



Duncan, Douglas <dduncan@usgs.gov>

EGLCAPA050

4 messages

Marshall, Brian <bymarsha@usgs.gov>
To: Douglas Duncan <dduncan@usgs.gov>
Cc: Chuck Blome <cblome@usgs.gov>


Wed, Nov 19, 2014 at 4:39 PM

Doug,

We met with the OIG staff (Tim Musil and Kathleen Richardson) today. I gave them a copy of the problem description and plan (attached) as well as a copy of the data integrity presentation. Let me know if you have any questions; it will be after the holiday before we know more about the extent and magnitude of deviations caused by the data manipulations.

Brian

Brian D. Marshall
Geochemistry Lab Manager
Energy Resources Science Center
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 **EGLCAPA050 141118.pdf**
212K

Duncan, Douglas <dduncan@usgs.gov>
To: "Marshall, Brian" <bymarsha@usgs.gov>
Cc: Chuck Blome <cblome@usgs.gov>

Wed, Nov 19, 2014 at 5:10 PM

Brian and Chuck,

Thanks for meeting with Kathleen and Tim. I understand from Chuck's phone message that they plan an addendum, rather than rewriting the report, which is good. That implies no changes to their findings. We shall see.

Doug

Douglas W. Duncan
Acting Coordinator
Energy Resources Program
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Duncan, Douglas <dduncan@usgs.gov>
To: Rebecca Bageant <rbageant@usgs.gov>
Cc: Vito Nuccio <vnuccio@usgs.gov>, Jon Kolak <jkolak@usgs.gov>, Ken Skipper <kskipper@usgs.gov>

Wed, Nov 19, 2014 at 5:12 PM

Becky,

Here is Brian's report of the meeting with OIG. Chuck Blome left me a voice message saying that OIG plans to write an

addendum rather than rewriting the report that is currently in draft. I expect and hope that means no changes in the findings.

Doug

Douglas W. Duncan
Acting Coordinator
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U.S. Geological Survey

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Bageant, Rebecca <bbageant@usgs.gov>
To: "Duncan, Douglas" <dduncan@usgs.gov>

Thu, Nov 20, 2014 at 9:27 AM

Thanks for the update!

Becky Bageant
OIG/GAO Audit Liaison Officer
USGS, Office of Budget, Planning and Integration
Phone (703) 648-4328
Fax (703) 648-5068
bbageant@usgs.gov

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Energy Geochemistry Laboratory Corrective Action (CA) and Preventive Action (PA) System

EGLCAPA050

☒ CA ☐ PA

Status: new

Closed? No

Title: Data traceability loss and manipulation of ICP-MS data

Type: non-conformance

Severity: severe

Notified By: walk-in

Description and Plan

Anticipated Closure Date: 12/31/2014

Description

Due to both the investigation related to the Ni issue (EGLCAPA042) and to implementation of a new spreadsheet template for re-formatting data prior to LIMS upload, it was discovered that the ICP-MS analyst had been manipulating data using spreadsheet calculations to correct for minor calibration failures and to improve results on unknowns based on standard analyses. The result of these actions is that the data in the LIMS for ICP-MS cannot be traced back to the raw instrument output. The analyst did not maintain records of the calculations that were performed.

Corrective Action Plan

The analyst has provided an explanation for the use of these calculations and will work with the Inorganic Lead to investigate the magnitude of the corrections for all jobs going back to 2/7/2012, the date of the QA Manual that clearly stated the requirements for traceability and for documentation of all data calculations. Once the extent of the condition is determined, then the data in the LIMS will be corrected and job submitters notified. It is possible that some jobs may be re-analyzed if the differences between the correct values and the original values are outside of 20%. Also, re-analysis may be required if the original jobs show instrument issues that were not corrected (i.e. low count rates). In the meantime, no new analyses are to be performed by ICP-MS (stop-work order on 10/24/2014).

As of 11/5/2014, the ICP-MS Analyst is attempting to reproduce the calculations that were performed, using the data from two runs, one for liquids and one for solids. The results of this effort to reproduce and explain the logic behind the calculations will be presented to the QA Lead and Inorganic Lead by the end of the week of 11/10/2014. The Inorganic Lead is training a new Analyst to operate the ICP-MS instruments; initially only samples that have been selected from the affected jobs will be analyzed. Because neither the Inorganic Lead nor the newly trained Analyst have been tested for competency in the technique, both will be required to read EGL Method 16 and Work Instruction 4. The re-analysis data, if it is to be uploaded to the LIMS, will need verification by an independent lab.

