Analytical Results for Municipal Biosolids Samples from a Monitoring Program Near Deer Trail, Colorado (U.S.A.), 2008

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, Metro Wastewater Reclamation District of Denver (Metro District), a large wastewater treatment plant in Denver, Colo., has applied Grade I, Class B biosolids to about 52,000 acres of nonirrigated farmland and rangeland near Deer Trail, Colo. (U.S.A.). In cooperation with the Metro District in 1993, the U.S. Geological Survey (USGS) began monitoring groundwater at part of this site (Yager and Arnold, 2003). 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 has recently 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 groundwater, and stream-bed sediment. Streams at the site are dry most of the year, so samples of stream-bed 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 2008. Crock and others (2008a) have presented earlier a compilation of analytical results for the biosolids samples collected and analyzed for 1999 thru 2006, and in a separate report (Crock and others, 2008b), data for the 2007 biosolids are reported. More information about the other monitoring components is presented elsewhere in the literature (for example, Yager and others, 2004a, 2004b, 2004c, 2004d, 2009). 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 groundwater 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 1999–2007, and this consistency continues with the samples for 2008, and total concentrations of regulated trace elements remain consistently lower than the regulatory limits for the entire monitoring period.

Data from the previous reports (Crock and others, 2008a,b) and 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, and phosphorus.

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, Colo. (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 can be found 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,b,c; 2009). As more information becomes available, it will be posted at the U.S. Geological Survey (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, Colo. The Metro District property and surrounding private property are herein referred to as “the study area.”

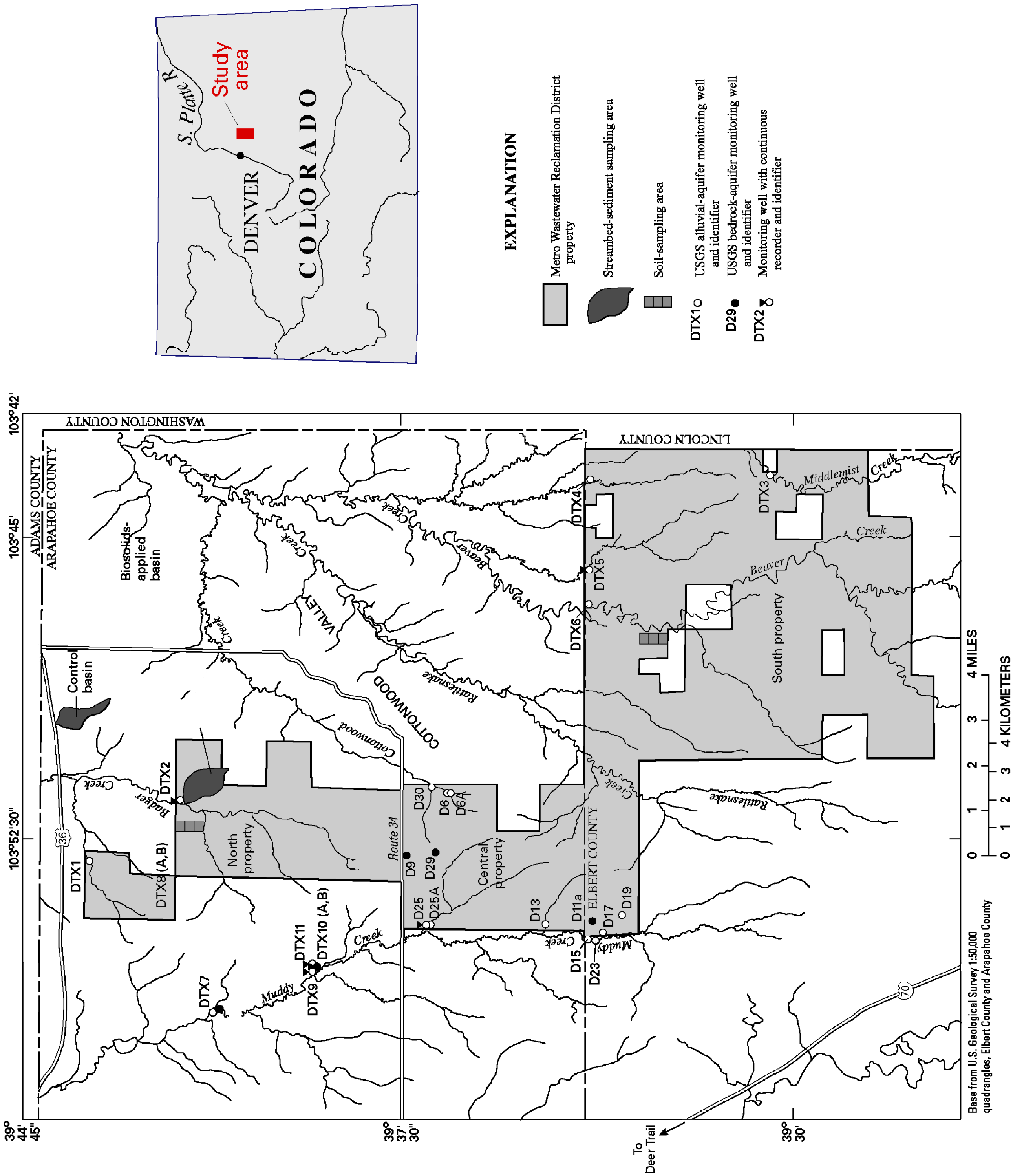

Figure 1. Metro Wastewater Reclamation District of Denver (Metro District) biosolids-application farm and study area location.

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 2008 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 indicated by 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 groundwater from the Lowry Landfill Superfund site in Denver. The 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, Colo., during 1999 through 2008. 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, bedrock aquifer, alluvial aquifers, and stream-bed sediments by comparing chemical data to regulatory standards, data from a site where biosolids have not been 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 2008 analytical data for biosolids only. Analytical results for biosolids collected between 1999 and 2007 can be found in Crock and others (2008a,b). A complete discussion of findings for all matrices and the other study area objectives is detailed in Yager and others (2004d, 2009).

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 District plant spread out in a plastic-lined box to dry.

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

Biosolids are moist (usually ranging 75–85 percent moisture) and have a firm, pudding-like 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 can be found at the following websites:

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,b,c; 2009).

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. One sample during 2007 (Bios06/07–June 2007) and one sample for 2008 (Bios06/08–June 2008) have been analyzed for gross alpha and gross beta radioactivity and plutonium isotopes. Analyses show no detectable plutonium in either sample. Negative results are an artifact of the counting statistics and should be viewed as less than the minimal detectable concentration for that analyte. Consult table 1 for a complete list of the priority elements determined by the various analytical methods. Additional elements were determined by the multi-element inductively coupled plasma–mass spectrometry (ICP–MS) method (Briggs and Meier, 1999; Taggart, 2002).

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, Geologic Discipline, Denver, Colo. 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,b,c; 2009). For quality control and quality assurance control, the National Institute of Standards and Technology (NIST) standard reference material (SRM) 2781 for domestic sludge was analyzed with the 2008 biosolids samples.

