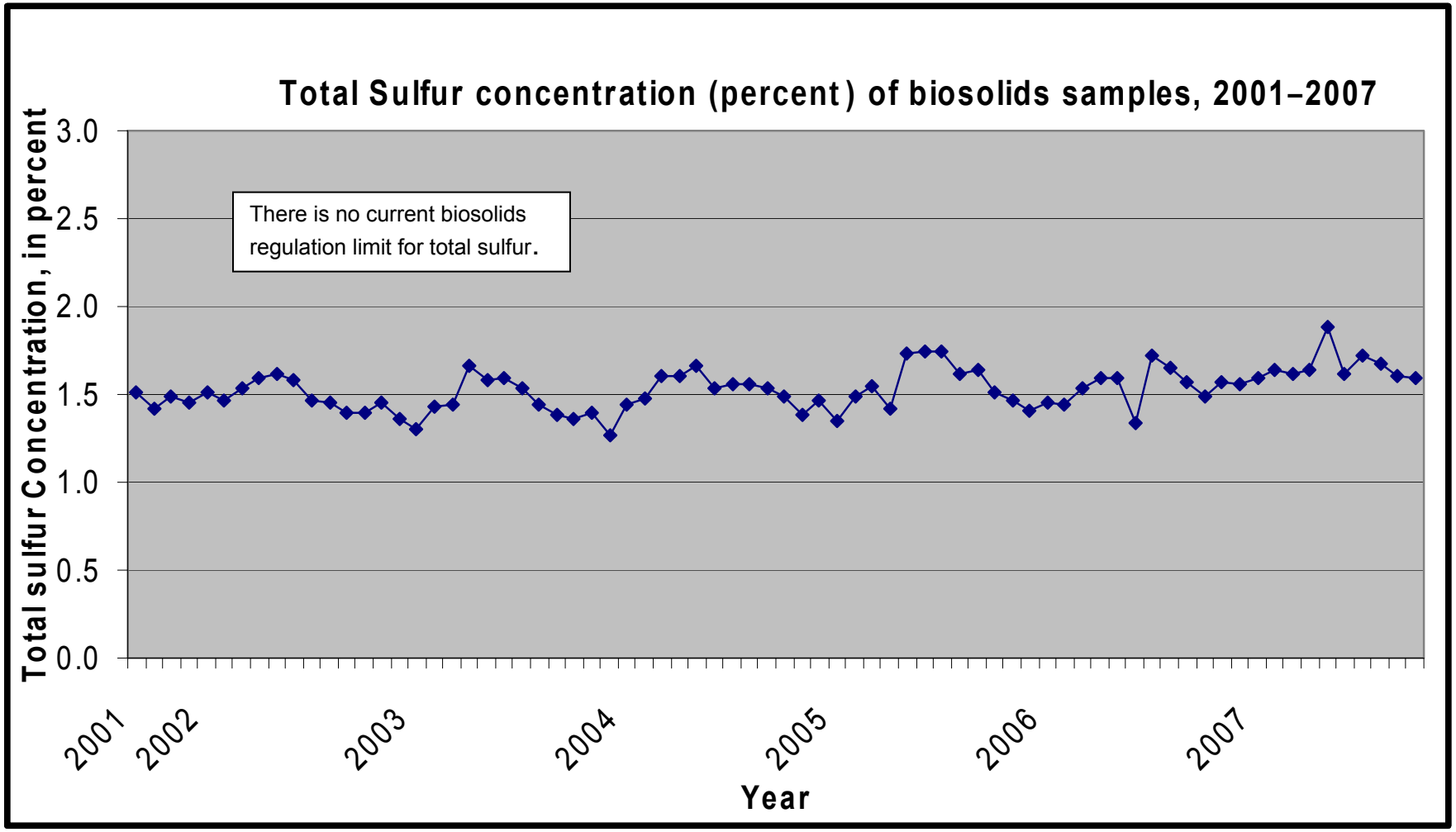


Figure 13. Total sulfur concentration of biosolids samples, 2001–2007.

Table 1. Priority parameters and analytical methods for biosolids samples.

| Element | Method | Reference |
|---|---|---|
| Arsenic | HG-AAS[1] | Hageman and Welch (1996); Taggart (2002) |
| Cadmium | ICP-MS[2] | Briggs and Meier (1999); Taggart (2002) |
| Copper | ICP-MS[2] | Briggs and Meier (1999); Taggart (2002) |
| Lead | ICP-MS[2] | Briggs and Meier (1999); Taggart (2002) |
| Mercury | CV-AFS[3] | Hageman (2007) |
| Molybdenum | ICP-MS[2] | Briggs and Meier (1999); Taggart (2002) |
| Nickel | ICP-MS[2] | Briggs and Meier (1999); Taggart (2002) |
| Selenium | HG-AAS[1] | Hageman and Welch (1996); Taggart (2002) |
| Zinc | ICP-MS[2] | Briggs and Meier (1999); Taggart (2002) |
| Total sulfur | Combustion, IR detection[4] | Brown and Curry (2002) |

[1]Hydride generation – atomic absorption spectrometry.
[2]Inductively coupled plasma – mass spectrometry.
[3]Continuous flow – cold vapor – atomic fluorescence spectrometry.
[4]Automated combustion in oxygen, measured by a solid-state infrared detector.