As of 11/18/2014, the ICP-MS Analyst has not been able to reproduce the calculations in detail. The Analyst will be advised to provide the logic of and justification for the data manipulations. Although all raw data has been found and protected, there are issues with reproducing the original data because the raw data requires additional files that have either been overwritten or not saved. These "method" files contain calibration information required to re-process the raw data to calculate concentrations. These files would not be required if all report files are intact. However, the report files have not been preserved in all cases. Beginning in Fall, 2013 new spreadsheet macros were developed that enabled the preservation of report files. Therefore, the investigation will focus on these files initially in an attempt to find the extent and magnitude of the data manipulations.

Proposed corrective actions include:

1. Training on data integrity for all EGL staff. This training occurred on 11/13/2014.
2. Asking each Analyst for each method that generates quantitative data whether any modifications to instrument-generated data are performed; if so then QA Lead will check methods for proper documentation. Revisions of Method documents may be required. Analysts will need to respond by email for record-keeping.
3. The QA Manual will be revised to require a data integrity check between data in LIMS and data generated by instrumentation (and modified by subsequent calculations in some cases). The frequency of these checks will be established by consideration of typical times between data acquisition and data publication.



Duncan, Douglas <dduncan@usgs.gov>

Fwd: EGLCAPA050

1 message

Blome, Charles <cblome@usgs.gov>
To: Douglas Duncan <dduncan@usgs.gov>

Fri, Nov 7, 2014 at 12:36 PM

Doug,

This form is just the beginning of the investigation and explains why we are looking into the issue. Many thanks.

Chuck

----- Forwarded message -----

From: **Marshall, Brian** <bdmarsha@usgs.gov>
Date: Fri, Nov 7, 2014 at 10:23 AM
Subject: EGLCAPA050
To: Chuck Blome <cblome@usgs.gov>

Chuck,

Here is the current version of the problem description and plan. This will continue to evolve as the investigation continues.

Brian

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EGLCAPA050 141105.pdf
209K

Energy Geochemistry Laboratory Corrective Action (CA) and Preventive Action (PA) System

EGLCAPA050

☒ CA ☐ PA

Status: new

Closed? No

Title: Data traceability loss and manipulation of ICP-MS data

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Data Review Summary					
The review showed that in 25 to 30 percent of the samples, some analysis had standard deviations greater than 20 percent due to normalization including ICP, ICPMS, C1, Hg, Se, and S analysis. Updated values will be in future public database releases found on http://energy.cr.usgs.gov/prov/og					
For specific questions please e-mail energylabs@usgs.gov					
Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
5502	WoCQI	Coal	Carboniferous	2006	Done
6500	NaCQI	Coal	Pennsylvanian	2006	Done
6511	WoCQI	Parting	Triassic	2006	Done
6511	Department of State	Parting	Triassic	2006	Done
6514	Appalachian Framework	Coal	Pennsylvanian	2006	Done
6516	Appalachian Framework	Coal	Pennsylvanian	2006	Done
6517	WoCQI	Coal	Carboniferous	2006	Done
6520	WoCQI	Coal	Jurassic	2006	Done
6520	Afghan Reconstruction	Coal	Jurassic	2006	Done
6521	Appalachian Framework	Coal	Pennsylvanian	2006	Done
6522	Appalachian Framework	Coal	Pennsylvanian	2006	Done
6523	WoCQI	Coal	Permian	2006	Done
6541	Health Effects	Sediment Profile 1 West #1	NA	2006	Done
6542	WoCQI	Coal	Carboniferous	2006	Done
6542	Health Effects	Coal Sample KMS-1	Carboniferous	2006	Done
6542	Health Effects	Tailings pond sediments	NA	2006	Done
6544	WoCQI	Coal	Triassic	2006	Done
6544	Department of State	Coal	Triassic	2006	Done
6545	WoCQI	Coal	Tertiary	2006	Done
6553	Health Effects	Sediment Playground Location 016	NA	2006	Done
7002	WoCQI	Coal	Triassic	2007	Done
7002	Department of State	Coal	Triassic	2007	Done
7009	USAID	Coal	Jurassic	2007	Done
7009	Afghan Reconstruction	Coal	Jurassic	2007	Done
7010	USAID	Coal	Neogene	2007	Done
7010	Afghan Reconstruction	Coal	Neogene	2007	Done
7014	WoCQI	Coal	Tertiary	2007	Done
7016	WoCQI	Coal and black shale	Permian	2007	Done

Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
7020	WoCQI	Coal	Cretaceous	2007	Done
E0709017	Appalachian Framework	Coal	Pennsylvanian	2007	Done
E0803006	Okefenokee Peat	Water from peat	modern	2008	Done
EA19	Gulf Coast Coal Assessment	Sabine mine, channel sample	Eocene	1996	Done
EA38	USAID	Coal	Eocene	1997	Done
EA50	USAID	Coal	Eocene	1997	Done
EA51	Gulf Coast Coal Assessment	Coal	Tertiary		Done
EA58	WoCQI	Coal	Permian	1999	Done
EA58	US-India S&T Agreement	Coal	Permian	1999	Done
EA77	WoCQI	Coal	Tertiary	1997	Done
EB07	Gulf Coast Coal Assessment	Coal	Tertiary	1997	Done
EB08	WoCQI	Coal	Tertiary	1997	Done
EB09	WoCQI	Coal	Tertiary	1997	Done
EB11	Gulf Coast Coal Assessment	Coal	Tertiary	1997	Done
EB12	USAID	Coal	Permian	1997	Done
EB13	USAID	Coal	Permian	1997	Done
EB59	USAID	Coal	Permian	1999	Done
EB59	US-India S&T Agreement	Coal	Permian	1999	Done
EB59T	US-India S&T Agreement	Coal	Permian	1999	Done
EB59T	Gulf Coast Coal Assessment	Coal	Tertiary	1999	Done
EB65	WoCQI	Coal	Tertiary	1999	Done
EB83	WoCQI	Coal	Carboniferous	1999	Done
EB84	WoCQI	Coal	Tertiary	1999	Done
EB85	USAID	Coal	Paleogene	1999	Done
EB85	Indo-US Mined Land Reclamation	Coal	Paleogene	1999	Done
EB95	WoCQI	Coal		1999	Done
EB95	USAID	Coal	Permian	1999	Done
EB95	US-India S&T Agreement	Coal	Permian	1999	Done
EB95	Gulf Coast Coal Assessment	Coal	Cretaceous	1999	Done
EC08	Appalachian Framework	Coal	Pennsylvanian	2000	Done
EC09	Appalachian Framework	Coal	Pennsylvanian	2000	Done
EC10	Appalachian Framework	Coal	Pennsylvanian	2007	Done
EC16	Indo-US Mined Land Reclamation	Rhyolite	Precambrian	1999	Done

Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
EC20	WoCQI	Coal	Permian	1999	Done
EC34	WoCQI	Coal	Tertiary	1999	Done
EC38	WoCQI	Coal	Jurassic	1999	Done
EC53	WoCQI	Coal		1999	Done
ERP-00005	Gulf Coast Coal Assessment	Coal	Tertiary	2000	Done
ERP-00009	Indo-US Mined Land Reclamation	Parting	Paleogene	1999	Done
ERP-00014	WoCQI	Coal		1999	Done
ERP-00016	WoCQI	Coal	Permian	1999	Done
ERP-00024	WoCQI	Coal	Permian	1999	Done
ERP-00059	Appalachian Framework	Coal	Pennsylvanian	1999	Done
ERP-00060	Appalachian Framework	Coal	Pennsylvanian	1999	Done
ERP-00062	Gulf Coast Coal Assessment	Coal	Tertiary	2000	Done
ERP-00067	Gulf Coast Coal Assessment	Coal	Tertiary	2000	Done
ERP-00073	WoCQI	Coal	Permian	2000	Done
ERP-00077	WoCQI	Coal	Permian	2000	Done
ERP-00083	WoCQI	Coal	Tertiary	2000	Done
ERP-00084	WoCQI	Coal	Tertiary	2000	Done
ERP-00100	Indo-US Mined Land Reclamation	Coal	Paleogene	2002	Done
ERP-00106	Appalachian Framework	Coal	Pennsylvania	2000	Done
ERP-00107	WoCQI	Coal	Carboniferous-Permian	2000	Done
ERP-00108	WoCQI	Coal	Tertiary	2000	Done
ERP-00114	WoCQI	Coal		2000	Done
ERP-00115	WoCQI	Coal	Carboniferous	2000	Done
ERP-00116	WoCQI	Coal		2000	Done
ERP-00117	WoCQI	Coal	Paleogene	2000	Done
ERP-00118	WoCQI	Coal	Cretaceous	2000	Done
ERP-00129	WoCQI	Coal	Tertiary	2000	Done
ERP-00130	WoCQI	Coal	Tertiary	2000	Done
ERP-00131	WoCQI	Coal	Tertiary	2000	Done
ERP-00132	WoCQI	Coal		2001	Done
ERP-00133	WoCQI	Coal	Tertiary	2001	Done
ERP-00136	WoCQI	Coal	Tertiary	2001	Done
ERP-00137	WoCQI	Coal	Tertiary	2001	Done

Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
ERP-00154	WoCQI	Coal	Tertiary	2001	Done
ERP-00155	WoCQI	Coal	Cretaceous	2001	Done
ERP-00156	WoCQI	Coal	Carboniferous	2001	Done
ERP-00176	WoCQI	Coal	Tertiary	2001	Done
ERP-00177	Appalachian Framework	Coal	Pennsylvanian	2001	Done
ERP-00192	WoCQI	Coal	Tertiary	2001	Done
ERP-00193	WoCQI	Coal	Permian	2001	Done
ERP-00193	WoCQI	Coal	Permian	2002	Done
ERP-00194	WoCQI	Coal	Tertiary	2001	Done
ERP-00195	WoCQI	Coal	Tertiary	2001	Done
ERP-00196	WoCQI	Coal	Tertiary	2001	Done
ERP-00197	WoCQI	Coal	Tertiary	2001	Done
ERP-00198	WoCQI redo of ERP-00107	Coal	Carboniferous-Permian	2000	Done
ERP-00217	Appalachian Framework	Coal	Pennsylvania	2001	Done
ERP-00218	WoCQI	Coal	Permian	2001	Done
ERP-00219	WoCQI	Coal	Permian	2001	Done
ERP-00220	WoCQI	Coal	Tertiary	2001	Done
ERP-00221	WoCQI	Coal	Carboniferous	2001	Done
ERP-00222	WoCQI	Coal	Cretaceous	2001	Done
ERP-00223	WoCQI	Coal	Tertiary	2001	Done
ERP-00227	WoCQI	Coal	Carboniferous	2001	Done
ERP-00236	Appalachian Framework	Coal	Pennsylvania	2002	Done
ERP-00237	Appalachian Framework	Coal	Pennsylvania	2002	Done
ERP-00249	Appalachian Framework	Coal	Pennsylvanian	2002	Done
ERP-00258	WoCQI	Coal and black shale	Permian	2002	Done
ERP-00259	WoCQI	briquette	Permian	2002	Done
ERP-00259	WoCQI	Coal	Permian	2002	Done
ERP-00261	WoCQI	Coal	Permian	2002	Done
ERP-00278	WoCQI	Coal	Tertiary	2002	Done
ERP-00279	WoCQI	Coal	Tertiary	2002	Done
ERP-00292	WoCQI	Coal	Pennsylvanian	2002	Done
ERP-00293	WoCQI	Coal	Carboniferous	2002	Done
ERP-00294	WoCQI	Coal	Tertiary	2002	Done

Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
ERP-00295	WoCQI	Coal	Tertiary	2002	Done
ERP-00296	WoCQI	Coal	Permian	2002	Done
ERP-00296	Department of State	Coal	Permian	2002	Done
ERP-00297	WoCQI	Coal	Neogene	2002	Done
ERP-00297	Department of State	Coal	Neogene	2002	Done
ERP-00298	WoCQI	Coal	Tertiary	2002	Done
ERP-00298	WoCQI	Coal	Tertiary	2003	Done
ERP-00299	WoCQI	Coal	Permian	2002	Done
ERP-00300	WoCQI	Coal	Jurassic	2002	Done
ERP-00300	Afghan Reconstruction	Coal	Jurassic	2002	Done
ERP-00301	WoCQI	Coal	Tertiary	2002	Done
ERP-00301	Gulf Coast Coal Assessment	Coal	Tertiary	2002	Done
ERP-00323	WoCQI	Coal		2002	Done
ERP-00324	WoCQI	Coal	Tertiary	2002	Done
ERP-00326	WoCQI	Coal	Carboniferous	2002	Done
ERP-00329	WoCQI	Coal	Tertiary		Done
ERP-00335	Appalachian Framework	Coal	Pennsylvanian	2003	Done
ERP-00336	CEEP	Coal LM1-2.0	Carboniferous	2003	Done
ERP-00337	WoCQI	Coal	Permian	2002	Done
ERP-00339	WoCQI	Coal	Jurassic	2002	Done
ERP-00339	Department of State	Coal	Jurassic	2002	Done
ERP-00351	WoCQI	Coal	Permian	2003	Done
ERP-00356	WoCQI	Coal	Carboniferous	2003	Done
ERP-00356	Department of State	Coal	Carboniferous	2003	Done
ERP-00372	WoCQI	Coal		2003	Done
ERP-00374	WoCQI	Coal	Carboniferous	2003	Done
ERP-00375	WoCQI	Coal	Carboniferous	2003	Done
ERP-00376	WoCQI	Coal	Permian	2003	Done
ERP-00377	WoCQI	Coal	Permian	2003	Done
ERP-00378	WoCQI	Coal	Triassic	2003	Done
ERP-00379	WoCQI	Coal	Tertiary	2003	Done
ERP-00380	WoCQI	Coal	Carboniferous	2003	Done
ERP-00381	WoCQI	Coal	Permian	2004	Done

Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
ERP-00382	WoCQI	Coal	Permian	2004	Done
ERP-00389	WoCQI	Coal	Carboniferous	2003	Done
ERP-00389	Department of State	Coal	Carboniferous	2003	Done
ERP-00392	WoCQI	Coal		2003	Done
ERP-00396	WoCQI	Coal	Jurassic	2003	Done
ERP-00396	WoCQI	Coal	Jurassic	2005	Done
ERP-00397	WoCQI	Coal	Tertiary	2003	Done
ERP-00398	WoCQI	Coal		2003	Done
ERP-00399	WoCQI	Coal	Jurassic	2004	Done
ERP-00400	WoCQI	Coal	Permian	2004	Done
ERP-00401	WoCQI	Coal	Permian	2005	Done
ERP-00402	WoCQI	Coal	Permian	2005	Done
ERP-00403	WoCQI	Coal	Permian	2005	Done
ERP-00405	WoCQI	Coal	Permian	2003	Done
ERP-00407	WoCQI	Coal	Permian	2003	Done
ERP-00413	Appalachian Framework	Coal	Pennsylvanian	2003	Done
ERP-00420	USAID	Coal	Jurassic	2003	Done
ERP-00420	Afghan Reconstruction	Coal	Jurassic	2003	Done
ERP-00423	NaCQI	Coal	Pennsylvanian	2003	Done
ERP-00424	NaCQI	Coal	Pennsylvanian	2003	Done
ERP-00425	WoCQI	Coal	Permian	2003	Done
ERP-00427	NaCQI	Coal	Pennsylvanian	2003	Done
ERP-00435	Department of Defense	Stone coal	Cambrian	2005	Done
ERP-00437	WoCQI	Coal	Jurassic	2004	Done
ERP-00437	WoCQI	Coal	Jurassic	2005	Done
ERP-00448	WoCQI	Coal	Carboniferous	2004	Done
ERP-00450	USAID	Coal	Jurassic	2004	Done
ERP-00450	Afghan Reconstruction	Coal	Jurassic	2004	Done
ERP-00451	Mussel project with WRD and NPS	River sediments (-8 fraction)	Quaternary	2004	Done
ERP-00452	Mussel project with WRD and NPS	River sediments (+8 fraction)	Quaternary	2004	Done
ERP-00453	Mussel project with WRD and NPS	Coal from river; Big South Fork at Rough Shoals	Quaternary	2004	Done
ERP-00456	WoCQI	Coal	Paleocene	2005	Done
ERP-00457	WoCQI	Coal	Permian	2005	Done

Job Number	Collecting Project/Task Name	Sample Type	Geologic Age	Year Analysis Began	Data Review
ERP-00457	WoCQI	Coal	Permian	2003	Done
ERP-00458	WoCQI	Coal	Cenozoic	2005	Done
ERP-00459	WoCQI	Coal	Permian	2005	Done
ERP-00460	WoCQI	Coal	Tertiary	2005	Done
ERP-00461	WoCQI	Coal		2005	Done
ERP-00462	WoCQI	Dried peat	Quaternary	2005	Done
ERP-00466	Appalachian Framework	Coal	Pennsylvanian	2005	Done
ERP-00476	WoCQI	Coal	Carboniferous	2005	Done
ERP-00477	WoCQI	Coal	Jurassic	2006	Done
ERP-00477	Department of State	Coal	Jurassic	2006	Done
ERP-00489	USAID	Coal	Jurassic	2005	Done
ERP-00489	Afghan Reconstruction	Coal	Jurassic	2005	Done
ERP-00490	Appalachian Framework	Coal	Pennsylvanian	2005	Done