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 2008 data will also augment the chemical baseline that has been established earlier by Crock and others (2008a,b) against which any future change in the concentration of constituents analyzed for in this study may be recognized, measured, and compared. This data set will also enhance the "geochemical signature" for biosolids that will potentially enable scientists to recognize when biosolids have impacted soils or stream sediments.

All data for the 1999–2007 biosolids samples are presented in Crock and others (2008a,b) and are presented in figures 4–13 supplemented with the 2008 data. The concentration of all nine trace elements remained consistent throughout the study (1999–2008) and below the Grade 1 biosolids requirements. Reference material NIST SRM 2781 results are also presented in table 2. The certificate of analysis for NIST SRM 2781 can be found at *https://www-s.nist.gov/srmors/view_cert.cfm?srm=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. There may be a trend to higher As values, but the values remain well below the regulatory limits. The other eight elements varied by a factor of three 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-yr period (1999–2008) 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, 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, phosphorous, and bismuth have the highest concentrations in biosolids compared to soils (Yager and others, 2004a,b,c; 2009). Because of their high concentrations in biosolids compared to soils, these five elements would be the most likely "geochemical signature" to indicate that soils or stream sediments may have been impacted by biosolids.

Acknowledgments

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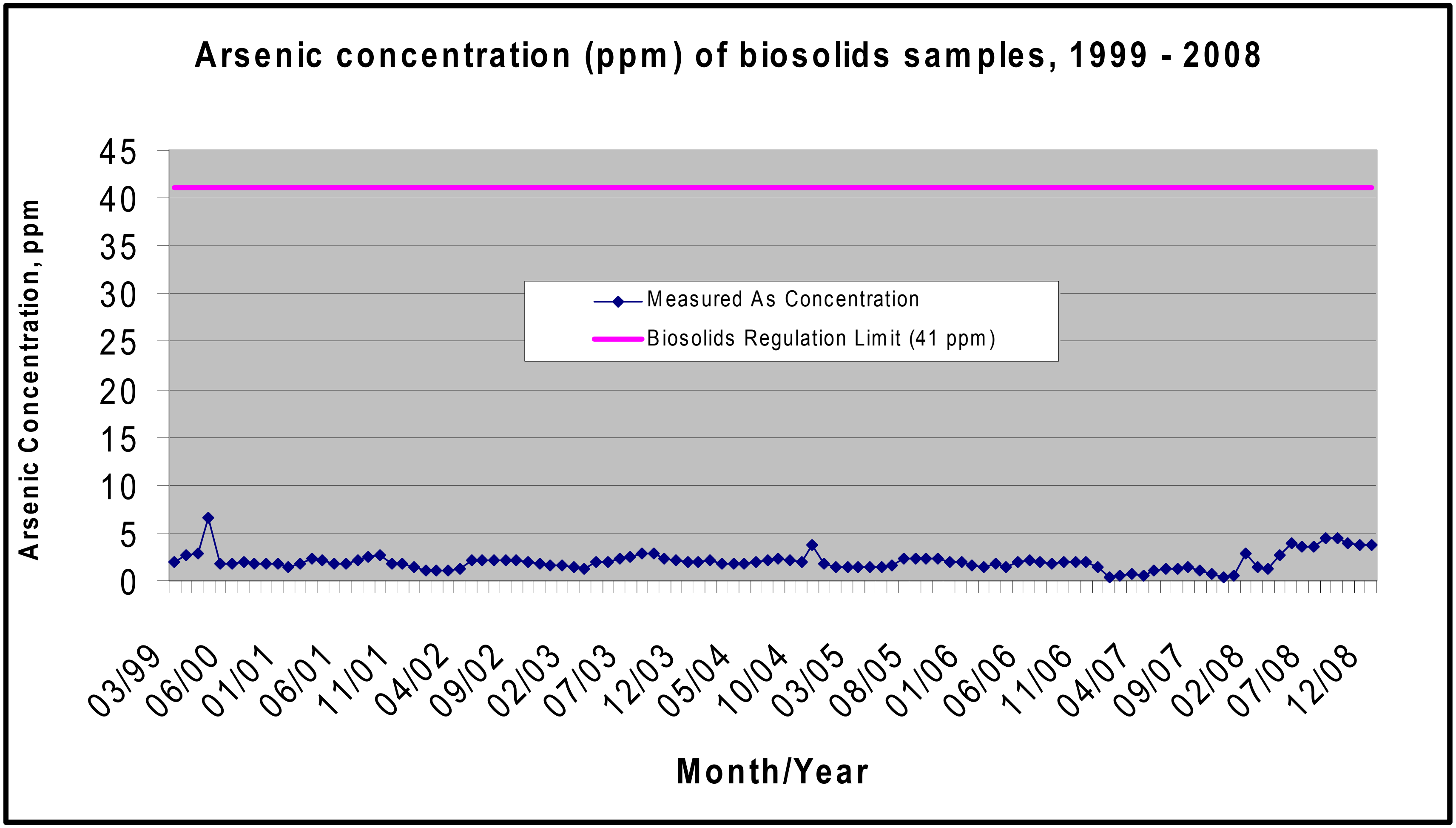

Figure 4. Arsenic concentrations of biosolids samples, 1999–2008.