Table 2. Analytical results for the 2007 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Ag, ppm | ICP–MS Al, % | ICP–MS As, ppm | HG–AAS As, ppm | ICP–MS Ba, ppm | ICP–MS Be, ppm | ICP–MS Bi, ppm | ICP–MS Ca, % | ICP–MS Cd, ppm |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/07 | 16.7 | 1.49 | 2.0 | 0.3 | 381 | 0.35 | 41.8 | 3.36 | 1.9 |
| Bios 02/07 | 15.7 | 1.42 | 2.0 | 0.6 | 402 | 0.36 | 36.6 | 3.24 | 2.1 |
| Bios 03/07 | 14.7 | 1.32 | 1.8 | 0.7 | 365 | 0.32 | 34.1 | 3.22 | 2.3 |
| Bios 04/07 | 18.3 | 1.54 | 2.3 | 0.5 | 414 | 0.40 | 40.9 | 3.89 | 2.6 |
| Bios 05/07 | 19.7 | 1.72 | 3.1 | 1.1 | 450 | 0.42 | 45.1 | 4.30 | 2.5 |
| Bios 06/07 | 22.8 | 1.53 | 2.8 | 1.2 | 422 | 0.40 | 43.6 | 4.13 | 2.4 |
| Bios 07/07 | 23.3 | 1.69 | 3.5 | 1.3 | 539 | 0.46 | 49.8 | 4.56 | 2.9 |
| Bios 08/07 | 19.5 | 1.61 | 3.4 | 1.4 | 457 | 0.47 | 47.5 | 4.54 | 2.6 |
| Bios 09/07 | 18.5 | 1.55 | 3.1 | 1.0 | 430 | 0.40 | 46.9 | 4.24 | 2.4 |
| Bios 10/07 | 17.8 | 1.48 | 2.7 | 0.8 | 426 | 0.32 | 47.9 | 3.98 | 2.3 |
| Bios 11/07 | 16.5 | 1.30 | 2.0 | 0.4 | 372 | 0.32 | 43.0 | 3.64 | 1.9 |
| Bios 12/07 | 17.7 | 1.30 | 2.3 | 0.5 | 400 | 0.29 | 45.8 | 3.58 | 2.2 |
| NIST 2781 | 38.8 | 1.72 | 8.4 | 7.2 | 662 | 0.68 | 34.6 | 4.55 | 13.1 |
| NIST 2781 Recommended/ certified value | 98 +/- 8 | 1.6 +/- 0.1 | 7.82 +/- 0.28 | 7.82 +/- 0.28 | | | | 3.9 +/-0.1 | 12.78 +/-0.72 |

Table 2. Analytical results for the 2007 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Ce, ppm | ICP–MS Co, ppm | ICP–MS Cr, ppm | ICP–MS Cs, ppm | ICP–MS Cu, ppm | ICP–MS Fe, % | ICP–MS Ga, ppm | CV–AAS Hg, ppm | ICP–MS K, % |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/07 | 20.3 | 4.2 | 34 | 0.40 | 714 | 1.87 | 5.0 | 0.61 | 0.31 |
| Bios 02/07 | 24.3 | 4.0 | 35 | 0.50 | 634 | 1.75 | 5.0 | 0.63 | 0.34 |
| Bios 03/07 | 20.8 | 3.8 | 34 | 0.43 | 600 | 1.71 | 4.6 | 0.83 | 0.30 |
| Bios 04/07 | 27.7 | 5.5 | 41 | 0.49 | 748 | 2.36 | 6.1 | 0.76 | 0.36 |
| Bios 05/07 | 27.7 | 5.1 | 47 | 0.63 | 824 | 2.66 | 6.6 | 1.42 | 0.37 |
| Bios 06/07 | 26.4 | 5.0 | 42 | 0.54 | 801 | 2.55 | 6.3 | 0.73 | 0.36 |
| Bios 07/07 | 26.5 | 5.9 | 54 | 0.66 | 952 | 3.47 | 7.6 | 1.03 | 0.40 |
| Bios 08/07 | 29.7 | 6.3 | 47 | 0.61 | 851 | 2.78 | 6.4 | 1.04 | 0.35 |
| Bios 09/07 | 33.6 | 5.6 | 45 | 0.53 | 820 | 2.65 | 5.7 | 1.26 | 0.34 |
| Bios 10/07 | 29.1 | 4.2 | 47 | 0.46 | 816 | 2.42 | 4.8 | 2.38 | 0.37 |
| Bios 11/07 | 26.3 | 3.3 | 39 | 0.40 | 698 | 2.02 | 4.0 | 0.92 | 0.32 |
| Bios 12/07 | 25.5 | 4.0 | 39 | 0.35 | 754 | 1.91 | 3.7 | 0.85 | 0.32 |
| NIST 2781 | 79.3 | 7.0 | 188 | 0.87 | 670 | 3.19 | 7.1 | 2.78 | 0.55 |
| NIST 2781 Recommended/ certified value | | | 202 +/- 9 | | 627.4 +/- 13.5 | 2.8 +/- 0.1 | | 3.64 +/- 0.25 | 0.49 +/- 0.03 |

Table 2. Analytical results for the 2007 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS La, ppm | ICP–MS Li, ppm | ICP–MS Mg, % | ICP–MS Mn, ppm | ICP–MS Mo, ppm | ICP–MS Na, % | ICP–MS Nb, ppm | ICP–MS Ni, ppm | ICP–MS P, % |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/07 | 13.0 | 7.7 | 0.459 | 289 | 22.4 | 0.152 | 63 | 20 | 3.12 |
| Bios 02/07 | 14.7 | 6.8 | 0.437 | 269 | 20.9 | 0.154 | 76 | 20 | 2.51 |
| Bios 03/07 | 12.3 | 5.8 | 0.419 | 298 | 19.8 | 0.147 | 58 | 19 | 2.47 |
| Bios 04/07 | 18.8 | 5.7 | 0.497 | 342 | 24.7 | 0.183 | 82 | 23 | 3.07 |
| Bios 05/07 | 19.9 | 5.7 | 0.544 | 424 | 25.3 | 0.178 | 91 | 26 | 3.33 |
| Bios 06/07 | 19.6 | 4.4 | 0.494 | 378 | 23.8 | 0.171 | 77 | 24 | 3.12 |
| Bios 07/07 | 18.7 | 4.9 | 0.559 | 506 | 34.0 | 0.174 | 96 | 28 | 3.63 |
| Bios 08/07 | 21.4 | 5.1 | 0.473 | 765 | 34.2 | 0.142 | 71 | 26 | 3.03 |
| Bios 09/07 | 23.2 | 3.6 | 0.472 | 1030 | 32.5 | 0.146 | 56 | 24 | 3.06 |
| Bios 10/07 | 20.4 | 4.3 | 0.534 | 570 | 26.5 | 0.161 | 51 | 26 | 3.21 |
| Bios 11/07 | 18.3 | 3.3 | 0.472 | 358 | 22.2 | 0.141 | 48 | 22 | 2.83 |
| Bios 12/07 | 17.0 | 3.6 | 0.429 | 292 | 22.2 | 0.132 | 43 | 21 | 3.06 |
| NIST 2781 | 22.8 | 6.3 | 0.597 | 920 | 47.1 | 0.202 | 89 | 82 | 3.08 |
| NIST 2781 Recommended/ certified value | | | 0.59 +/- 0.04 | | 46.7 +/- 3.2 | 0.21 +/- 0.02 | | 80.2 +/- 2.3 | 2.42 +/- 0.09 |

