



Analytical Results for Municipal Biosolids Samples from a Monitoring Program Near Deer Trail, Colorado (U.S.A.), 2010

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. 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 was recently extended through the end of 2010 and is now completed. 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 runoff effects. This report summarizes analytical results for the biosolids samples collected at the Metro District wastewater treatment plant in Denver and analyzed for 2010.

In general, the objective of each component of the study was to determine whether concentrations of nine trace elements (“priority analytes”) (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 (background).

Previous analytical results indicate that the elemental composition of biosolids from the Denver plant was consistent during 1999–2009, and this consistency continues with the samples for 2010. Total concentrations of regulated trace elements remain consistently lower than the regulatory limits for the entire monitoring period. Concentrations of none of the priority analytes appear to have increased during the 12 years of this study.

Introduction

Since 1993, the Metro Wastewater Reclamation District of Denver (Metro District) has been applying biosolids from the Denver metropolitan area to its 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 produces the Metro District biosolids can be found at <http://www.metrowastewater.com> (last visited December 29, 2010). The biosolids-application areas, dates of application, and application rates provided by the Metro District for its 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> (last visited December 29, 2010).

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; data for the 2008 biosolids samples are presented in Crock and others (2009), and data for 2009 biosolids samples are presented in Crock and others (2010). More information about the other monitoring components is presented elsewhere in the literature (for example, Yager and others, 2004a,b,c,d, 2009). Priority parameters for biosolids identified by the stakeholders and also regulated by the State of 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. Total sulfur was added as an element of concern in the fall of 2001 and continues to be determined and monitored.

Data from previous reports (Crock and others, 2008a,b, 2009, 2010) and this report were used to compile an inorganic-chemical biosolids signature that can be contrasted with the “natural” 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.

In 1999, the Metro District property, known as the METROGRO Farm, encompassed about 81 mi² (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.”

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 2010 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 an agricultural field 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 2009. 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 soils, crops, bedrock aquifers, 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 2010 analytical data for biosolids only. Analytical results for biosolids collected between 1999 and 2009 can be found in Crock and others (2008a,b, 2009, 2010). 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 laboratory.

Biosolids are moist (usually ranging 75–85 percent moisture) and have a firm, pudding-like texture. The regulations require 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) (U.S. Environmental Protection Agency, 1993; Colorado Department of Public Health and Environment, 1998). 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 (all last visited December 29, 2010):

<http://www.cdph.state.co.us/wq/PermitsUnit/biosolids/index.html>,

<http://www.epa.gov/owm/mtb/biosolids/503pe/index.htm>, and

<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. A random subsample from the 2009 and 2010 samples were analyzed during 2010 for plutonium isotopes. Consult table 1 in this report 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 consisting 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 Crustal Geophysics and Geochemistry Science Center, Denver, Colo. The biosolids material was air dried using forced air and an infrared lamp (surface temperature $\approx 40^{\circ}\text{C}$) 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, the National Institute of Standards and Technology (NIST) standard reference material (SRM) 2781 for domestic sludge was analyzed along with the 2010 biosolids samples.

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

Parameter	Method	Reference
Arsenic	HG-AAS ¹	Hageman and Welsch (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)

¹Hydride Generation–Atomic Absorption Spectrometry

²Inductively Coupled Plasma–Mass Spectrometry

³Continuous Flow–Cold Vapor–Atomic Fluorescence Spectrometry

⁴Automated combustion in oxygen, measured by a solid-state infrared detector

Discussion and Results

Biosolids exceeding the regulatory 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 2010 data will also augment the chemical baseline that has been established earlier by Crock and others (2008a,b, 2009, 2010) against which any future change in the concentration of constituents analyzed for in this study may be identified, measured, and compared. This data set will also build on the

“geochemical signature” for biosolids that will potentially enable scientists to recognize when biosolids have impacted soils or stream sediments.

All data for the 1999–2009 biosolids samples are presented in Crock and others (2008a,b, 2009, 2010) and are presented in figures 4–13 supplemented with the 2010 data presented in this report. The concentrations of all nine trace elements show little variation when plotted throughout the study (1999–2010) and below the Grade 1 biosolids requirements. Analytical results for reference material NIST SRM 2781 results are also presented in table 2 in this report. Table 3 presents the analytical results for the biosolids samples analyzed for plutonium isotopes. The certificate of analysis for NIST SRM 2781 can be found at

https://www-s.nist.gov/srmors/view_cert.cfm?srm=2781 (last visited December 29, 2010).

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. 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.)

Table 2. Analytical results for year 2010 biosolids samples.

Sample number	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	ICP-MS Ce, ppm	ICP-MS Co, ppm
Bios 01/10	7.6	0.98	1.5	1.40	337	0.20	40.9	2.93	1.8	19.2	2.8
Bios 02A/10	7.8	1.00	1.6	1.36	339	0.23	42.6	2.89	1.7	20.4	3.0
Bios 02B/10	7.9	1.03	1.6	1.38	343	0.22	41.4	3.02	1.9	21.3	3.1
Bios 02C/10	7.3	0.96	1.4	1.35	315	0.21	40.3	2.88	1.6	19.7	3.1
Bios 02/10 average	7.7	1.00	1.5	1.36	332	0.22	41.4	2.93	1.7	20.5	3.1
Bios 03/10	7.7	0.95	1.6	1.35	348	0.24	42.3	3.05	1.9	18.1	3.2
Bios 04/10	7.8	0.99	1.5	1.43	350	0.26	39.7	3.04	1.8	24.0	3.3
Bios 05/10	8.3	1.12	1.9	1.44	357	0.20	44.0	3.23	1.9	21.0	3.3
Bios 06A/10	8.0	1.02	2.0	1.57	349	0.20	41.6	3.15	1.8	17.0	3.0
Bios 06B/10	8.2	1.01	2.0	1.42	345	0.19	41.4	3.13	1.9	17.1	3.1
Bios 06C/10	8.3	1.02	1.8	1.48	357	0.23	41.1	3.18	1.8	19.0	3.0
Bios 06/10 average	8.2	1.02	1.9	1.49	350	0.21	41.4	3.15	1.8	17.7	3.0
Bios 07/10	7.7	1.07	2.2	1.85	356	0.26	42.6	3.11	1.9	17.5	3.1
Bios 08/10	8.0	1.14	2.1	1.67	378	0.25	44.2	3.20	1.9	25.7	3.2
Bios 09/10	8.3	0.99	1.9	1.59	366	0.27	46.5	3.39	1.7	21.2	3.0
Bios 10/10	11.0	1.54	2.8	1.69	538	0.36	62.5	4.45	2.7	33.4	4.4
Bios 11/10	9.1	1.05	2.0	1.60	385	0.21	46.7	3.39	2.2	20.8	3.3
Bios 12/10	9.1	1.02	1.8	1.27	373	0.26	48.1	3.25	2.4	20.5	3.2
NIST 2781	26.5	1.35	7.6	7.44	623	0.50	89.5	3.79	11.7	74.4	6.0
NIST 2781											
Recommended or 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 Fluorescence Spectrometry

