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To: Philip Trowbridge, RMP Manager
Jay Davis, RMP Lead Scientist
From: Don Yee, Quality Assurance Officer
Date: December 13, 2017
Re: 2016 RMP Bird Egg Samples Quality Assurance Report

Introduction

In 2016, bird egg samples were collected from 4 areas for terns with 84 individual samples analyzed for Hg and 12 composites analyzed for Se. Samples for cormorants were collected from 3 areas, with 9 composites analyzed for Hg, Se, PFCs, PCBs, and PBDEs. The sampling and analysis was conducted by the Regional Monitoring Program for Water Quality in San Francisco Bay. The details of the cruise and sample collection methods are described in the RMP Quality Assurance Program Plan, cruise plans, cruise reports, and field sampling reports. These documents are available from the SFEI website (http://www.sfei.org/programs/sf-bay-regional-monitoring-program).

The samples were analyzed for the following compounds by the laboratories indicated:

- *USGS-WERC – Tern Hg*
- *MPSL-DFW – Cormorant Hg, Tern & Cormorant Se*
- *SGS-AXYS – Cormorant PFC*
- *DFW-WPCL – Cormorant PCB, PBDE*

The SFEI Data Services Team checked the laboratory results using the methods and data quality objectives in the RMP Quality Assurance Project Plan (QAPP). Overall, 99% or more of the results for analyte groups except PBDEs and PCBs in cormorant eggs were determined to be acceptable for use in RMP reports and calculations. For egg PBDEs and PCBs, results for the only lab replicate was lost due to misprocessing of the sample, so only 90% of PCBs were reportable, and 83% of PBDEs were reported, with some additional results not reported due to sporadic blank contamination at concentrations similar to those in field samples.

This memo provides a high-level summary of the quality assurance assessment for each dataset. Non-conformances with the QAPP and corrective actions needed for the next round of monitoring are highlighted in gray shading. The details of the quality assurance assessment of each dataset are provided in Appendix A.

The data have been approved by the RMP Manager and Lead Scientist, and all results have been uploaded to the San Francisco Regional Data Center and CEDEN; rejected results are uploaded but can only be downloaded by special request rather than though standard queries.

Quality Assurance Summary for 2016 RMP Bird Egg Samples

USGS-WERC – Tern Hg
2016 tern egg samples analyzed for Hg by USGS-WERC had no significant issues, with 100% reportable results. There were no non-detects, no detected blank contamination, and precision and recovery were well within targets for all samples. Sample concentrations were also within ~25% of previous year means.

MPSL-DFW – Cormorant Hg
2016 cormorant egg samples analyzed for Hg by MPSL had no significant issues, with 100% reportable results. There were no non-detects, no detected blank contamination, and precision and recovery were well within targets for all samples, and mean concentrations were similar to previous years.

MPSL-DFW – Tern & Cormorant Se
2016 cormorant egg samples analyzed for Se by MPSL had no significant issues, with 100% reportable results. There were no non-detects, no detected blank contamination, and precision and recovery were well within targets for all samples, and mean concentrations were almost identical to previous years.

SGS-AXYS – Cormorant PFC
2016 cormorant egg samples analyzed for PFCs by SGS-AXYS had no significant issues, with 99% reportable results (1 result lab-rejected). There were numerous non-detects for half the analytes, but no detected blank contamination. Precision and recovery were well within targets for all samples, and mean concentrations were somewhat similar to previous years ranging from 30-200% of past results depending on compound.

DFW-WPCL – Cormorant PCB

2016 cormorant egg samples analyzed for PCBs by WPCL had several issues, with 90% reportable results, due to the misprocessing of the only lab replicate sample, so there is no way to distinguish lab from field variability. There were numerous non-detects for 9 of the PCBs, but no detected blank contamination. Precision on the one lab replicate was poor in part due to an extraction problem noted by the lab (no congeners reported for that sample), but variation among site replicates was also large (in part perhaps natural variation). Recoveries were within targets for all but 5 PCBs, flagged but not censored. Mean concentrations were within 2-3x of previous years for most congeners, but as low as ~10% (10x lower) for a few individual compounds (largely those near detection limits).