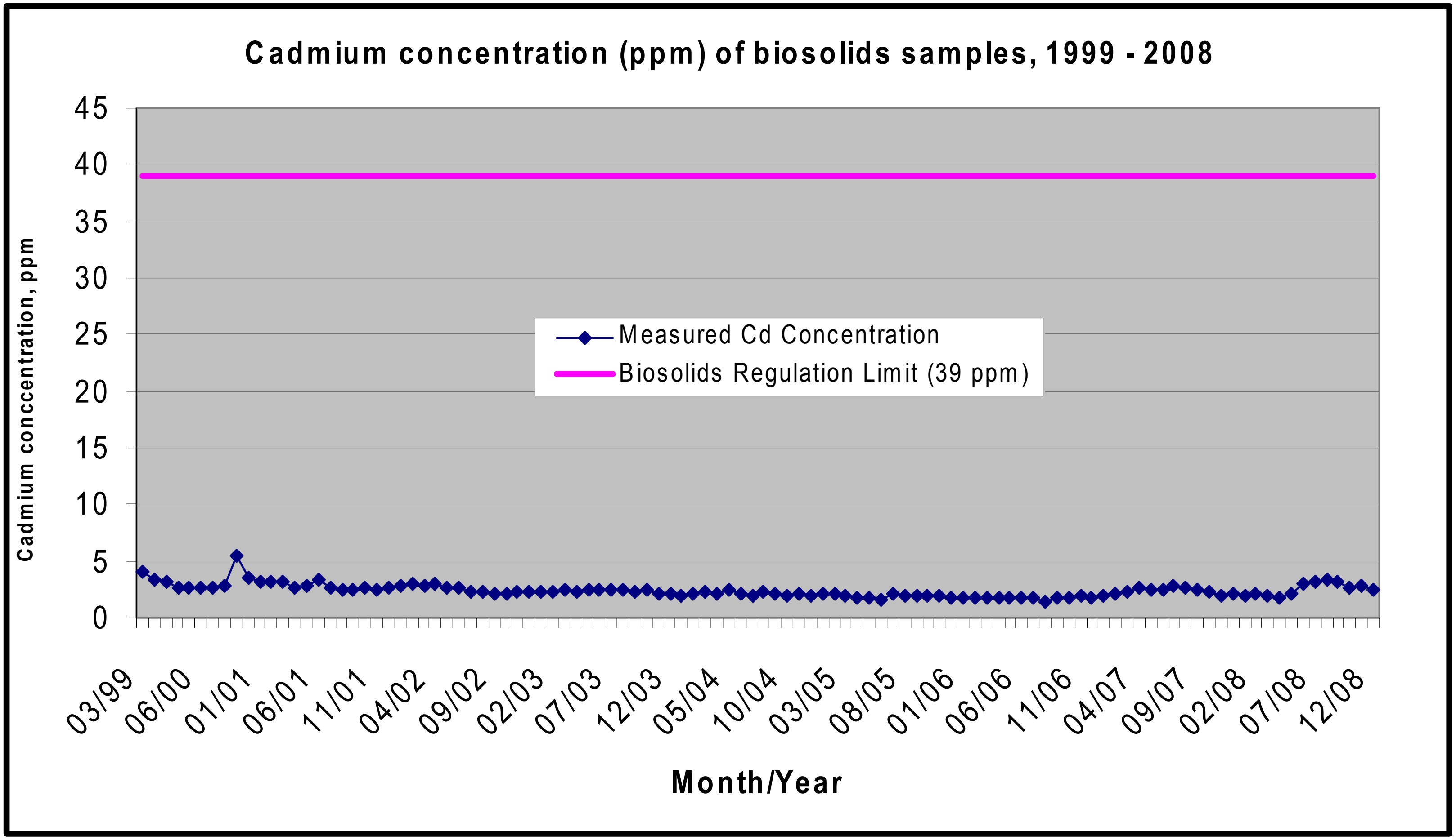

Figure 5. Cadmium concentrations of biosolids samples, 1999–2008.

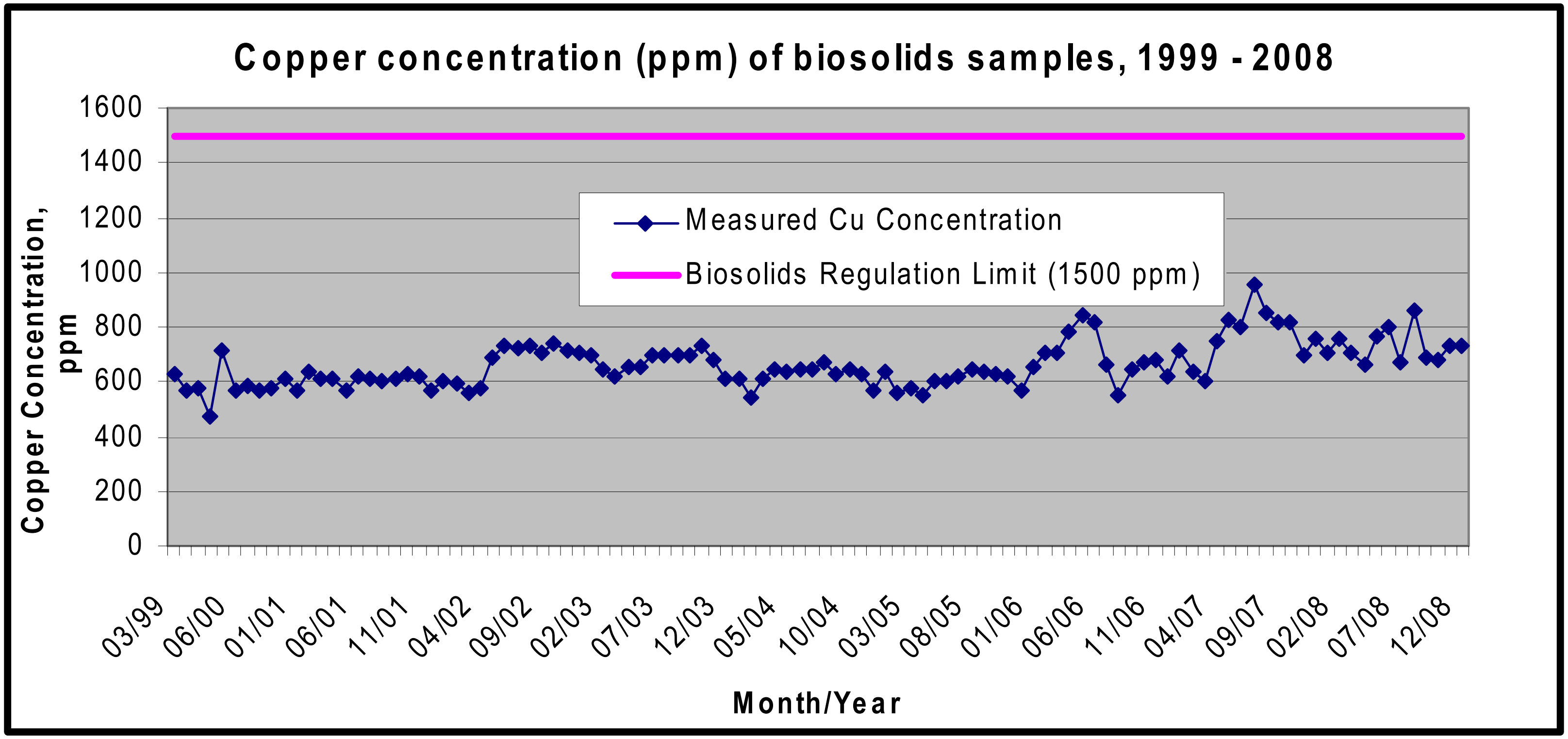

Figure 6. Copper concentrations of biosolids samples, 1999–2008.