- Hydride Generation-Atomic Absorption Spectrometry

- Combustion-IR Detection

ppm, parts per million or mg Kg⁻¹

%, percent

Table 2. Analytical results for year 2010 biosolids samples.—Continued

Sample number	ICP-MS Cr, ppm	ICP-MS Cs, ppm	ICP-MS Cu, ppm	ICP-MS Fe, %	ICP-MS Ga, ppm	CV-AFS** Hg, ppm	ICP-MS K, %	ICP-MS La, ppm	ICP-MS Li, ppm	ICP-MS Mg, %	ICP-MS Mn, ppm	ICP-MS Mo, ppm
Bios 01/10	26.7	0.36	594	1.57	2.7	1.05	0.239	12.1	3.4	0.311	173	14.9
Bios 02A/10	27.7	0.38	592	1.66	2.8	1.06	0.269	13.3	3.2	0.346	217	14.7
Bios 02B/10	28.4	0.38	601	1.71	2.8	1.22	0.279	13.7	3.1	0.356	221	14.9
Bios 02C/10	26.3	0.35	578	1.61	2.7	2.28	0.262	12.9	2.9	0.328	210	14.5
Bios 02/10 average	27.5	0.37	590	1.66	2.8	1.52	0.270	13.3	3.1	0.343	216	14.7
Bios 03/10	27.5	0.38	607	1.59	2.7	0.92	0.298	12.6	2.9	0.344	221	13.2
Bios 04/10	26.6	0.42	614	1.51	2.9	1.26	0.285	16.0	3.0	0.327	264	11.9
Bios 05/10	28.8	0.53	684	1.70	3.2	1.30	0.313	15.6	3.7	0.348	236	14.6
Bios 06A/10	29.2	0.45	679	1.87	2.9	1.10	0.257	14.4	3.2	0.324	222	13.6
Bios 06B/10	29.5	0.46	682	1.84	3.0	1.65	0.257	14.7	3.1	0.321	220	13.6
Bios 06C/10	30.5	0.45	690	1.89	2.9	1.37	0.260	15.5	2.9	0.319	221	13.6
Bios 06/10 average	29.7	0.45	684	1.87	2.9	1.37	0.258	14.9	3.1	0.321	221	13.6
Bios 07/10	27.8	0.52	671	1.99	3.1	1.18	0.286	15.0	3.0	0.369	231	17.7
Bios 08/10	30.4	0.54	714	2.08	3.3	1.31	0.334	19.3	3.1	0.322	361	18.6
Bios 09/10	30.2	0.43	665	1.99	2.9	1.28	0.257	19.1	2.7	0.306	413	21.2
Bios 10/10	45.8	0.72	933	2.80	4.1	1.64	0.375	25.8	2.5	0.485	559	33.8
Bios 11/10	31.6	0.42	711	1.88	2.9	1.62	0.262	16.7	3.0	0.308	359	26.5
Bios 12/10	29.3	0.39	712	1.77	2.9	1.23	0.263	15.6	2.5	0.298	244	22.9
NIST 2781	142.0	0.92	584	2.70	7.5	3.47	0.447	20.4	5.8	0.496	794	41.8
NIST 2781 Recommended or certified value	202 ± 9		627.4 ± 13.5	2.8 ± 0.1		3.64 ± 0.25	0.49 ± 0.03			0.59 ± 0.04		46.7 ± 3.2

Table 2. Analytical results for year 2010 biosolids samples.—Continued

Sample number	ICP-MS Na, %	ICP-MS Nb, ppm	ICP-MS Ni, ppm	ICP-MS P, %	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
Bios 01/10	0.124	2	17.6	2.30	31.6	7.6	1.55	3.7	0.5	9.7	247
Bios 02A/10	0.131	2	18.4	2.38	37.1	8.3	1.62	3.4	0.6	9.8	246
Bios 02B/10	0.138	2	18.8	2.45	35.5	8.7	1.63	2.9	0.6	9.6	259
Bios 02C/10	0.123	2	17.3	2.30	35.1	8.2	1.57	2.8	0.6	9.4	242
Bios 02/10 average	0.131	2	18.2	2.38	35.9	8.4	1.61	3.0	0.6	9.6	249
Bios 03/10	0.142	2	18.1	2.45	35.7	8.4	1.51	2.8	0.6	9.4	256
Bios 04/10	0.132	2	18.0	2.31	35.1	9.3	1.52	2.5	0.7	9.8	258
Bios 05/10	0.148	2	18.7	2.31	42.6	10.9	1.63	2.7	0.9	11.3	273
Bios 06A/10	0.132	2	17.4	2.34	38.3	9.3	1.71	2.4	0.7	13.6	285
Bios 06B/10	0.132	2	17.6	2.31	39.1	9.3	1.72	2.6	0.7	13.7	282
Bios 06C/10	0.131	2	18.3	2.35	37.4	9.2	1.71	2.4	0.7	13.5	279
Bios 06/10 average	0.132	2	17.8	2.33	38.3	9.3	1.71	2.5	0.7	13.6	282
Bios 07/10	0.137	2	18.5	2.26	43.0	10.5	1.88	2.7	0.9	15.4	276
Bios 08/10	0.151	2	19.0	2.29	49.0	12.3	1.89	2.5	0.9	14.0	271
Bios 09/10	0.122	2	20.2	2.17	48.3	9.3	1.78	2.7	0.7	10.9	256
Bios 10/10	0.175	3	28.1	2.90	63.0	13.3	1.92	4.4	1.4	11.2	320
Bios 11/10	0.121	2	21.8	2.37	43.4	9.4	1.76	3.7	0.7	9.4	260
Bios 12/10	0.127	2	19.1	2.45	46.5	8.4	1.77	2.8	0.6	10.4	272
NIST 2781	0.181	54	74.9	2.41	185	18.7	1.59	7.0	64.1	15.9	257
NIST 2781 Recommended or certified value	0.21 ± 0.02		80.2 ± 2.3	2.42 ± 0.09	202.1 ± 6.5					16.0 ± 1.6	

Table 2. Analytical results for year 2010 biosolids samples.—Continued

Sample number	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 01/10	0.97	0.145	0.10	38.7	9.3	2.3	712
Bios 02A/10	1.07	0.149	0.10	37.3	9.5	2.4	713
Bios 02B/10	1.50	0.145	0.29	38.5	9.7	2.6	734
Bios 02C/10	1.16	0.134	0.14	36.6	9.3	2.3	694
Bios 02/10 average	1.24	0.143	0.18	37.5	9.5	2.4	714
Bios 03/10	1.41	0.142	0.11	40.4	9.6	2.4	687
Bios 04/10	1.26	0.143	0.11	44.1	10.7	2.7	669
Bios 05/10	1.47	0.144	0.14	58.1	13.0	3.0	749
Bios 06A/10	1.17	0.150	0.12	59.6	11.6	2.8	776
Bios 06B/10	1.27	0.145	0.12	60.0	11.3	2.9	772
Bios 06C/10	1.69	0.159	0.11	61.1	11.8	3.0	777
Bios 06/10 average	1.38	0.151	0.12	60.2	11.6	2.9	775
Bios 07/10	1.47	0.157	0.12	56.5	13.1	3.1	810
Bios 08/10	1.80	0.140	0.34	52.0	12.9	3.3	848
Bios 09/10	1.36	0.149	0.16	41.9	11.4	2.8	848
Bios 10/10	1.90	0.200	0.42	51.8	16.8	4.0	1090
Bios 11/10	1.38	0.146	0.12	38.1	10.8	3.1	846
Bios 12/10	1.25	0.141	0.12	38.7	10.3	2.6	821
NIST 2781	5.59	0.201	0.26	36.6	85.1	23.3	1110
NIST 2781 Recommended or certified value		0.32 ± 0.03					1,273 ± 53