DFW-WPCL – Cormorant PBDE

2016 cormorant egg samples analyzed for PBDEs by WPCL had several issues, with 83% reportable results. There were numerous non-detects for half of the PBDEs, and detected blank contamination for 3 congeners, with nearly all PBDE 17 and 28 results censored for being less than 3x the blank. Precision on the one lab replicate was poor in part due to an

extraction problem noted by the lab and results for all congeners in that replicate rejected (thus there is no way to evaluate analytical variability separately from field variability), but variation among site replicates was also moderately large (in part perhaps natural variation). Recoveries were outside the target average 35% error for 5 congeners, flagged but not censored. Mean concentrations were within about 2x of 2012 results, but as low as ~10% (10x lower) for a few individual compounds (usually those nearer detection limits).

With the level of deviations in precision seen in PCBs & PBDE samples, if we were continuing analyses in future years with the lab, we might ask for archives to be analyzed. However, the lab is discontinuing analyses for outside groups, so diagnosing/fixing discrepancies is of limited usefulness, and the variation among field replicates provides some estimates of precision in place of lab replicates. Archived samples are likely better spent in evaluating potential new labs for analyzing these bird egg samples.

Appendix: Dataset QA Summaries

USGS-WERC

Tern Eggs

Hg and Moisture

QA Issues for Project Manager to Review

Reporting Issues for Lab to Review

Formatting Issues for Data Manager to Review

Hold time review (especially desired by stormwater programs)
All samples were analyzed for mercury between 124 and 173 days after collection within the 1 year hold time used by the USGS for bird eggs.

QA Review
Dataset completeness
Results were reported for mercury and moisture in 84 tern egg composites (Total) analyzed in 1 lab batch, with all 100% results reportable. Lab replicates and matrix spike/matrix spike replicates (MS/MSDs) were analyzed for mercury. Other client samples, continuing calibration verification (CCV), certified reference materials (CRM), equipment blanks, and method blanks were also reported. All results were submitted not blank corrected.
The QA review was conducted on wet weight LABQA results (converted from the original dry weight results submitted by USGS).

Overall acceptability
Overall the data were acceptable. MDLs were sufficient with no non-detects reported for mercury in any of the composites. Mercury was not measured in the method or equipment blanks at levels above the method detection limits.

Accuracy was evaluated using the certified reference materials with the average error of 1.49% being well below the target MQO of 35%. Matrix spike accuracy was also below the target MQO (average %error of 10.23%). No qualifiers were necessary.

Precision was evaluated for mercury using the laboratory replicates of the composites. The average RSD of 1.17% was well below the 35% target MQO. The precision of matrix spike replicates was also well below the target MQO (average RSD of 1.39%). Certified reference materials were not used for the evaluation as they were two different materials (fish protein and lobster hepatopancreas).

Average mercury concentrations were compared to the average RMP EEPS concentrations for Forster's Terns from previous years (2002 - 2012) and ranged from 73% to 95% (0.7x to ~1x greater), depending on the unit basis (fww being 73% and ww 95%). Moisture results were comparable 74% versus 70.5%.

MDLs sensitivity
MDLs were sufficient with no non-detects reported for mercury in any of the composites.

QB averages (procedural, field blank)
Mercury was not measured in the method or equipment blanks at levels above the method detection limits.

Accuracy (using a variety of SRMs or Matrix spike QRECs)
Accuracy was evaluated using the certified reference materials with the average error of 1.49% being well below the target MQO of 35%. Matrix spike accuracy was also below the target MQO (average %error of 10.23%). No qualifiers were necessary.

Average precision from replicate field sample
Precision was evaluated for mercury using the laboratory replicates of the composites. The average RSD of 1.17% was well below the 35% target MQO. The precision of matrix spike replicates was also well below the target MQO (average RSD of 1.39%). Certified reference materials were not used for the evaluation as they were two different materials (fish protein and lobster hepatopancreas).

Comparison of dissolved and total phases
Not Applicable

Comparison to previous years
Average mercury concentrations were compared to the average RMP EEPS concentrations for Forster's Terns from previous years (2002 - 2012) and ranged from 73% to 95% (0.7x to ~1x greater), depending on the unit basis (fww being 73% and ww 95%). Moisture results were comparable 74% versus 70.5%.