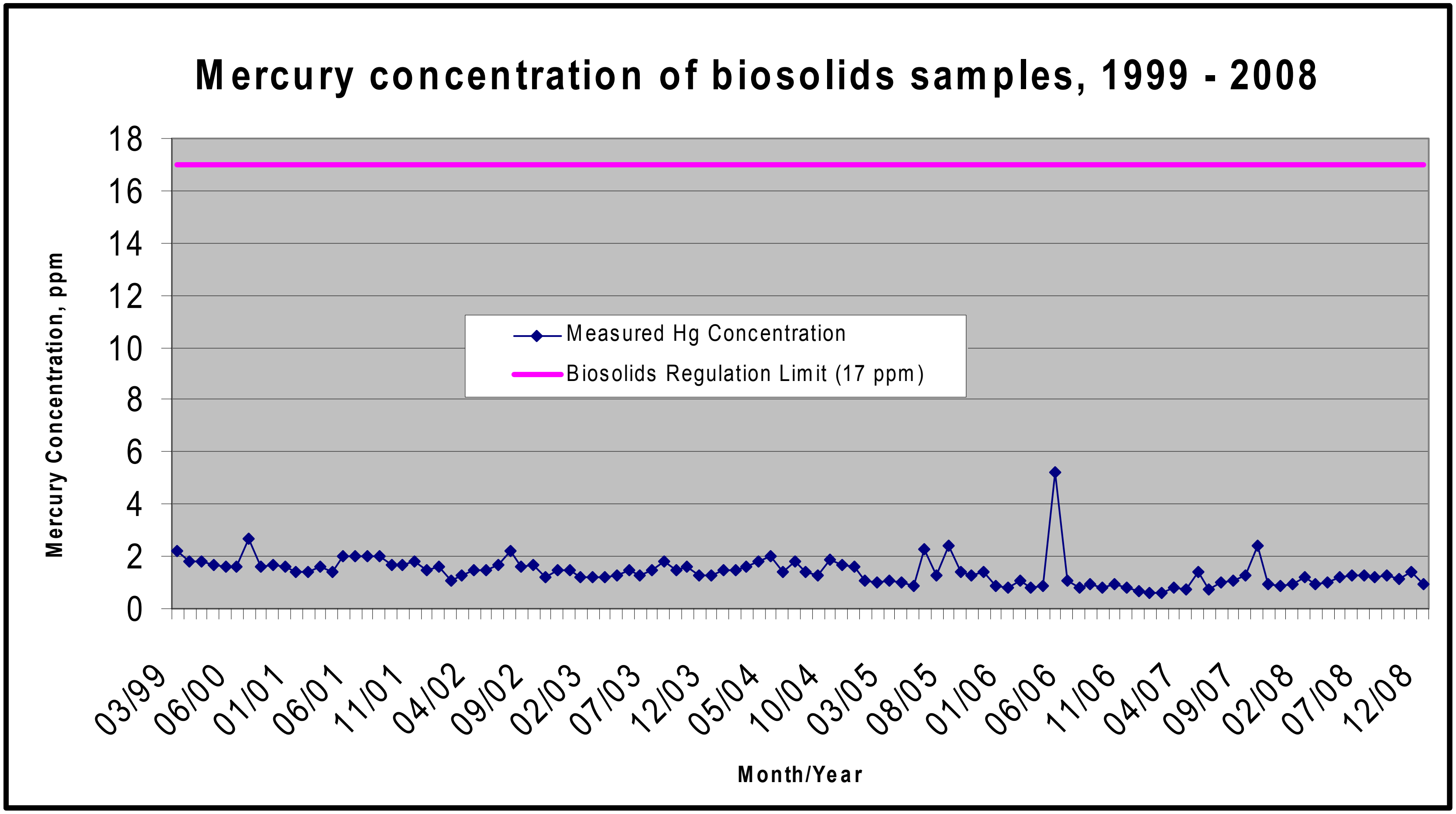

Figure 7. Mercury concentrations of biosolids samples, 1999–2008.

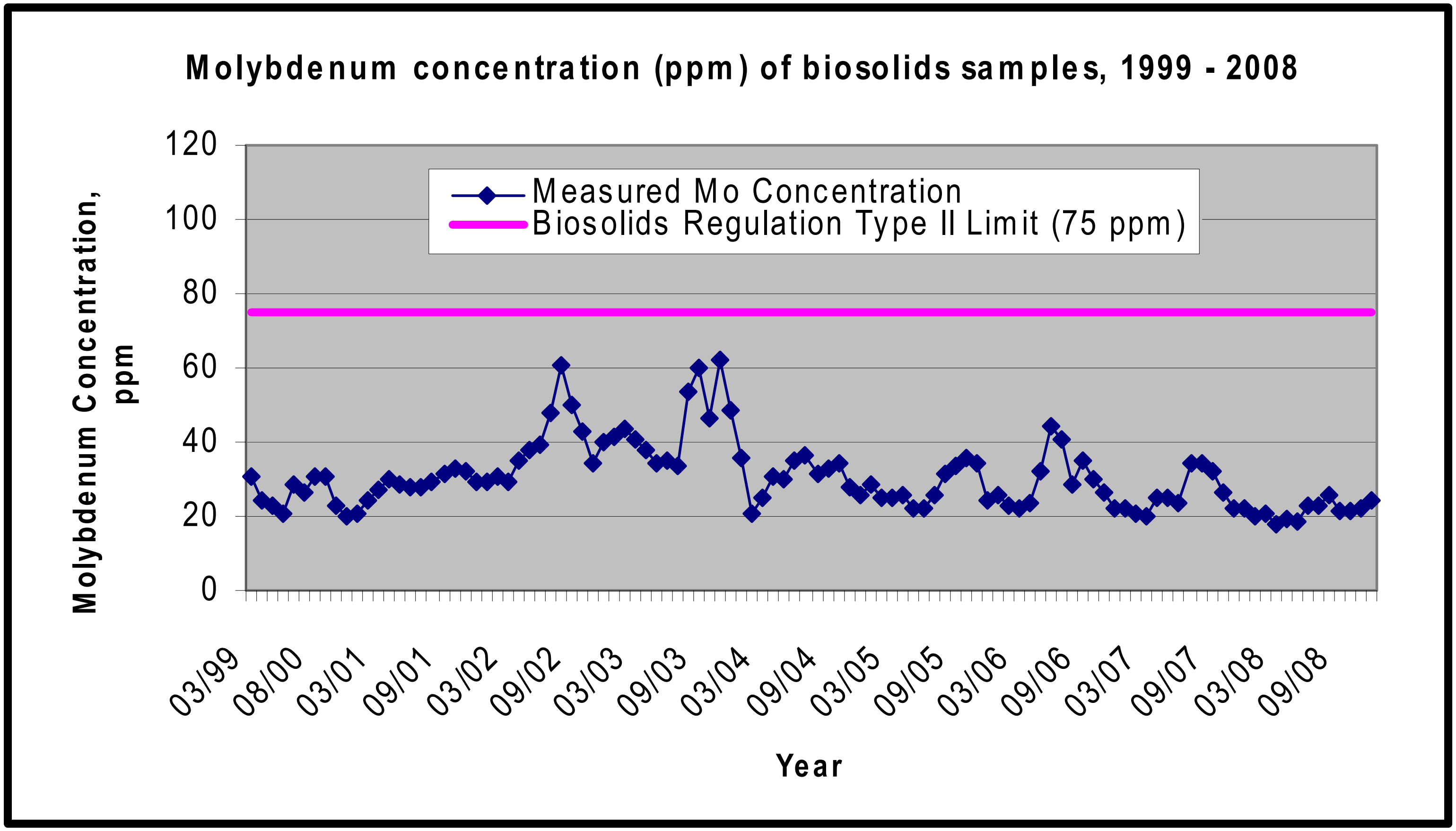

Figure 8. Molybdenum concentrations of biosolids samples, 1999–2008.