Table 3. Analytical results for selected samples of 2009 and 2010 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 06/09	0.0144	0.022	0.068	0.10	-0.00356	0.0070	0.037	0.10
Bios 01/10	0.00460	0.0090	0.045	0.10	0.0137	0.0090	0.028	0.10
NIST 2781	-0.00816	0.017	0.060	0.10	0.0268	0.012	0.034	0.10

Pu²³⁸ result: Concentration of Pu²³⁸, pico curies/g (pc/g)

CSU Pu²³⁸: Combined standard uncertainty for Pu²³⁸, pc/g

SSMDC Pu²³⁸: Sample specific minimum detectable concentration for Pu²³⁸, pc/g

MDC Pu²³⁸: Minimum detectable concentration for Pu²³⁸, pc/g

Pu²³⁹⁻²⁴⁰ result: Concentration of Pu²³⁹⁻²⁴⁰, pc/g

CSU Pu²³⁹⁻²⁴⁰: Combined standard uncertainty for Pu²³⁹⁻²⁴⁰, pc/g

SSMDC Pu²³⁹⁻²⁴⁰: Sample specific minimum detectable concentration for Pu²³⁹⁻²⁴⁰, pc/g

MDC Pu²³⁹⁻²⁴⁰: Minimum detectable concentration for Pu²³⁹⁻²⁴⁰, pc/g

Note all values are below minimum detectable concentrations for all isotopes.

Arsenic concentration (ppm) of biosolids samples, 1999 - 2010

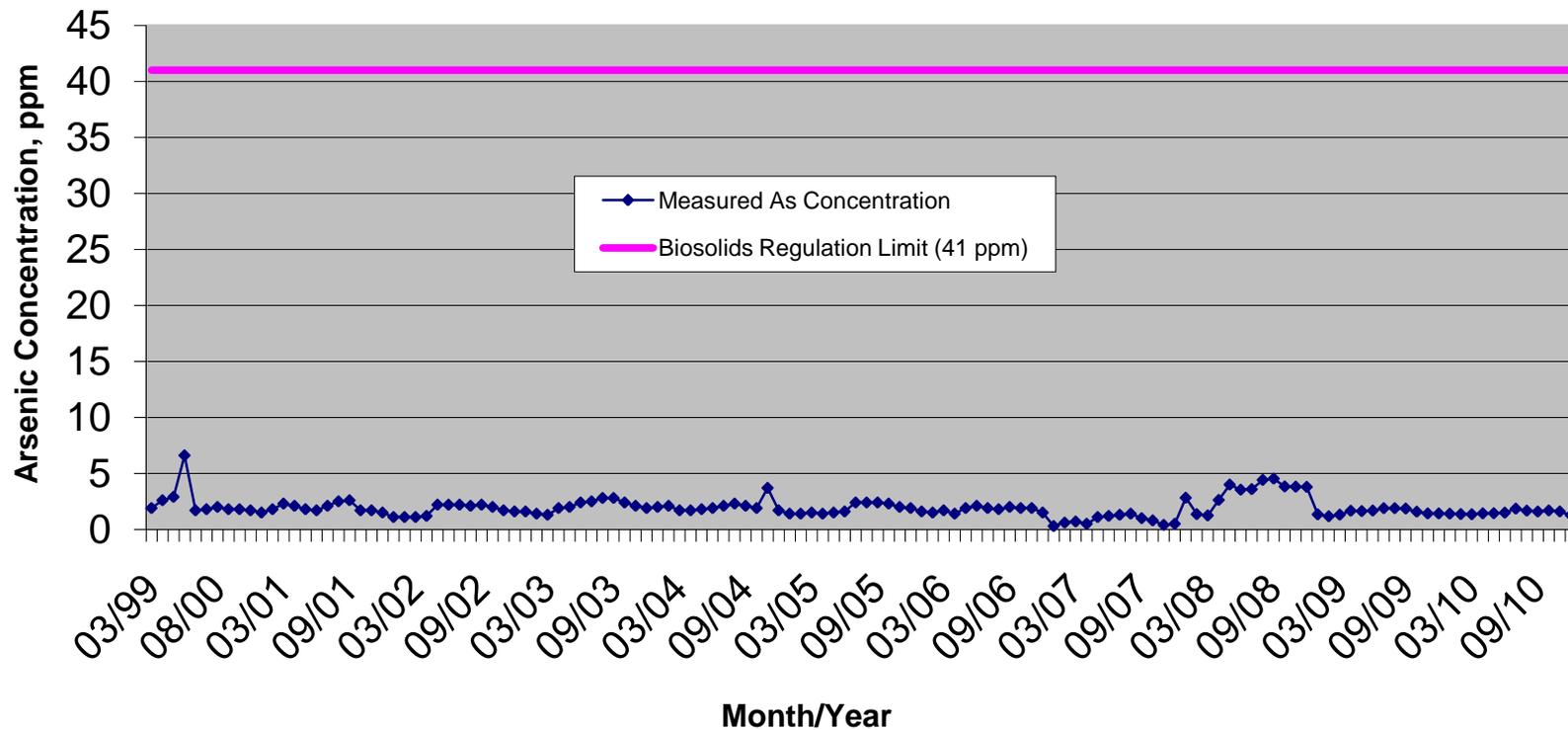


Figure 4. Arsenic concentrations of biosolids samples, 1999–2010.