Table 2. Analytical results for the 2007 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Pb, ppm | ICP–MS Rb, ppm | Total S, IR S, % | ICP–MS Sb, ppm | ICP–MS Sc, ppm | HG–AAS Se, ppm | ICP–MS Sr, ppm | ICP–MS Th, ppm | ICP–MS Ti, % |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/07 | 44 | 8.4 | 1.57 | 1.9 | 2.1 | 11 | 280 | 1.4 | 0.28 |
| Bios 02/07 | 43 | 10.6 | 1.56 | 2.1 | 1.9 | 9.1 | 265 | 1.4 | 0.27 |
| Bios 03/07 | 43 | 9.9 | 1.59 | 2.3 | 1.9 | 9.8 | 255 | 1.4 | 0.24 |
| Bios 04/07 | 54 | 11.8 | 1.64 | 2.4 | 2.3 | 12 | 316 | 2.3 | 0.32 |
| Bios 05/07 | 55 | 12.7 | 1.62 | 2.7 | 2.7 | 13 | 346 | 2.6 | 0.38 |
| Bios 06/07 | 68 | 11.9 | 1.64 | 2.5 | 2.4 | 17 | 323 | 2.4 | 0.34 |
| Bios 07/07 | 68 | 13.3 | 1.88 | 3.4 | 3.0 | 11 | 389 | 3.0 | 0.39 |
| Bios 08/07 | 61 | 12.0 | 1.62 | 2.9 | 2.6 | 14 | 304 | 2.6 | 0.27 |
| Bios 09/07 | 59 | 11.3 | 1.72 | 2.8 | 2.2 | 14 | 300 | 2.1 | 0.33 |
| Bios 10/07 | 58 | 10.6 | 1.67 | 2.6 | 2.0 | 16 | 299 | 2.0 | 0.32 |
| Bios 11/07 | 56 | 9.1 | 1.60 | 2.4 | 1.7 | 12 | 271 | 1.7 | 0.27 |
| Bios 12/07 | 56 | 8.2 | 1.59 | 2.2 | 1.6 | 12 | 301 | 1.3 | 0.29 |
| NIST 2781 | 215 | 18.5 | 1.54 | 7.0 | 90 | 18 | 250 | 6.6 | 0.35 |
| NIST 2781 Recommended/ certified value | 202.1 +/- 6.5 | | | | | 16.0 +/- 1.6 | | | 0.32 +/- 0.03 |

Table 2. Analytical results for the 2007 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Tl, ppm | ICP–MS U, ppm | ICP–MS V, ppm | ICP–MS Y, ppm | ICP–MS Zn, ppm |
|---|---|---|---|---|---|
| Bios 01/07 | 0.1 | 49.0 | 60 | 4.2 | 787 |
| Bios 02/07 | 0.1 | 46.2 | 55 | 4.8 | 705 |
| Bios 03/07 | 0.1 | 51.0 | 55 | 4.0 | 683 |
| Bios 04/07 | 0.1 | 64.6 | 80 | 5.2 | 822 |
| Bios 05/07 | 0.2 | 80.2 | 83 | 5.5 | 935 |
| Bios 06/07 | 0.1 | 86.8 | 52 | 5.3 | 899 |
| Bios 07/07 | 0.2 | 87.0 | 51 | 6.1 | 1,160 |
| Bios 08/07 | 0.2 | 67.0 | 44 | 5.4 | 1,060 |
| Bios 09/07 | 0.1 | 64.5 | 50 | 4.8 | 986 |
| Bios 10/07 | 0.1 | 56.5 | 41 | 4.6 | 962 |
| Bios 11/07 | 0.1 | 50.6 | 26 | 3.9 | 811 |
| Bios 12/07 | 0.1 | 51.4 | 20 | 4.1 | 868 |
| NIST 2781 | 0.3 | 44.3 | 107 | 30.8 | 1,310 |
| NIST 2781 Recommended/ certified value | | | | | 1,273 +/- 53 |

Table 3. Analytical results for the 2006 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Ag, ppm | ICP–MS Al, % | ICP–MS As, ppm | HG–AAS As, ppm | ICP–MS Ba, ppm | ICP–MS Be, ppm | ICP–MS Bi, ppm | ICP–MS Ca, % | ICP–MS Cd, ppm |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 19.3 | 1.18 | 1.6 | 1.6 | 355 | 0.29 | 33.4 | 3.03 | 1.7 |
| Bios 02/06 | 19.0 | 1.10 | 1.8 | 1.5 | 349 | 0.27 | 34.4 | 3.00 | 1.8 |
| Bios 03/06 | 17.6 | 1.14 | 2.0 | 1.7 | 366 | 0.28 | 31.3 | 2.91 | 1.8 |
| Bios 04/06 | 19.5 | 1.12 | 1.7 | 1.4 | 383 | 0.26 | 32.7 | 3.10 | 1.8 |
| Bios 05/06 | 16.7 | 1.14 | 1.9 | 1.9 | 379 | 0.28 | 32.0 | 3.34 | 1.8 |
| Bios 06/06 | 17.7 | 1.05 | 2.2 | 2.1 | 387 | 0.31 | 29.0 | 3.48 | 1.7 |
| Bios 07/06 | 15.5 | 1.35 | 2.6 | 1.9 | 379 | 0.33 | 30.6 | 2.72 | 1.8 |
| Bios 08/06 | 15.1 | 1.91 | 2.0 | 1.8 | 399 | 0.51 | 21.6 | 2.43 | 1.4 |
| Bios 09/06 | 16.6 | 1.29 | 2.5 | 2.0 | 371 | 0.34 | 30.0 | 2.42 | 1.8 |
| Bios 10/06 | 16.7 | 1.28 | 2.5 | 1.9 | 370 | 0.37 | 32.6 | 2.62 | 1.8 |
| Bios 11/05 | 17.2 | 1.32 | 2.5 | 1.9 | 375 | 0.35 | 33.1 | 2.55 | 1.9 |
| Bios 12/06 | 15.3 | 1.11 | 1.9 | 1.5 | 348 | 0.32 | 34.5 | 2.78 | 1.7 |
| NIST 2781 | 83.8 | 1.41 | 7.8 | 7.1 | 601 | 0.51 | 29.4 | 5.20 | 11.1 |
| NIST 2781 | 27.0 | 1.37 | 7.3 | 6.5 | 534 | 0.59 | 28.4 | 3.53 | 10.8 |
| NIST 2781 Recommended/ certified value | 98 +/- 8 | 1.6 +/- 0.1 | 7.82 +/- 0.28 | 7.82 +/- 0.28 | | | | 3.9 +/-0.1 | 12.78 +/-0.72 |