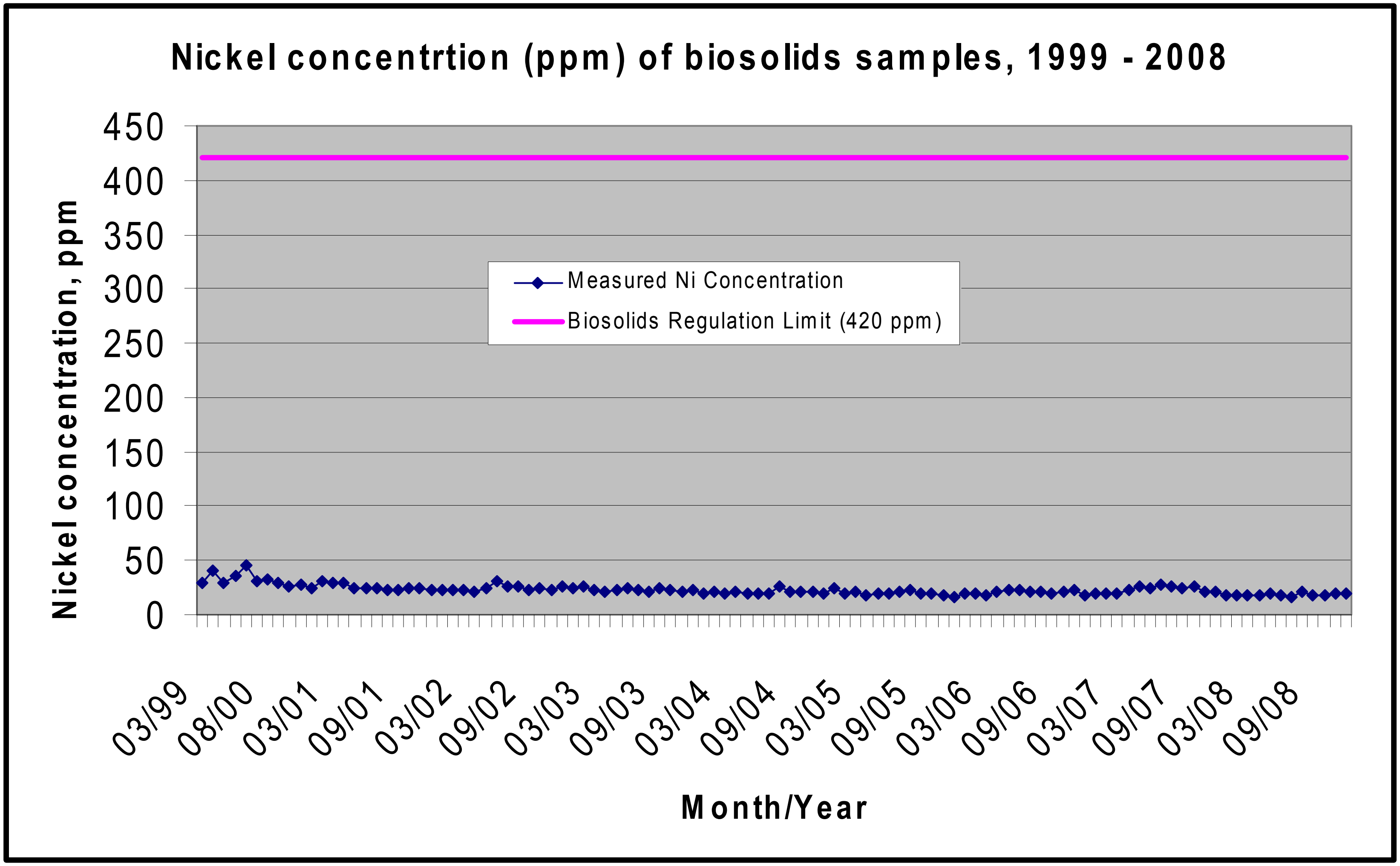

Figure 9. Nickel concentrations of biosolids samples, 1999–2008.

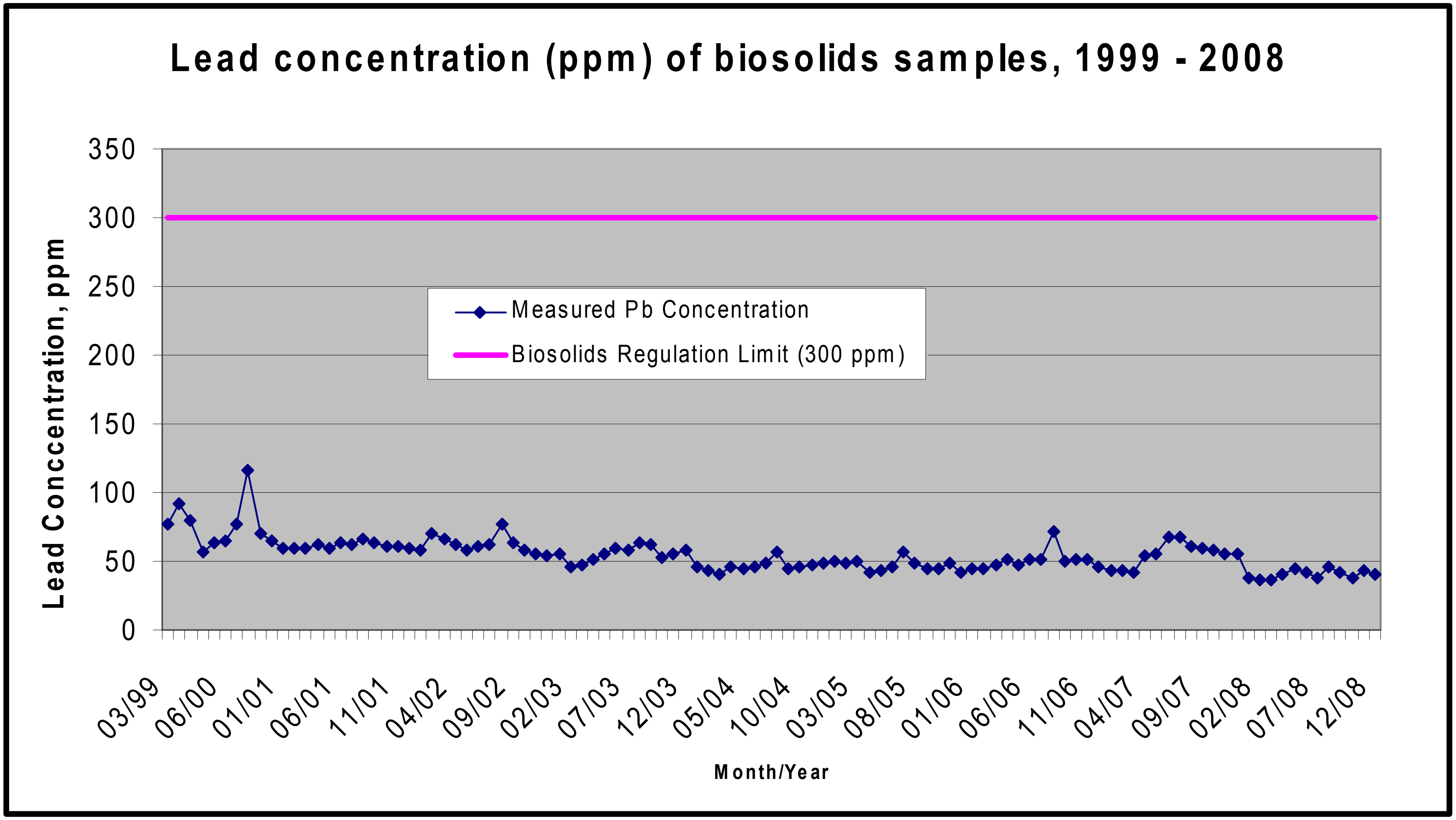

Figure 10. Lead concentrations of biosolids samples, 1999–2008.