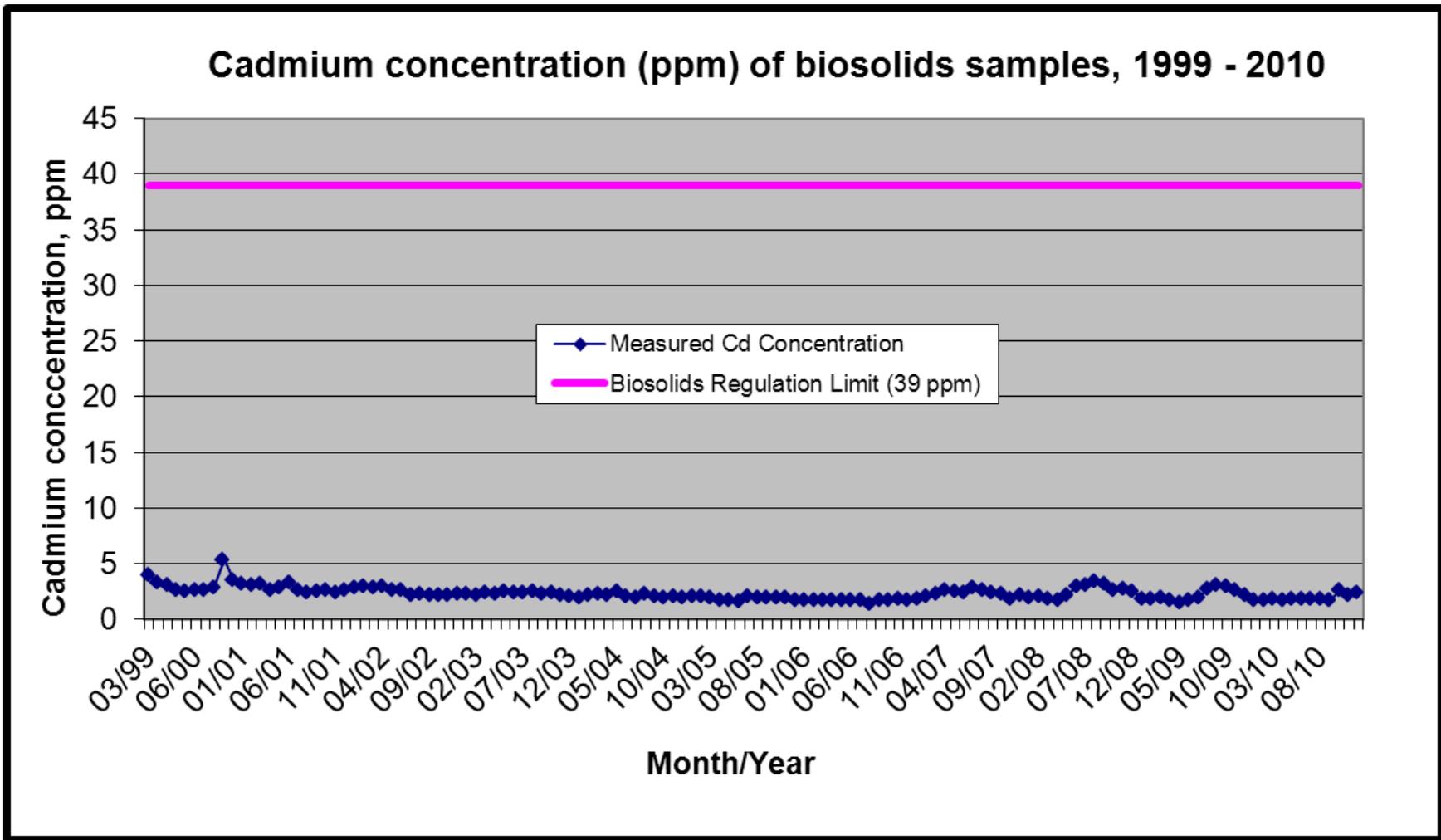


Figure 5. Cadmium concentrations of biosolids samples, 1999–2010.

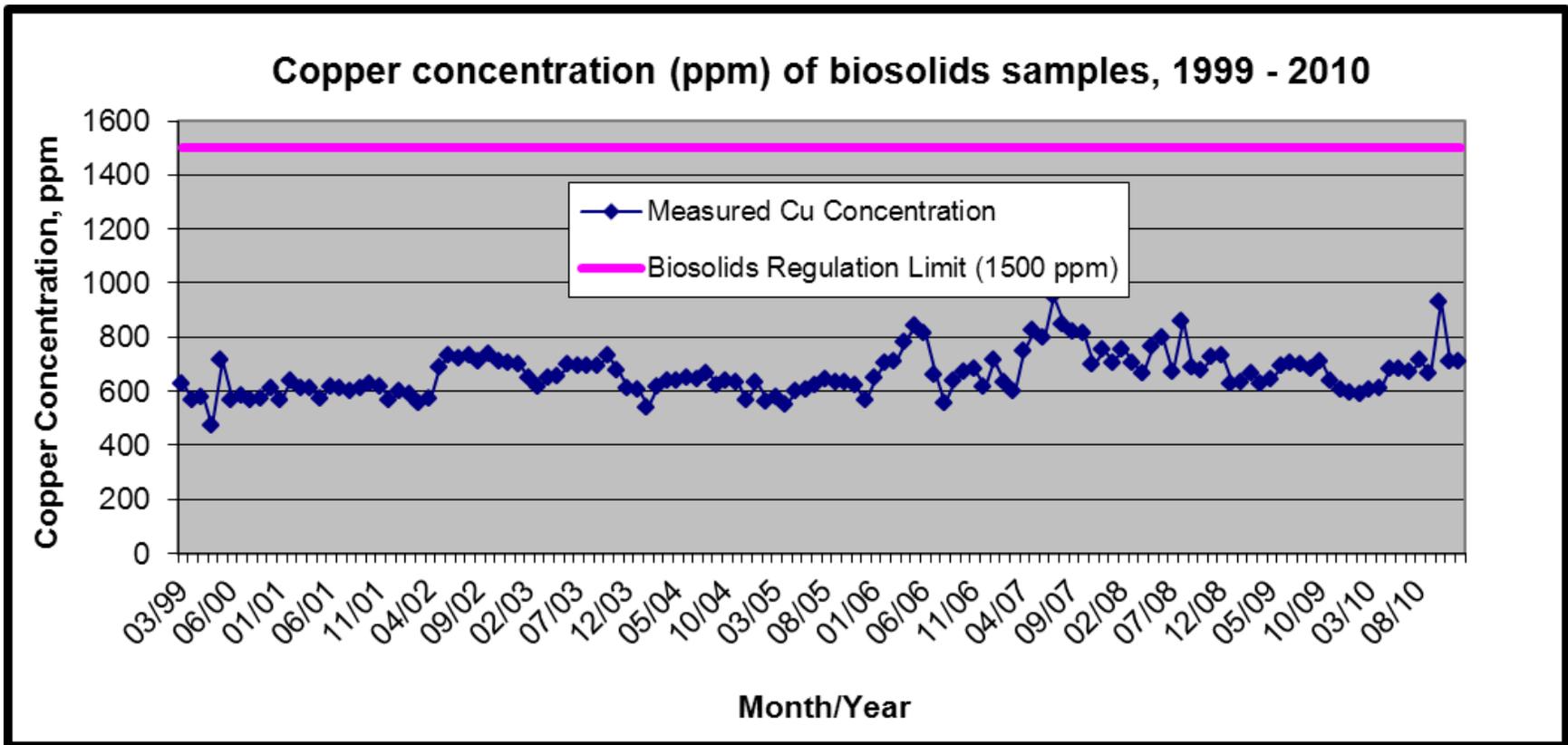


Figure 6. Copper concentrations of biosolids samples, 1999–2010.