Table 3. Analytical results for the 2006 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Ce, ppm | ICP–MS Co, ppm | ICP–MS Cr, ppm | ICP–MS Cs, ppm | ICP–MS Cu, ppm | ICP–MS Fe, % | ICP–MS Ga, ppm | CV–AAS Hg, ppm | ICP–MS K, % |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 18.3 | 10.7 | 35 | 0.30 | 653 | 2.06 | 6.0 | 0.81 | 0.30 |
| Bios 02/06 | 20.9 | 7.6 | 35 | 0.29 | 708 | 2.11 | 8.1 | 1.1 | 0.28 |
| Bios 03/06 | 17.6 | 6.9 | 36 | 0.31 | 709 | 2.28 | 9.0 | 0.81 | 0.30 |
| Bios 04/06 | 18.7 | 5.2 | 47 | 0.33 | 784 | 2.47 | 10.6 | 0.90 | 0.30 |
| Bios 05/06 | 20.6 | 5.2 | 54 | 0.36 | 845 | 2.66 | 9.9 | 5.2 | 0.35 |
| Bios 06/06 | 27.3 | 6.8 | 56 | 0.40 | 815 | 3.15 | 6.6 | 1.1 | 0.38 |
| Bios 07/06 | 21.7 | 5.4 | 44 | 0.55 | 661 | 2.38 | 5.7 | 0.79 | 0.29 |
| Bios 08/06 | 39.9 | 4.6 | 37 | 0.65 | 554 | 2.21 | 6.0 | 0.91 | 0.69 |
| Bios 09/06 | 21.0 | 5.1 | 33 | 0.46 | 641 | 2.07 | 5.5 | 0.83 | 0.30 |
| Bios 10/06 | 18.8 | 4.8 | 36 | 0.44 | 670 | 2.00 | 5.6 | 0.96 | 0.27 |
| Bios 11/05 | 18.4 | 5.1 | 34 | 0.44 | 681 | 2.06 | 5.8 | 0.81 | 0.32 |
| Bios 12/06 | 18.3 | 4.2 | 31 | 0.34 | 617 | 1.78 | 4.6 | 0.66 | 0.26 |
| NIST 2781 | 69.8 | 7.2 | 218 | 0.80 | 685 | 3.24 | 7.2 | 3.8 | 0.59 |
| NIST 2781 | 69.1 | 5.9 | 150 | 0.73 | 600 | 2.63 | 6.3 | 2.4 | 0.43 |
| NIST 2781 Recommended/ certified value | | | 202 +/- 9 | | 627.4 +/- 13.5 | 2.8 +/- 0.1 | | 3.64 +/- 0.25 | 0.49 +/- 0.03 |

Table 3. Analytical results for the 2006 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS La, ppm | ICP–MS Li, ppm | ICP–MS Mg, % | ICP–MS Mn, ppm | ICP–MS Mo, ppm | ICP–MS Na, % | ICP–MS Nb, ppm | ICP–MS Ni, ppm | ICP–MS P, % |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 11.6 | 2.6 | 0.410 | 260 | 25.4 | 0.130 | 72 | 19 | 2.73 |
| Bios 02/06 | 13.3 | 1.6 | 0.502 | 279 | 22.7 | 0.108 | 78 | 19 | 2.84 |
| Bios 03/06 | 11.1 | 1.1 | 0.346 | 291 | 21.9 | 0.111 | 94 | 19 | 2.59 |
| Bios 04/06 | 12.1 | 1.9 | 0.316 | 288 | 23.9 | 0.114 | 120 | 20 | 2.62 |
| Bios 05/06 | 13.0 | 1.6 | 0.343 | 329 | 32.2 | 0.128 | 110 | 22 | 2.71 |
| Bios 06/06 | 18.8 | 1.6 | 0.443 | 419 | 44.1 | 0.117 | 60 | 23 | 2.92 |
| Bios 07/06 | 17.1 | 4.4 | 0.468 | 331 | 40.7 | 0.123 | 48 | 21 | 2.66 |
| Bios 08/06 | 24.5 | 4.9 | 1.51 | 407 | 28.6 | 0.277 | 39 | 21 | 3.67 |
| Bios 09/06 | 17.5 | 3.8 | 0.392 | 453 | 35.1 | 0.133 | 54 | 20 | 2.46 |
| Bios 10/06 | 16.0 | 3.7 | 0.435 | 338 | 30.0 | 0.125 | 53 | 21 | 2.57 |
| Bios 11/05 | 15.1 | 4.0 | 0.416 | 260 | 26.4 | 0.149 | 74 | 22 | 2.67 |
| Bios 12/06 | 14.5 | 2.9 | 0.349 | 185 | 22.1 | 0.123 | 66 | 17 | 2.42 |
| NIST 2781 | 20.1 | 4.6 | 0.485 | 976 | 40.6 | 0.178 | 78 | 87 | 3.06 |
| NIST 2781 | 19.4 | 5.9 | 0.499 | 742 | 37.3 | 0.183 | 75 | 71 | 2.53 |
| NIST 2781 Recommended/ certified value | | | 0.59 +/- 0.04 | | 46.7 +/- 3.2 | 0.21 +/- 0.02 | | 80.2 +/- 2.3 | 2.42 +/- 0.09 |