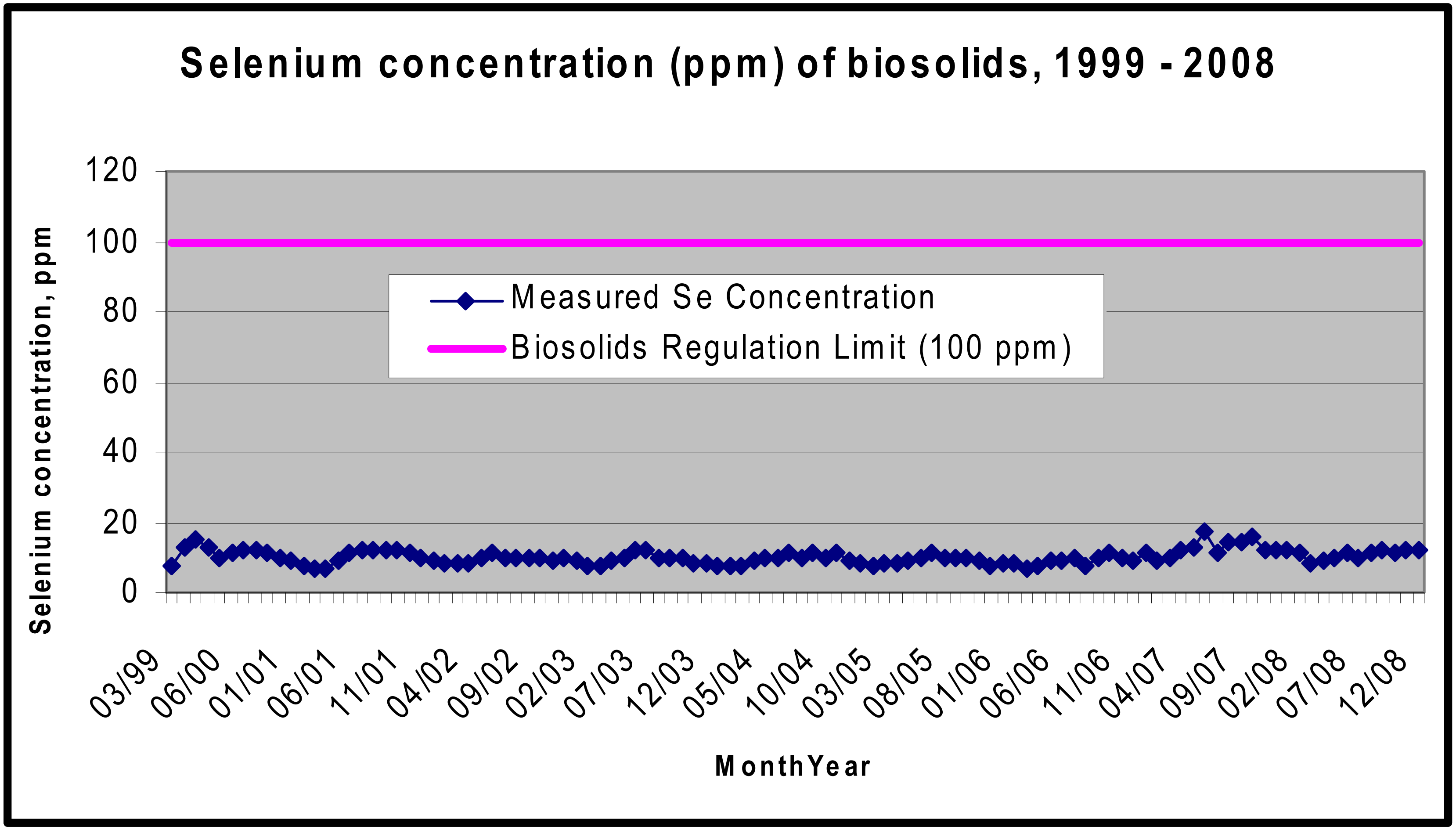

Figure 11. Selenium concentrations of biosolids samples, 1999–2008.