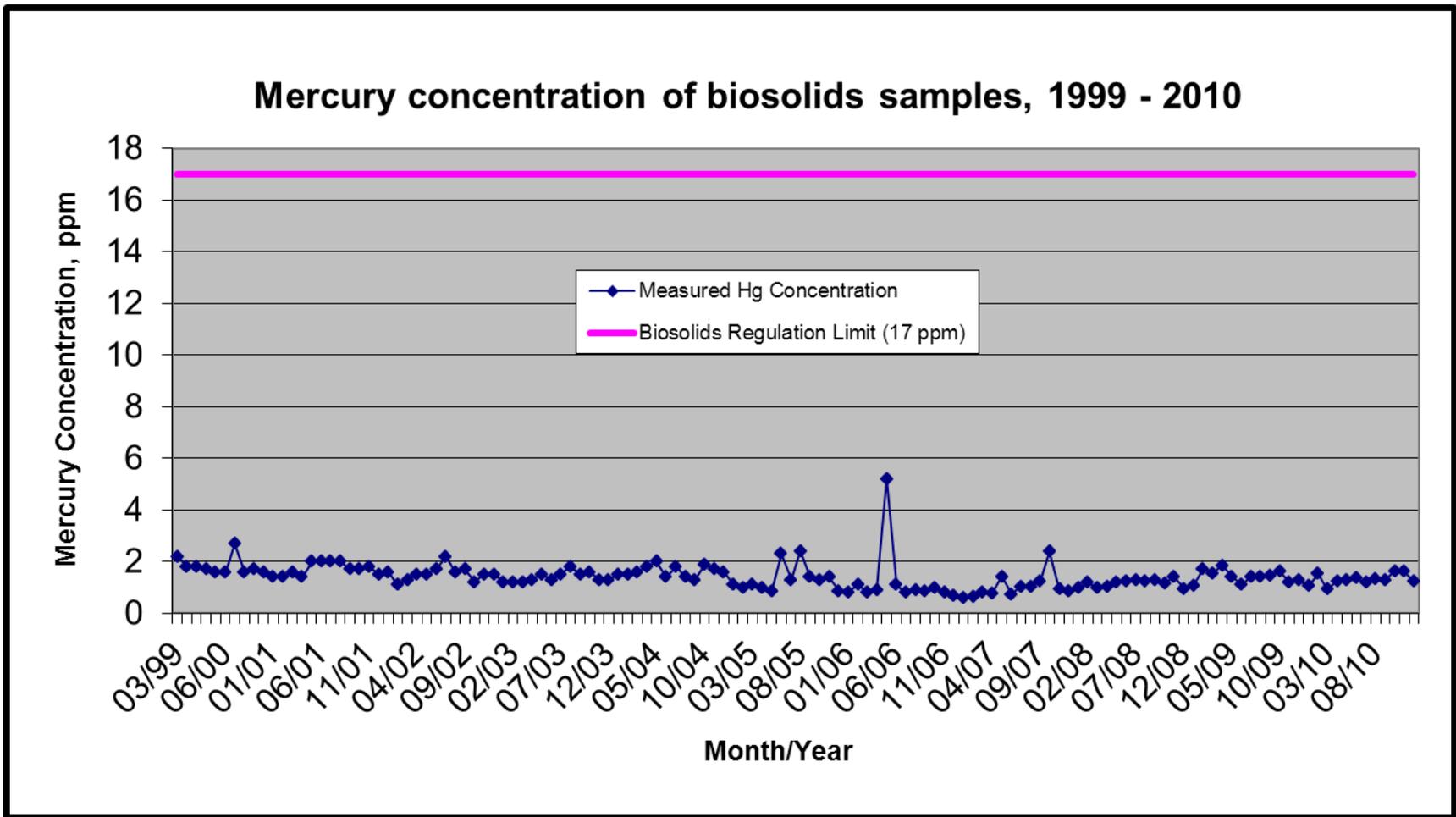


Figure 7. Mercury concentrations of biosolids samples, 1999–2010.

Molybdenum concentration (ppm) of biosolids samples, 1999 - 2010

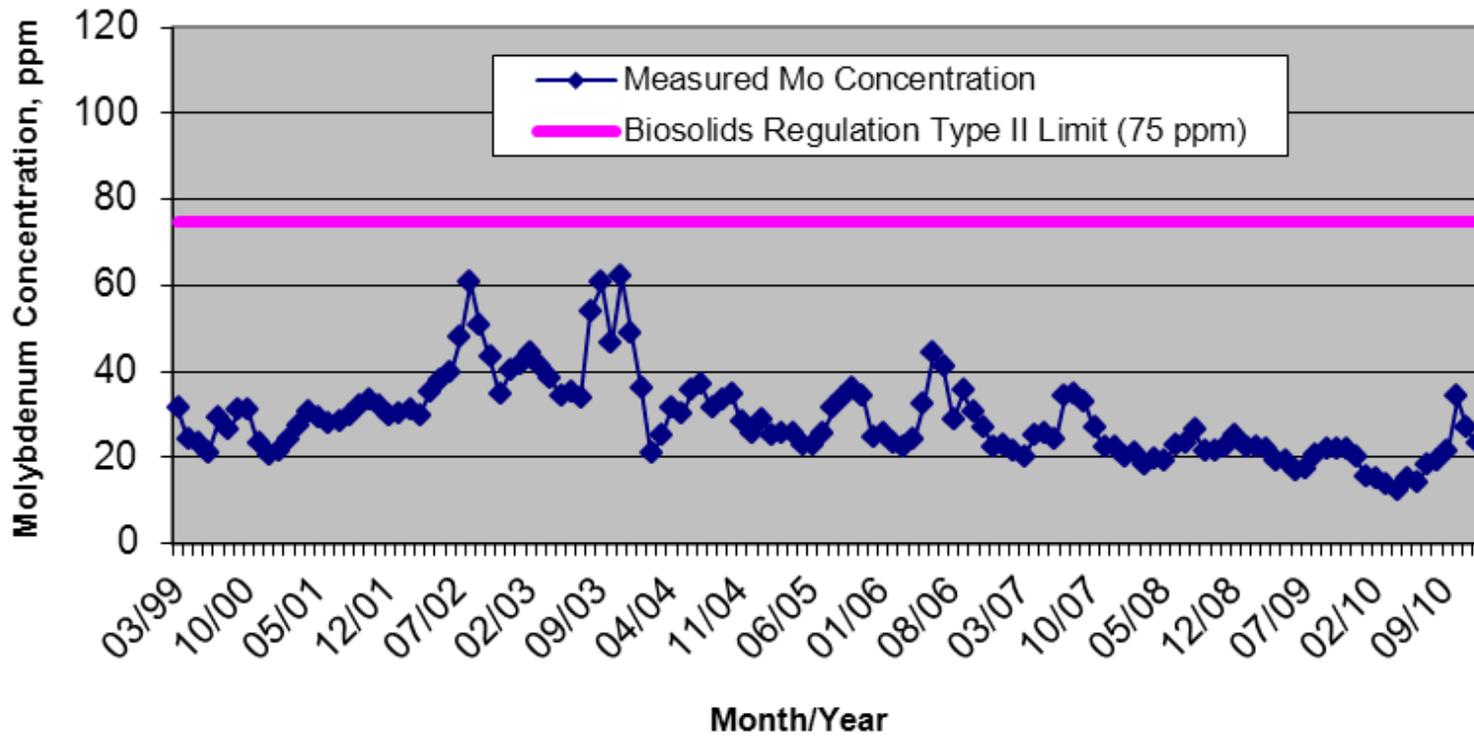


Figure 8. Molybdenum concentrations of biosolids samples, 1999–2010.