Table 3. Analytical results for the 2006 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Pb, ppm | ICP–MS Rb, ppm | Total S, IR S, % | ICP–MS Sb, ppm | ICP–MS Sc, ppm | HG–AAS Se, ppm | ICP–MS Sr, ppm | ICP–MS Th, ppm | ICP–MS Ti, % |
|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 45 | 7.4 | 1.47 | 2.4 | 1.6 | 8.0 | 231 | 1.6 | 0.26 |
| Bios 02/06 | 44 | 6.8 | 1.41 | 2.1 | 1.8 | 8.2 | 238 | 1.5 | 0.29 |
| Bios 03/06 | 47 | 7.5 | 1.45 | 2.2 | 2.1 | 6.9 | 255 | 1.6 | 0.30 |
| Bios 04/06 | 52 | 8.1 | 1.44 | 2.5 | 2.4 | 7.4 | 265 | 1.7 | 0.35 |
| Bios 05/06 | 47 | 9.0 | 1.54 | 2.0 | 2.4 | 8.8 | 266 | 1.7 | 0.28 |
| Bios 06/06 | 52 | 9.8 | 1.59 | 1.9 | 2.2 | 9.1 | 243 | 2.0 | 0.29 |
| Bios 07/06 | 52 | 10.5 | 1.59 | 2.5 | 1.8 | 10 | 213 | 1.8 | 0.24 |
| Bios 08/06 | 72 | 25.1 | 1.34 | 1.8 | 2.0 | 7.4 | 206 | 11.4 | 0.18 |
| Bios 09/06 | 51 | 9.9 | 1.72 | 1.9 | 1.6 | 10 | 221 | 2.1 | 0.22 |
| Bios 10/06 | 51 | 8.8 | 1.65 | 2.0 | 1.6 | 11 | 228 | 1.8 | 0.22 |
| Bios 11/05 | 52 | 10.2 | 1.57 | 1.9 | 1.7 | 9.6 | 244 | 1.9 | 0.22 |
| Bios 12/06 | 46 | 7.6 | 1.49 | 2.3 | 1.6 | 9.4 | 248 | 1.8 | 0.22 |
| NIST 2781 | 188 | 17.4 | 1.44 | 5.5 | 97.6 | 14 | 248 | 5.3 | 0.35 |
| NIST 2781 | 185 | 15.6 | 1.48 | 5.2 | 66.4 | 15 | 213 | 4.9 | 0.20 |
| NIST 2781 Recommended/ certified value | 202.1 +/- 6.5 | | | | | 16.0 +/- 1.6 | | | 0.32 +/- 0.03 |

Table 3. Analytical results for the 2006 biosolids samples using the traditional methods of digestion.

| Sample ID | ICP–MS Tl, ppm | ICP–MS U, ppm | ICP–MS V, ppm | ICP–MS Y, ppm | ICP–MS Zn, ppm |
|---|---|---|---|---|---|
| Bios 01/06 | 0.1 | 32.1 | 66 | 3.6 | 684 |
| Bios 02/06 | 0.1 | 33.0 | 81 | 3.8 | 696 |
| Bios 03/06 | 0.1 | 32.0 | 97 | 3.7 | 716 |
| Bios 04/06 | 0.1 | 33.5 | 122 | 3.9 | 794 |
| Bios 05/06 | 0.1 | 35.6 | 125 | 4.4 | 819 |
| Bios 06/06 | 0.1 | 35.8 | 77 | 4.5 | 831 |
| Bios 07/06 | 0.1 | 38.7 | 50 | 4.5 | 769 |
| Bios 08/06 | 0.2 | 26.6 | 42 | 8.5 | 609 |
| Bios 09/06 | 0.1 | 38.2 | 47 | 4.0 | 728 |
| Bios 10/06 | 0.1 | 39.8 | 53 | 4.2 | 787 |
| Bios 11/05 | 0.1 | 42.2 | 63 | 4.2 | 750 |
| Bios 12/06 | 0.1 | 38.0 | 50 | 3.8 | 690 |
| NIST 2781 | 0.3 | 35.2 | 116 | 30 | 1,320 |
| NIST 2781 | 0.3 | 37.8 | 83 | 25 | 1,120 |
| NIST 2781 Recommended/ certified value | | | | | 1,273 +/- 53 |

Table 4. Analytical results for the 2006 and 2007 biosolids samples using the microwave digestion method.