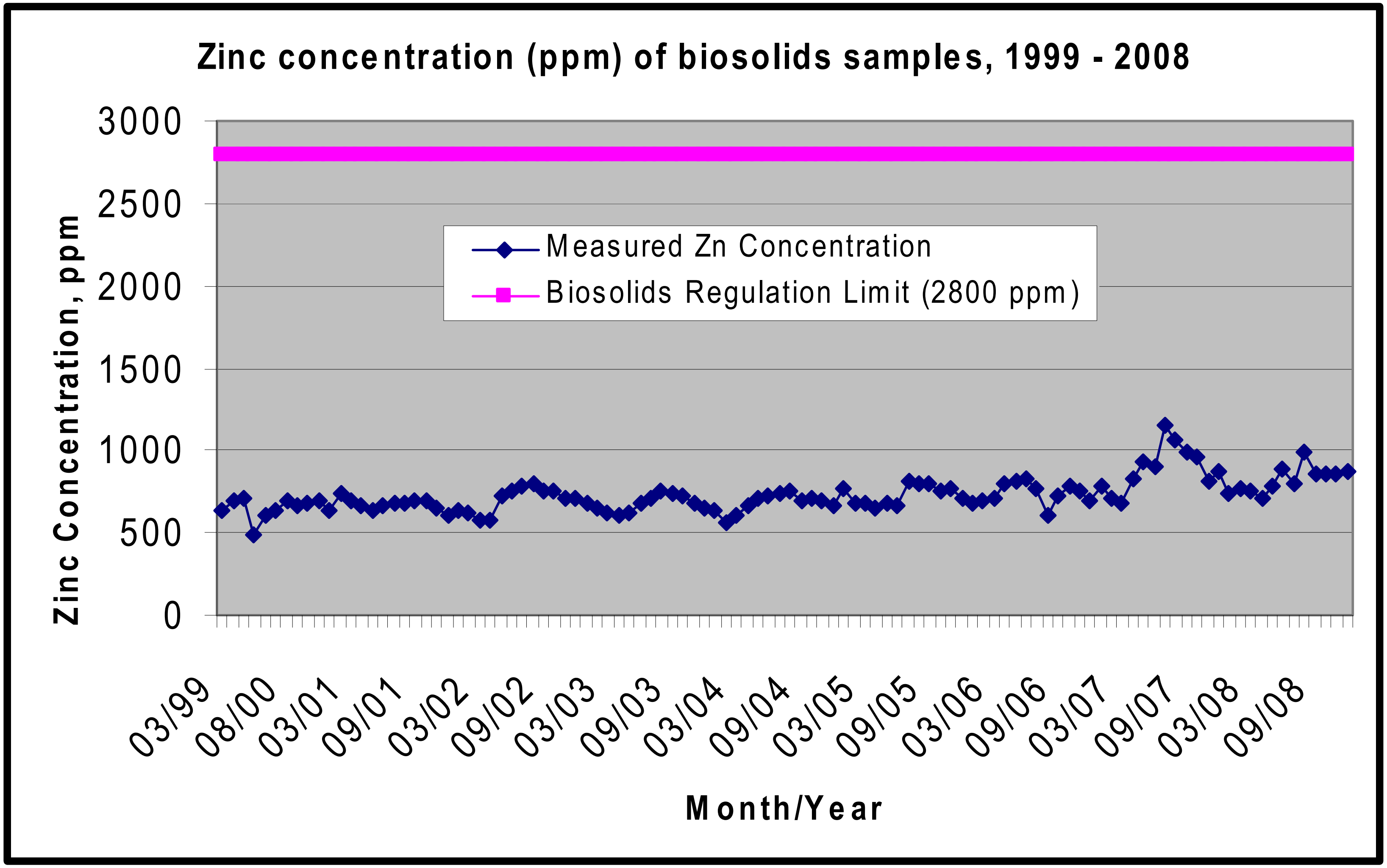

Figure 12. Zinc concentrations of biosolids samples, 1999–2008.

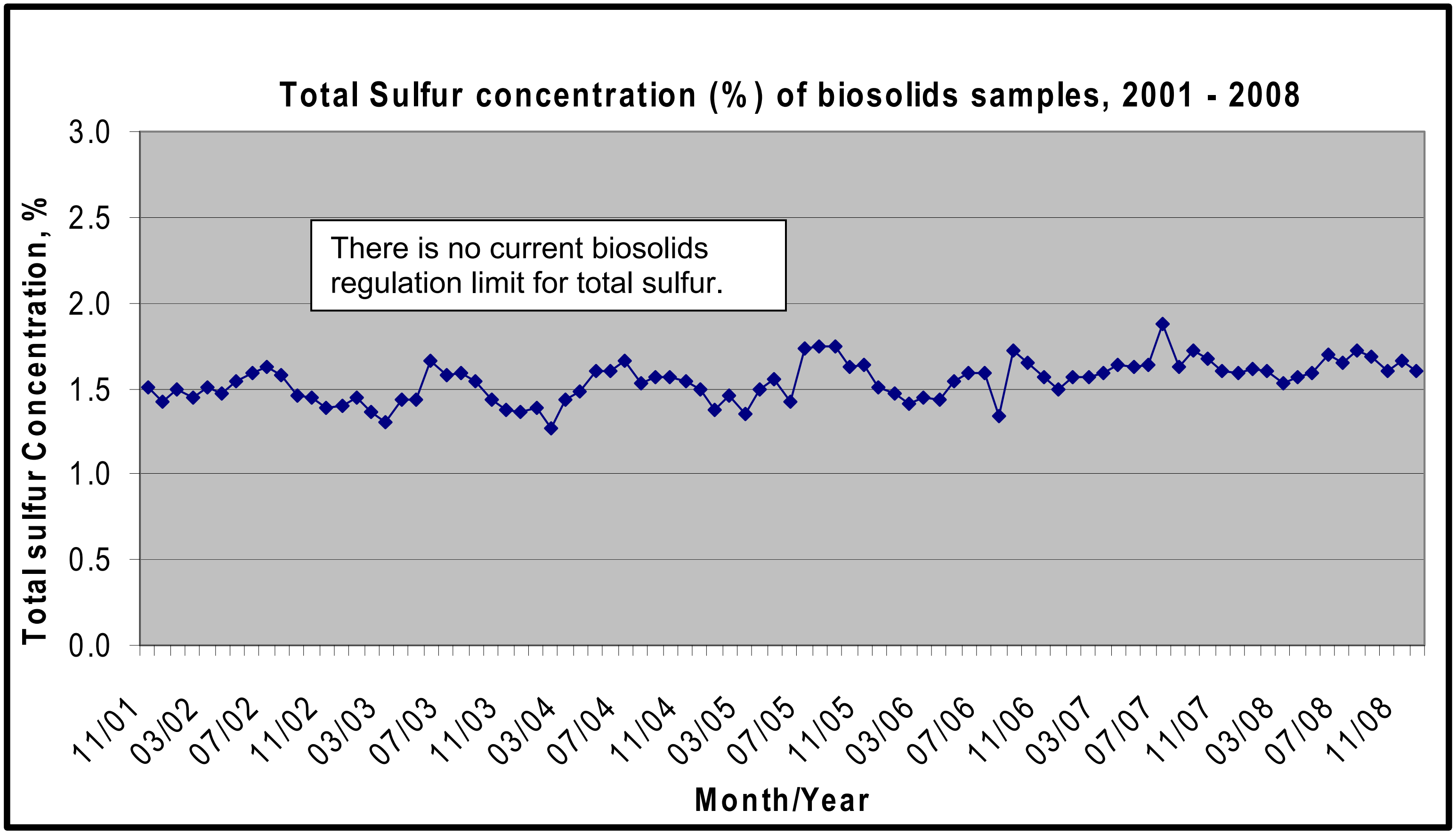

Figure 13. Total sulfur concentration of biosolids samples, 2001–2008.

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 2008 biosolids samples.

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 01/08	14.6	1.18	1.6	2.8	360	0.28	41.4	2.99	2.0
Bios 02/08	15.8	1.06	1.6	1.4	379	0.31	43.4	3.22	2.1
Bios 03/08	13.1	1.00	1.5	1.2	351	0.26	39.3	3.04	1.9
Bios 04/08	12.3	0.91	1.4	2.6	329	0.23	37.9	3.08	1.8
Bios 05/08	13.5	1.04	1.6	4.0	369	0.27	41.9	3.32	2.2
Bios 06/08	13.4	0.97	1.7	3.5	390	0.28	42.6	3.27	3.0
Bios 07/08	10.9	0.90	1.9	3.6	354	0.26	38.0	3.06	3.1
Bios 08/08	13.2	1.08	2.5	4.4	413	0.30	42.4	3.06	3.4
Bios 09/08	11.2	0.89	2.0	4.5	366	0.26	39.2	2.83	3.2
Bios 10/08	11.0	0.85	1.9	3.8	367	0.21	41.3	3.01	2.6
Bios 11/08	15.1	0.88	1.9	3.8	389	0.27	40.0	2.92	2.8
Bios 12/08	12.7	0.83	1.9	3.8	399	0.24	48.4	3.20	2.5
NIST 2781	37.2	1.00	7.4	7.3	684	0.47	30.7	3.50	12.2
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

* - ICP-MS determination after a total, four acid digestion

** - Cold Vapor - Atomic Absorption Spectrometry

- Hydride Generation - Atomic Absorption Spectrometry

- Combustion - IR Detection

Table 2. Analytical results for the 2008 biosolids samples.