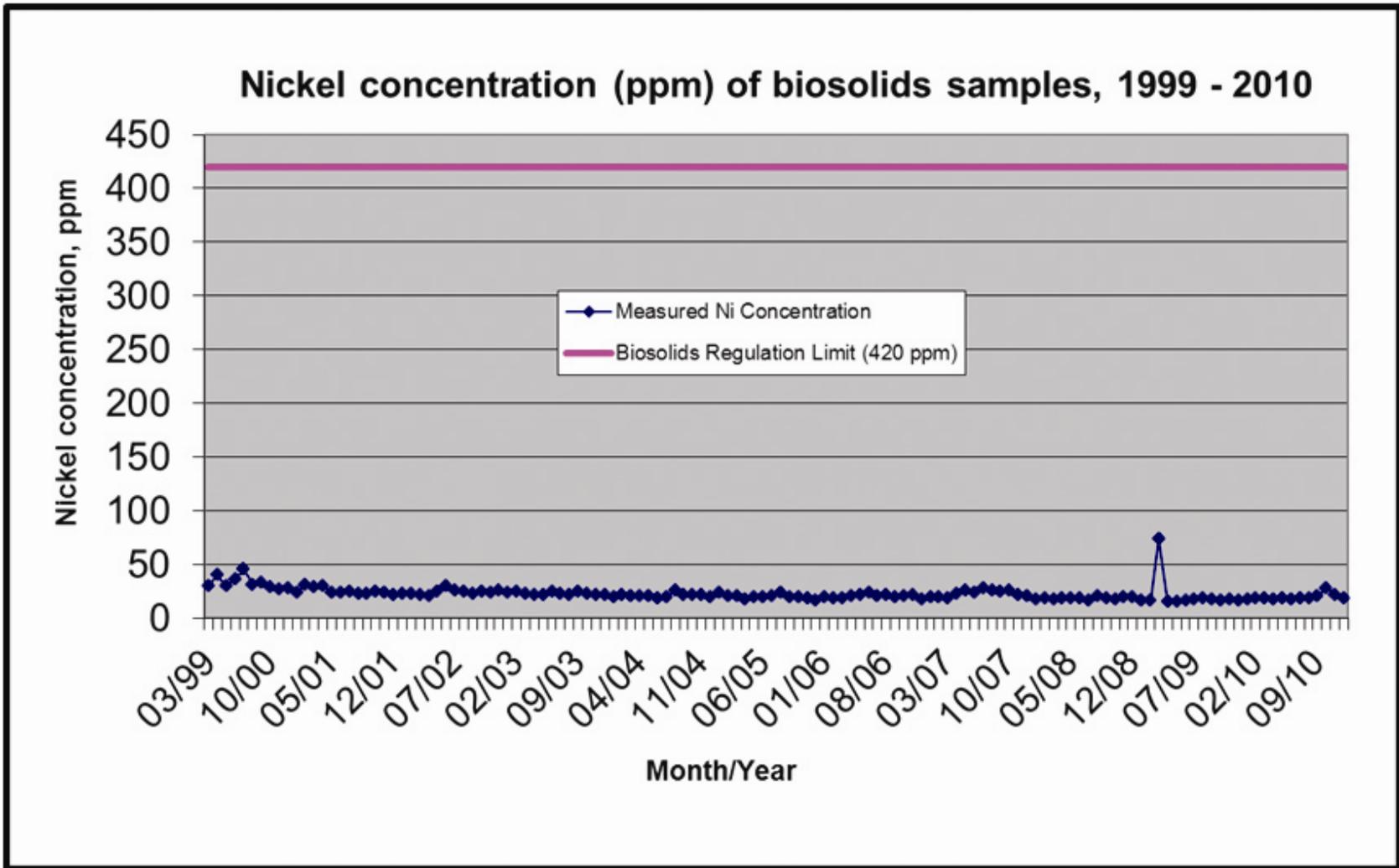


Figure 9. Nickel concentrations of biosolids samples, 1999–2010.

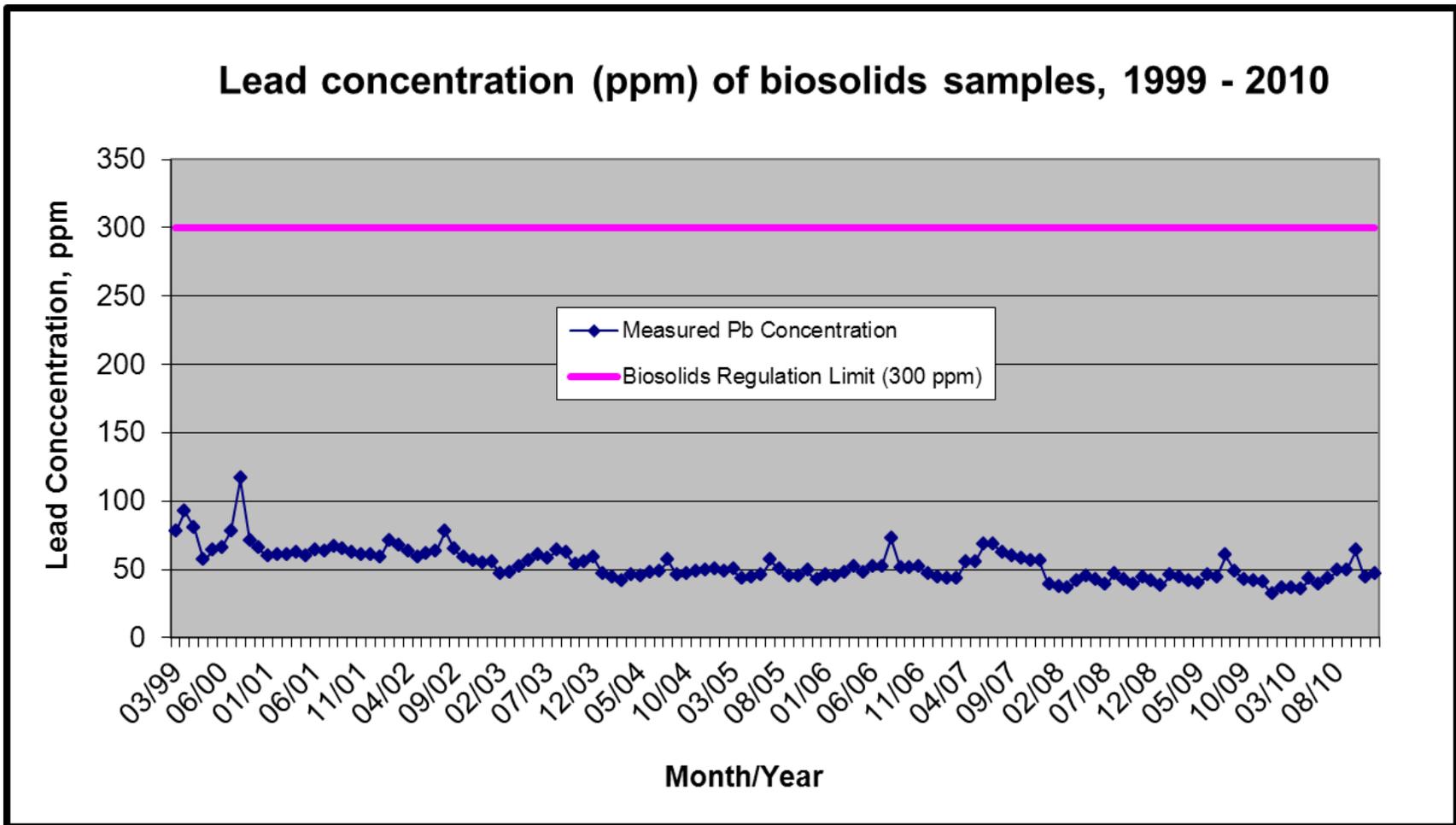


Figure 10. Lead concentrations of biosolids samples, 1999–2010.