| Sample ID | ICP–MS Ag, ppm | ICP–MS Al, % | ICP–MS As, ppm | ICP–MS Ba, ppm | ICP–MS Be, ppm | ICP–MS Bi, ppm | ICP–MS Ca, % | ICP–MS Cd, ppm | ICP–MS Ce, ppm | ICP–MS Co, ppm | ICP–MS Cr, ppm | ICP–MS Cs, ppm | ICP–MS Cu, ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 20.3 | 0.93 | 1.9 | 332 | 0.21 | 35.0 | 2.73 | 1.9 | 11.5 | 10.3 | 29 | 0.25 | 607 |
| Bios 02/06 | 20.5 | 0.93 | 1.7 | 340 | 0.20 | 36.0 | 2.68 | 2.0 | 14.0 | 6.6 | 30 | 0.25 | 647 |
| Bios 03/06 | 18.3 | 0.89 | 1.7 | 344 | 0.22 | 33.0 | 2.48 | 1.9 | 10.9 | 5.6 | 30 | 0.25 | 610 |
| Bios 04/06 | 21.1 | 0.91 | 1.5 | 353 | 0.22 | 33.6 | 2.54 | 2.0 | 11.1 | 4.3 | 33 | 0.25 | 668 |
| Bios 05/06 | 17.8 | 0.93 | 2.1 | 349 | 0.23 | 34.6 | 2.58 | 1.8 | 12.8 | 4.5 | 42 | 0.29 | 702 |
| Bios 06/06 | 20.1 | 0.92 | 2.2 | 359 | 0.23 | 32.1 | 2.68 | 1.8 | 18.0 | 5.5 | 38 | 0.30 | 676 |
| Bios 07/06 | 17.0 | 1.04 | 2.4 | 352 | 0.31 | 33.3 | 2.85 | 2.1 | 14.3 | 5.7 | 41 | 0.46 | 668 |
| Bios 08/06 | 16.9 | 0.97 | 2.3 | 281 | 0.23 | 23.8 | 2.59 | 1.4 | 13.6 | 4.7 | 32 | 0.48 | 591 |
| Bios 09/06 | 17.7 | 1.01 | 2.8 | 360 | 0.23 | 33.1 | 2.65 | 2.1 | 13.4 | 5.2 | 34 | 0.38 | 676 |
| Bios 10/06 | 16.0 | 1.05 | 2.7 | 364 | 0.26 | 35.9 | 2.87 | 1.9 | 14.3 | 5.0 | 34 | 0.39 | 715 |
| Bios 11/05 | 18.4 | 1.00 | 2.3 | 344 | 0.31 | 36.5 | 2.76 | 2.0 | 13.0 | 4.6 | 33 | 0.36 | 660 |
| Bios 12/06 | 17.4 | 0.94 | 2.0 | 351 | 0.27 | 39.4 | 3.08 | 1.9 | 12.8 | 4.4 | 31 | 0.30 | 657 |
| Bios 01/07 | 15.0 | 1.00 | 2.0 | 335 | 0.26 | 42.0 | 2.88 | 1.9 | 10.9 | 3.5 | 30 | 0.30 | 647 |
| Bios 02/07 | 19.4 | 1.20 | 1.9 | 386 | 0.32 | 43.1 | 3.47 | 2.4 | 15.8 | 4.0 | 38 | 0.45 | 666 |
| Bios 03/07 | 19.2 | 1.10 | 2.1 | 345 | 0.35 | 39.8 | 3.44 | 2.6 | 14.6 | 4.0 | 36 | 0.38 | 634 |
| Bios 04/07 | 15.7 | 1.08 | 2.1 | 330 | 0.31 | 40.7 | 3.56 | 2.2 | 14.9 | 4.8 | 36 | 0.36 | 652 |
| Bios 05/07 | 15.6 | 1.03 | 2.3 | 316 | 0.28 | 36.7 | 3.29 | 2.0 | 13.4 | 3.7 | 34 | 0.39 | 607 |
| Bios 06/07 | 22.1 | 1.12 | 2.7 | 354 | 0.27 | 42.6 | 3.87 | 2.4 | 16.6 | 4.5 | 36 | 0.40 | 728 |
| Bios 07/07 | 19.1 | 1.12 | 2.4 | 401 | 0.33 | 42.6 | 3.69 | 2.5 | 12.9 | 4.4 | 38 | 0.44 | 740 |
| Bios 08/07 | 18.2 | 1.18 | 2.8 | 392 | 0.29 | 46.9 | 4.09 | 2.4 | 16.9 | 4.9 | 41 | 0.48 | 757 |
| Bios 09/07 | 16.0 | 0.97 | 2.3 | 318 | 0.28 | 38.8 | 3.24 | 2.0 | 13.8 | 4.2 | 31 | 0.36 | 629 |
| Bios 10/07 | 13.3 | 1.09 | 2.3 | 361 | 0.24 | 46.2 | 3.49 | 2.4 | 14.1 | 3.5 | 40 | 0.37 | 727 |
| Bios 11/07 | 16.2 | 0.99 | 2.0 | 331 | 0.29 | 44.4 | 3.29 | 2.0 | 14.8 | 3.0 | 36 | 0.34 | 674 |
| Bios 12/07 | 15.8 | 1.01 | 2.1 | 349 | 0.24 | 46.4 | 3.11 | 2.0 | 15.1 | 3.2 | 35 | 0.26 | 676 |
| NIST 2781 | 27.1 | 1.03 | 7.4 | 555 | 0.41 | 31.5 | 3.77 | 12.1 | 48.0 | 6.0 | 158 | 0.66 | 608 |
| NIST 2781 | 26.4 | 1.12 | 6.6 | 558 | 0.44 | 32.1 | 3.80 | 12.2 | 42.8 | 5.8 | 153 | 0.66 | 583 |
| NIST 2781 | 27.0 | 0.99 | 7.7 | 555 | 0.35 | 31.9 | 3.75 | 12.1 | 53.5 | 6.0 | 156 | 0.67 | 612 |
| NIST 2781 Recommended/ certified value | 98 +/- 8 | 1.6 +/- 0.1 | 7.82 +/- 0.28 | | | | 3.9 +/-0.1 | 12.78 +/- 0.72 | | | 202 +/- 9 | | 627.4 +/- 13.5 |

Table 4. Analytical results for the 2006 and 2007 biosolids samples using the microwave digestion method.