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 01/08	33.8	3.3	38	0.38	706	1.63	3.9	0.96	0.28
Bios 02/08	19.1	3.5	37	0.38	755	1.61	4.5	1.18	0.28
Bios 03/08	17.3	3.2	33	0.39	706	1.81	6.4	0.97	0.26
Bios 04/08	16.0	3.3	37	0.35	666	1.88	6.6	1.02	0.26
Bios 05/08	24.8	3.2	34	0.42	767	1.73	5.4	1.18	0.30
Bios 06/08	19.8	3.1	33	0.43	800	1.84	4.8	1.25	0.26
Bios 07/08	15.6	2.9	31	0.47	670	1.84	4.2	1.30	0.23
Bios 08/08	24.2	3.5	36	0.66	861	2.15	4.2	1.22	0.31
Bios 09/08	19.3	3.1	32	0.49	689	1.77	3.3	1.30	0.25
Bios 10/08	22.4	3.0	31	0.40	679	1.62	3.5	1.16	0.22
Bios 11/08	20.7	3.2	31	0.47	728	1.67	3.8	1.40	0.26
Bios 12/08	22.3	3.1	32	0.42	733	1.58	3.4	0.95	0.25
NIST 2781	63.5	5.9	154	0.88	631	2.70	6.8	3.35	0.39
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 2008 biosolids samples.

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 01/08	21.6	2.8	0.416	257	19.8	0.130	44	18	2.54
Bios 02/08	14.0	2.1	0.421	242	20.8	0.117	48	19	2.53
Bios 03/08	11.7	1.9	0.365	244	18.0	0.104	88	17	2.45
Bios 04/08	12.2	0.6	0.346	256	19.3	0.108	82	18	2.38
Bios 05/08	18.0	1.4	0.520	290	18.8	0.129	60	19	2.50
Bios 06/08	14.8	0.7	0.366	299	22.6	0.107	57	19	2.37
Bios 07/08	12.6	0.5	0.303	293	22.8	0.092	49	17	2.05
Bios 08/08	19.3	1.7	0.318	431	26.0	0.110	43	21	2.24
Bios 09/08	14.2	0.7	0.271	474	21.2	0.095	42	18	1.91
Bios 10/08	15.1	< 0.3	0.270	329	21.1	0.091	72	18	1.92
Bios 11/08	15.0	0.3	0.283	273	21.8	0.101	61	19	2.00
Bios 12/08	16.2	< 0.3	0.306	237	24.5	0.095	49	19	2.01
NIST 2781	18.7	2.7	0.397	772	40.7	0.136	100	74	2.06
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 2008 biosolids samples.

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 01/08	38	8.2	1.61	2.9	1.3	12	267	1.5	0.20
Bios 02/08	37	8.2	1.60	6.8	1.2	11	254	1.4	0.21
Bios 03/08	36	7.9	1.53	6.3	1.5	8.6	244	1.5	0.22
Bios 04/08	41	7.7	1.57	5.8	1.4	8.8	241	1.4	0.23
Bios 05/08	45	9.8	1.59	6.8	1.3	10	261	1.7	0.21
Bios 06/08	42	9.1	1.70	7.8	1.3	11	269	1.7	0.22
Bios 07/08	38	8.7	1.65	6.7	1.2	10	237	1.5	0.19
Bios 08/08	46	12.4	1.72	6.0	1.7	11	260	2.2	0.25
Bios 09/08	41	9.5	1.68	3.4	1.3	12	235	1.9	0.19
Bios 10/08	38	8.1	1.60	3.8	1.2	11	242	1.6	0.20
Bios 11/08	43	9.6	1.66	3.2	1.3	12	256	1.8	0.20
Bios 12/08	41	9.1	1.60	3.0	1.1	12	253	2.4	0.20
NIST 2781	188	17.6	1.59	7.0	69	17	257	5.5	0.27
NIST 2781 Recommended/ Certified Value	202.1 +/- 6.5					16.0 +/- 1.6			0.32 +/- 0.03

Table 2. Analytical results for the 2008 biosolids samples.

Sample ID	ICP-MS Tl, ppm	ICP-MS U, ppm	ICP-MS V, ppm	ICP-MS Y, ppm	ICP-MS Zn, ppm
Bios 01/08	0.1	41.7	18	3.6	745
Bios 02/08	0.1	39.8	14	3.6	768
Bios 03/08	0.1	36.0	21	3.6	754
Bios 04/08	0.1	36.5	16	3.3	714
Bios 05/08	0.1	40.9	16	4.0	779
Bios 06/08	0.1	49.1	13	4.1	880
Bios 07/08	0.1	43.4	13	3.8	805
Bios 08/08	0.1	51.3	16	5.1	990
Bios 09/08	0.1	48.4	12	4.0	860
Bios 10/08	0.1	44.3	11	3.8	850
Bios 11/08	0.1	44.7	12	4.0	861
Bios 12/08	0.1	42.0	11	4.4	870
NIST 2781	0.3	39.2	78	30.9	1270
NIST 2781 Recommended/ Certified Value					1273 +/- 53

Table 3. Analytical results for selected samples of 2007 and 2008 biosolids samples for plutonium isotopes.

Sample Number	Pu^{238} Result	CSU Pu^{238}	SSMDC Pu^{238}	MDC Pu^{238}	$Pu^{239-240}$ Result	CSU $Pu^{239-240}$	SSMDC $Pu^{239-240}$	MDC $Pu^{239-240}$
Bios 06/07	-0.00269	0.0045	0.017	0.1	0.00177	0.0018	0.0065	0.1
Bios 06/08	-0.00341	0.0034	0.017	0.1	-0.00226	0.0034	0.015	0.1

Pu^{238} Result: Concentration of Pu^{238}, pico curies/g (pc/g)
CSU Pu^{238}: Combined standard uncertainty for Pu^{238}, pc/g
SSMDC Pu^{238}: Sample specific minimum detectable concentration for Pu^{238}, pc/g
MDC Pu^{238}: Minimum detectable concentration for Pu^{238}, pc/g
$Pu^{239-240}$ Result: Concentration of $Pu^{239-240}$, pc/g
CSU $Pu^{239-240}$: Combined standard uncertainty for $Pu^{239-240}$, pc/g
SSMDC $Pu^{239-240}$: Sample specific minimum detectable concentration for $Pu^{239-240}$, pc/g
MDC $Pu^{239-240}$: Minimum detectable concentration for $Pu^{239-240}$, pc/g