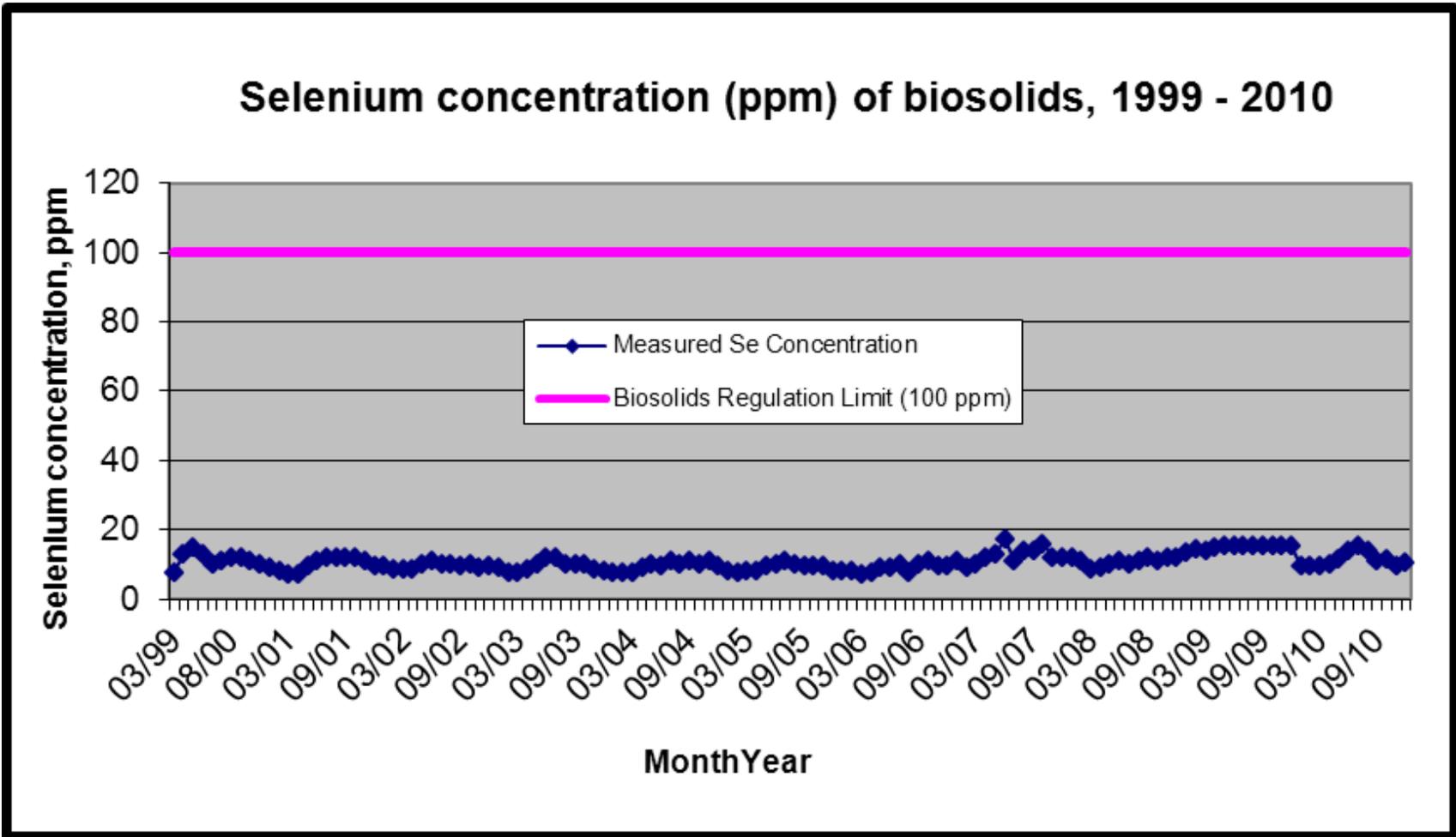


Figure 11. Selenium concentrations of biosolids samples, 1999–2010.

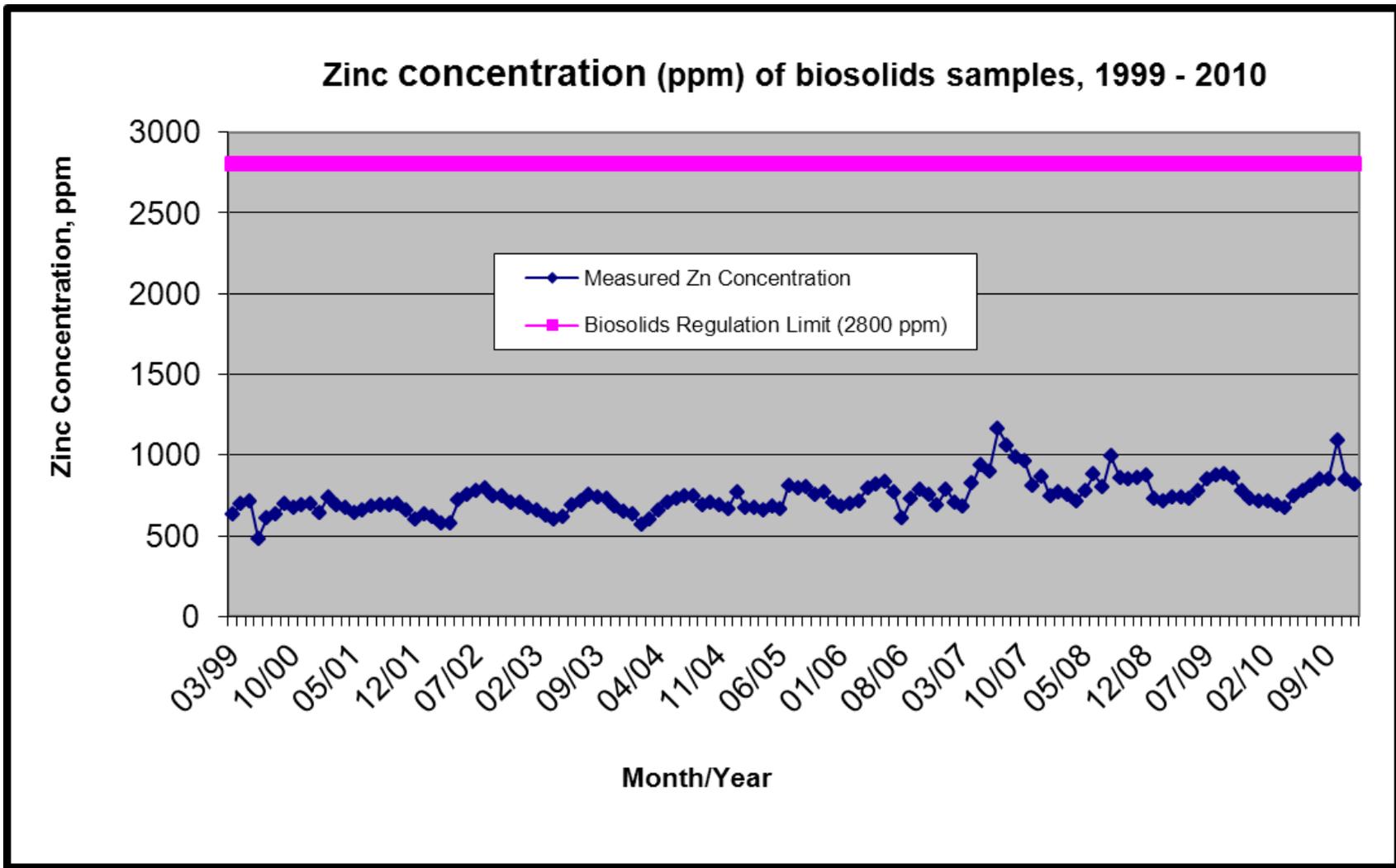


Figure 12. Zinc concentrations of biosolids samples, 1999–2010.