| Sample ID | ICP–MS Fe, % | ICP–MS Ga, ppm | ICP–MS K, % | ICP–MS La, ppm | ICP–MS Li, ppm | ICP–MS Mg, % | ICP–MS Mn, ppm | ICP–MS Mo, ppm | ICP–MS Na, % | ICP–MS Nb, ppm | ICP–MS Ni, ppm | ICP–MS P, % | ICP–MS Pb, ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 1.93 | 5.5 | 0.17 | 10.3 | 3.5 | 0.332 | 227 | 24.9 | 0.058 | 35 | 17 | 2.52 | 45 |
| Bios 02/06 | 1.90 | 7.2 | 0.17 | 12.0 | 3.3 | 0.469 | 252 | 22.0 | 0.053 | 30 | 17 | 2.73 | 43 |
| Bios 03/06 | 1.97 | 7.8 | 0.18 | 9.9 | 3.0 | 0.320 | 255 | 21.1 | 0.057 | 47 | 16 | 2.52 | 47 |
| Bios 04/06 | 2.10 | 8.8 | 0.17 | 10.0 | 2.9 | 0.320 | 238 | 23.7 | 0.057 | 55 | 20 | 2.53 | 64 |
| Bios 05/06 | 2.22 | 8.3 | 0.18 | 12.2 | 3.1 | 0.347 | 274 | 31.4 | 0.065 | 49 | 20 | 2.67 | 50 |
| Bios 06/06 | 2.67 | 5.2 | 0.18 | 17.2 | 3.0 | 0.475 | 341 | 43.6 | 0.054 | 31 | 20 | 2.85 | 55 |
| Bios 07/06 | 2.46 | 4.8 | 0.17 | 14.6 | 3.3 | 0.432 | 347 | 42.6 | 0.044 | 25 | 22 | 2.64 | 51 |
| Bios 08/06 | 2.19 | 4.0 | 0.16 | 12.0 | 3.9 | 1.590 | 400 | 29.6 | 0.041 | 18 | 20 | 3.83 | 95 |
| Bios 09/06 | 2.19 | 4.8 | 0.18 | 15.3 | 3.4 | 0.381 | 513 | 36.1 | 0.053 | 30 | 20 | 2.60 | 53 |
| Bios 10/06 | 2.13 | 5.2 | 0.18 | 14.9 | 3.3 | 0.416 | 375 | 31.2 | 0.053 | 27 | 21 | 2.74 | 49 |
| Bios 11/05 | 2.05 | 4.9 | 0.20 | 13.5 | 3.3 | 0.404 | 267 | 26.7 | 0.057 | 38 | 20 | 2.72 | 55 |
| Bios 12/06 | 1.92 | 4.3 | 0.18 | 14.0 | 3.2 | 0.347 | 189 | 22.7 | 0.056 | 33 | 18 | 2.62 | 50 |
| Bios 01/07 | 1.56 | 3.9 | 0.18 | 9.3 | 1.8 | 0.364 | 267 | 20.5 | 0.052 | 29 | 17 | 2.54 | 46 |
| Bios 02/07 | 1.86 | 4.6 | 0.22 | 12.7 | 3.6 | 0.410 | 294 | 23.6 | 0.056 | 38 | 22 | 2.68 | 48 |
| Bios 03/07 | 1.81 | 4.4 | 0.18 | 11.6 | 3.0 | 0.387 | 321 | 21.9 | 0.051 | 30 | 21 | 2.58 | 47 |
| Bios 04/07 | 2.02 | 4.6 | 0.19 | 13.3 | 2.1 | 0.403 | 301 | 22.2 | 0.057 | 31 | 20 | 2.66 | 51 |
| Bios 05/07 | 1.94 | 4.3 | 0.17 | 12.4 | 2.0 | 0.382 | 318 | 18.9 | 0.051 | 36 | 19 | 2.51 | 42 |
| Bios 06/07 | 2.26 | 5.3 | 0.20 | 15.1 | 2.4 | 0.413 | 350 | 21.5 | 0.059 | 34 | 21 | 2.81 | 60 |
| Bios 07/07 | 2.70 | 5.3 | 0.18 | 12.7 | 2.5 | 0.404 | 392 | 26.4 | 0.054 | 38 | 23 | 2.82 | 55 |
| Bios 08/07 | 2.48 | 5.2 | 0.19 | 15.8 | 2.9 | 0.395 | 699 | 31.8 | 0.052 | 29 | 26 | 2.66 | 61 |
| Bios 09/07 | 1.99 | 3.9 | 0.16 | 15.9 | 1.6 | 0.335 | 807 | 25.0 | 0.040 | 20 | 18 | 2.36 | 45 |
| Bios 10/07 | 2.15 | 3.7 | 0.19 | 15.2 | 2.2 | 0.447 | 522 | 24.2 | 0.063 | 21 | 24 | 2.82 | 52 |
| Bios 11/07 | 1.90 | 3.3 | 0.17 | 16.0 | 2.1 | 0.418 | 338 | 21.5 | 0.051 | 21 | 20 | 2.63 | 51 |
| Bios 12/07 | 1.72 | 2.9 | 0.18 | 14.8 | 2.3 | 0.379 | 264 | 20.8 | 0.053 | 20 | 20 | 2.68 | 46 |
| NIST 2781 | 2.52 | 5.5 | 0.26 | 18.4 | 5.4 | 0.492 | 819 | 37.1 | 0.084 | 22 | 74 | 2.58 | 186 |
| NIST 2781 | 2.45 | 5.2 | 0.26 | 16.9 | 4.5 | 0.510 | 796 | 38.5 | 0.064 | 19 | 72 | 2.46 | 193 |
| NIST 2781 | 2.51 | 5.5 | 0.26 | 20.6 | 6.2 | 0.481 | 812 | 36.3 | 0.082 | 22 | 73 | 2.43 | 192 |
| NIST 2781 Recommended/ certified value | 2.8 +/- 0.1 | | 0.49 +/- 0.03 | | | 0.59 +/- 0.04 | | 46.7 +/- 3.2 | 0.21 +/- 0.02 | | 80.2 +/- 2.3 | 2.42 +/- 0.09 | 202.1 +/- 6.5 |

Table 4. Analytical results for the 2006 and 2007 biosolids samples using the microwave digestion method.