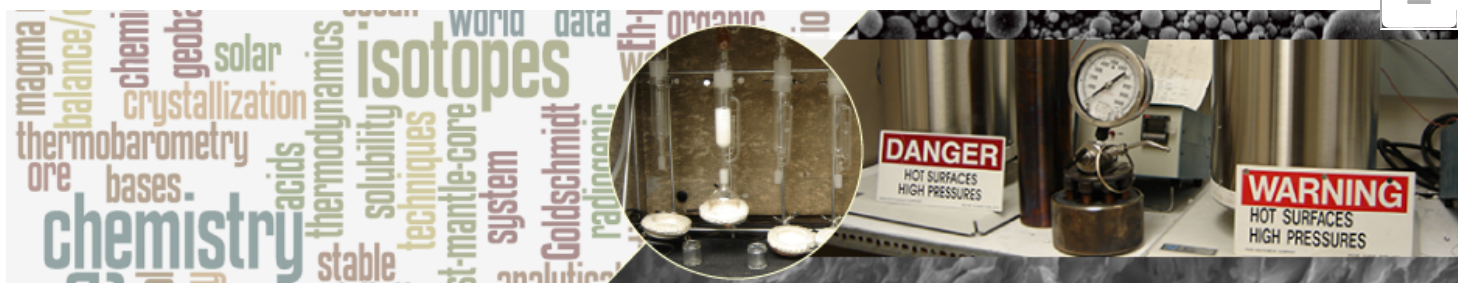


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Geochemistry Laboratories

Notice - Quality Assurance Update (4/10/2010):

Background:

A QUALITY CONTROL ISSUE WAS IDENTIFIED WITH SOME GEOCHEMICAL DATA PRODUCED BY THE DENVER ENERGY GEOCHEMICAL LABORATORY FROM 1996-2008.

Quality Assurance Update

The USGS Energy Resources Program Geochemistry Laboratory initiated a review of its quality assurance practices in 2008, covering quality control and methodology used in inorganic chemical analyses of coal, coal power plant ash, water and sediment samples.

This quality control review found that inorganic chemical analyses by the USGS ERP Geochemistry Laboratory from 1996 through 2008 incorporated quality practices that did not meet standards commonly in use at the time. The most serious shortcoming was the adjustment of raw data to a standard when the instrument reading for the standard was beyond acceptable limits, or when the frequency of repeat analyses of standards was insufficient. In general, adjustment of raw data to account for instrument drift is acceptable practice within strictly defined limits. During the period in question, the maximum adjustment of instrument readings, guided by calibration standards, was not allowed to exceed 10%. However, in some cases the adjustment exceeded 10% and/or was not constrained by an adequate number of control standards. Original instrument readings no longer exist for about 80% of the analyses in question and we are unable to determine the acceptability of drift corrections for most of the samples analyzed during this period. For these reasons, 1996-2008 data from the USGS ERP Inorganic Geochemistry Laboratory should be described as "semi-quantitative" and should be used with care.

DATA REVIEW SUMMARY RELATED TO DATA PRODUCED BY THE DENVER ENERGY GEOCHEMICAL LABORATORY FROM 1996-2008

The review showed that in 25-30 percent of the samples, some analysis had standard deviations greater than 20 percent due to normalization including ICP, ICPMS, Cl, Hg, Se, and S analysis.

For specific questions please e-mail energylabs@usgs.gov

Download the table of affected jobs directly:

- Tabular [.xls](#)

VERSION CONTROL

Previous Version: [Archived QA Manual](#)

GEOCHEMISTRY LABORATORIES TOPICS

[Project Homepage](#)

Project Staff

Energy Geochemistry Database

Sample Submittal

Methods

Proficiency Testing and Quality Control

Notice: Data Quality Issue (5/25/2016)

Notice: Quality Assurance Update (4/10/2010)

TOOLBOX



Publications & Advanced Search

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