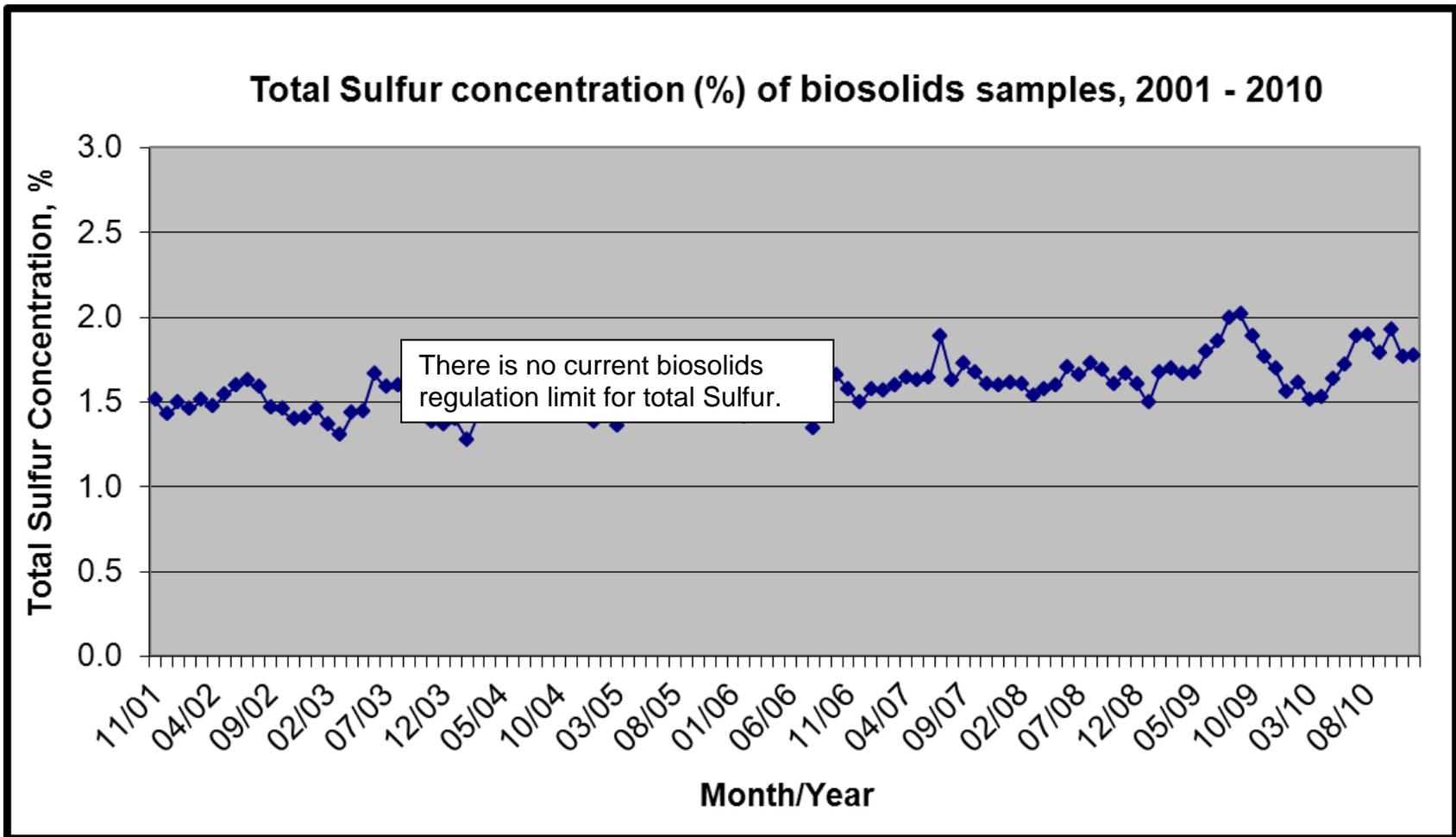


Figure 13. Total sulfur concentration of biosolids samples, 2001–2010.

In conclusion, chemical data for biosolids samples collected from the Metro District plant during a 12-year period (1999–2010) 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.

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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, chap. Q, *of* Taggart, J.E., Jr., ed., Analytical methods for chemical analysis of geological and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 2002–0223, available online 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., 2008a, Analytical results for municipal biosolids samples from a monitoring program near Deer Trail, Colorado (U.S.A.), 1999 through 2006: U.S. Geological Survey Open-File Report 2008–1172, 67 p., available online at <http://pubs.usgs.gov/of/2008/1172/>.
- Crock, J.G., Smith, D.B., Yager, T.J.B., Berry, C.J., and Adams, M.G., 2008b, Analytical results for municipal biosolids samples from a monitoring program near Deer Trail, Colorado (U.S.A.), 2007: U.S. Geological Survey Open-File Report 2008–1358, 35 p., available online at <http://pubs.usgs.gov/of/2008/1358/>.
- Crock, J.G., Smith, D.B., Yager, T.J.B., Berry, C.J., and Adams, M.G., 2009, Analytical results for municipal biosolids samples from a monitoring program near Deer Trail, Colorado (U.S.A.), 2008: U.S. Geological Survey Open-File Report 2009–1090, 29 p., available online at <http://pubs.usgs.gov/of/2009/1090/>.
- Crock, J.G., Smith, D.B., Yager, T.J.B., Berry, C.J., and Adams, M.G., 2010, Analytical results for municipal biosolids samples from a monitoring program near Deer Trail, Colorado (U.S.A.),

- 2009: U.S. Geological Survey Open-File Report 2010–1162, 23 p., available online at <http://pubs.usgs.gov/of/2010/1162/>.
- Hageman, P.L., 2007, TM 5-D2: Determination of mercury in aqueous and geologic materials by continuous flow–cold vapor–atomic fluorescence spectrometry (CVAFS): U.S. Geological Survey Techniques and Methods 5–D2, 6 p., available online 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., 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 02–51, 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, 7 p., available online 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 03–400, 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.
- Yager, T.J.B., Smith, D.B., and Crock, J.G., 2009, Biosolids, crop, and ground-water data for a biosolids-application area near Deer Trail, Colorado, 2004 through 2006: U.S. Geological Survey Digital Data Series 379, 57 p., available online at <http://pubs.usgs.gov/ds/379/>.