| Sample ID | ICP–MS Rb, ppm | ICP–MS Sb, ppm | ICP–MS Sc, ppm | ICP–MS Sr, ppm | ICP–MS Th, ppm | ICP–MS Ti, % | ICP–MS Tl, ppm | ICP–MS U, ppm | ICP–MS V, ppm | ICP–MS Y, ppm | ICP–MS Zn, ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Bios 01/06 | 3.8 | 1.1 | 1.3 | 212 | 0.8 | 0.04 | <0.1 | 31.5 | 61 | 2.9 | 652 |
| Bios 02/06 | 3.7 | 0.8 | 1.4 | 212 | 0.9 | 0.03 | <0.1 | 32.1 | 75 | 3.3 | 650 |
| Bios 03/06 | 3.7 | 1.2 | 1.1 | 221 | 0.5 | 0.05 | <0.1 | 31.5 | 85 | 2.9 | 629 |
| Bios 04/06 | 3.9 | 1.1 | 1.3 | 237 | 0.5 | 0.05 | <0.1 | 32.4 | 99 | 3.1 | 698 |
| Bios 05/06 | 4.2 | 1.2 | 1.4 | 222 | 0.8 | 0.05 | <0.1 | 37.5 | 101 | 3.2 | 706 |
| Bios 06/06 | 4.5 | 1.0 | 1.2 | 220 | 0.9 | 0.03 | <0.1 | 38.3 | 59 | 3.1 | 710 |
| Bios 07/06 | 6.0 | 0.9 | 1.4 | 211 | 0.8 | 0.03 | 0.1 | 37.5 | 51 | 3.6 | 779 |
| Bios 08/06 | 6.9 | 0.8 | 1.4 | 182 | 1.0 | 0.02 | 0.1 | 26.5 | 37 | 4.5 | 626 |
| Bios 09/06 | 5.3 | 1.0 | 1.3 | 230 | 0.6 | 0.03 | 0.1 | 37.8 | 50 | 3.3 | 756 |
| Bios 10/06 | 5.3 | 1.2 | 1.5 | 237 | 0.8 | 0.03 | 0.1 | 38.9 | 56 | 3.4 | 830 |
| Bios 11/05 | 5.2 | 1.2 | 1.5 | 235 | 0.8 | 0.04 | 0.1 | 40.9 | 66 | 3.6 | 748 |
| Bios 12/06 | 4.4 | 1.3 | 1.4 | 266 | 0.8 | 0.04 | <0.1 | 38.7 | 52 | 3.4 | 730 |
| Bios 01/07 | 4.3 | 0.9 | 1.1 | 255 | 0.7 | 0.03 | <0.1 | 48.1 | 50 | 3.2 | 689 |
| Bios 02/07 | 6.6 | 1.4 | 1.6 | 291 | 1.2 | 0.04 | 0.1 | 54.7 | 59 | 4.4 | 718 |
| Bios 03/07 | 5.7 | 1.4 | 1.4 | 273 | 1.1 | 0.04 | 0.1 | 56.9 | 56 | 3.7 | 687 |
| Bios 04/07 | 5.5 | 1.1 | 1.6 | 282 | 1.4 | 0.04 | 0.1 | 60.5 | 69 | 3.6 | 680 |
| Bios 05/07 | 5.5 | 1.0 | 1.2 | 267 | 0.8 | 0.03 | 0.1 | 63.2 | 60 | 3.6 | 676 |
| Bios 06/07 | 5.9 | 1.2 | 1.7 | 309 | 1.2 | 0.04 | 0.1 | 83.4 | 44 | 3.9 | 764 |
| Bios 07/07 | 6.0 | 1.2 | 1.6 | 322 | 1.2 | 0.04 | 0.1 | 73.0 | 37 | 4.0 | 891 |
| Bios 08/07 | 6.5 | 1.2 | 1.9 | 291 | 1.5 | 0.03 | 0.1 | 65.3 | 37 | 4.2 | 914 |
| Bios 09/07 | 5.3 | 1.0 | 1.2 | 244 | 1.0 | 0.02 | 0.1 | 51.8 | 35 | 3.2 | 740 |
| Bios 10/07 | 5.4 | 1.2 | 1.4 | 288 | 1.4 | 0.03 | 0.1 | 52.3 | 33 | 3.5 | 835 |
| Bios 11/07 | 4.7 | 1.2 | 1.3 | 277 | 1.1 | 0.03 | 0.1 | 49.0 | 23 | 3.1 | 739 |
| Bios 12/07 | 4.1 | 1.2 | 1.1 | 292 | 1.0 | 0.03 | 0.1 | 50.8 | 16 | 2.9 | 761 |
| NIST 2781 | 9.3 | 1.4 | 19.2 | 216 | 3.2 | 0.02 | 0.2 | 36.3 | 88 | 20.0 | 1,160 |
| NIST 2781 | 9.5 | 1.2 | 19.0 | 221 | 3.5 | 0.01 | 0.2 | 39.4 | 83 | 20.5 | 1,100 |
| NIST 2781 | 9.6 | 1.4 | 18.6 | 215 | 3.4 | 0.02 | 0.2 | 36.9 | 86 | 20.0 | 1,150 |
| NIST 2781 Recommended/ certified value | | | | | | 0.32 +/- 0.03 | | | | | 1,273 +/- 53 |

Table 5. Differences between the traditional four-acid digestion and the microwave digestion for the elements reported by ICP–MS determination.

[%, percent; ppm, parts per million]

| | | | Range of absolute differences (n=27) | |
|---|---|---|---|---|
| Element, concentration units | Average difference, n=27, (Microwave, four-Acid) | Difference as relative percentage of the average four-acid value | Maximum (in concentration units of the element) | Minimum (in concentration units of the element) |
| Ag, ppm | -2.6 | -12.0 | 4.5 | -57.4 |
| Al, % | -0.37 | -26.5 | -0.13 | -0.94 |
| As, ppm | -0.2 | -6.8 | 0.4 | -1.2 |
| Ba, ppm | -48 | -11.5 | 21 | -138 |
| Be, ppm | -0.10 | -25.7 | 0.03 | -0.28 |
| Bi, ppm | 0.8 | 2.2 | 6.5 | -8.4 |
| Ca, % | -0.34 | -9.7 | 0.30 | -1.40 |
| Cd, ppm | 0.1 | 2.3 | 1.3 | -1.0 |
| Ce, ppm | -11.8 | -40.1 | -4.5 | -31.3 |
| Co, ppm | -0.6 | -11.2 | 0.3 | -1.5 |
| Cr, ppm | -9 | -15.0 | 6 | -65 |
| Cs, ppm | -0.10 | -21.0 | -0.04 | -0.24 |
| Cu, ppm | -63 | -8.7 | 45 | -217 |
| Fe, % | -0.25 | -10.6 | 0.14 | -0.79 |
| Ga, ppm | -1.2 | -18.5 | -0.2 | -2.3 |
| K, % | -0.17 | -47.6 | -0.08 | -0.53 |
| La, ppm | -3.2 | -18.9 | 1.2 | -12.5 |
| Li, ppm | -1.0 | -25.0 | 1.9 | -5.9 |
| Mg, % | -0.046 | -9.4 | 0.080 | -0.162 |
| Mn, ppm | -38 | -8.5 | 70 | -223 |
| Mo, ppm | -1.5 | -5.2 | 2.7 | -10.0 |
| Na, % | -0.095 | -62.8 | -0.054 | -0.236 |
| Nb, ppm | -41 | -56.9 | -21 | -67 |
| Ni, ppm | -2 | -8.5 | 2 | -15 |
| P, % | -0.21 | -7.4 | 0.20 | -0.82 |
| Pb, ppm | -1 | -1.7 | 23 | -29 |
| Rb, ppm | -5.6 | -50.1 | -3.1 | -18.2 |
| Sb, ppm | -1.6 | -58.0 | -0.7 | -5.6 |
| Sc, ppm | -7.9 | -70.3 | -0.1 | -78.6 |
| Sr, ppm | -18 | -6.8 | 26 | -79 |
| Th, ppm | -1.5 | -54.7 | -0.2 | -10.4 |
| Ti, % | -0.25 | -88.7 | -0.16 | -0.36 |
| Tl, ppm | 0.0 | -30.9 | 0.0 | -0.1 |
| U, ppm | -1.9 | -4.0 | 8.5 | -17.0 |
| V, ppm | -8 | -12.7 | 4 | -33 |
| Y, ppm | -2.0 | -26.8 | -0.3 | -10.8 |
| Zn, ppm | -85 | -9.8 | 43 | -269 |