Ratio Checking Summary
Not Applicable

MPSL-DFG

Cormorant Eggs

Hg and Moisture

QA Issues for Project Manager to Review

Reporting Issues for Lab to Review

Formatting Issues for Data Manager to Review

Hold time review (especially desired by stormwater programs)

All samples were analyzed for mercury between 77 and 130 days after collection within the 1 year hold time specified by MPLS-DFG.

QA Review

Dataset completeness

Results were reported for mercury and moisture in 62 cormorant egg composites (Total) analyzed in 4 lab batches. Lab replicates, matrix spike/matrix spike replicates (MS/MSDs), certified reference materials (CRMs), and method blanks were also reported. Results were reported in ug/g ww and were submitted not blank corrected.

Overall acceptability

Overall the data were acceptable. MDLs were sufficient with no non-detects reported for mercury in any of the composites. Mercury was not measured in the method blanks at levels above the method detection limits (all non-detects).

Accuracy was evaluated using the certified reference materials with the average error of 8.96% being well below the target MQO of 35%. Matrix spike accuracy was also below the target MQO (average %error of 9.29%). No qualifiers were necessary.

Precision was evaluated for mercury using the laboratory replicates of the composites. The average RSD of 2.53% was well below the 35% target MQO. The precision of the certified reference material and matrix spike replicates were also well below the target MQO (average RSD of 10.18% and 17%, respectively). No qualifiers were added.

Average mercury concentrations were compared to the average RMP EEPS cormorant egg concentrations from previous years (2002 - 2012) and were similar and comparable (both 0.62 ug/g ww).

MDLs sensitivity

MDLs were sufficient with no non-detects reported for mercury in any of the composites.

QB averages (procedural, field blank)

Mercury was not measured in the method blanks at levels above the method detection limits (all non-detects).

Accuracy (using a variety of SRMs or Matrix spike QRECs)

Accuracy was evaluated using the certified reference materials with the average error of 8.96% being well below the target MQO of 35%. Matrix spike accuracy was also below the target MQO (average %error of 9.29%). No qualifiers were necessary.

Average precision from replicate field sample

Precision was evaluated for mercury using the laboratory replicates of the composites. The average RSD of 2.53% was well below the 35% target MQO. The precision of the certified reference material and matrix spike replicates were also well below the target MQO (average RSD of 10.18% and 17%, respectively). No qualifiers were added.

Comparison of dissolved and total phases
Not Applicable

Comparison to previous years
Average mercury concentrations were compared to the average RMP EEPS cormorant egg concentrations from previous years (2002 - 2012) and were similar and comparable (both 0.62 ug/g ww).

Ratio Checking Summary
Not Applicable

MPSL-DFG

Tern and Cormorant Eggs

Se and Moisture

QA Issues for Project Manager to Review

Reporting Issues for Lab to Review

Formatting Issues for Data Manager to Review

Hold time review (especially desired by stormwater programs)
All samples were analyzed for selenium between 180 and 232 days after collection. No hold time is specified for selenium in the RMP QAPP.

QA Review
Dataset completeness
Results were reported for selenium and moisture in 9 cormorant egg composites and 12 tern egg composites analyzed in 2 lab batches, with 100% of results reportable. Other client samples [including lab replicates and matrix spike/matrix spike replicates (MS/MSDs)], laboratory control samples, and method blanks were also reported. Results were reported in ug/g dw and selenium results were submitted blank corrected.

Overall acceptability
Overall the data were acceptable. MDLs were sufficient with no non-detects reported for selenium in any of the composites. Selenium was not measured in the method blanks at levels above the method detection limits (all non-detects).

Accuracy was evaluated using the matrix spike samples with the average error of 6.62% being well below the target MQO of 35%. Laboratory control sample accuracy was also below the target MQO (average %error of 4.48%). No qualifiers were necessary.

Precision was evaluated for selenium using the laboratory replicates of the composites. The average RSD of 23.1% was below the 35% target MQO. The precision of the matrix spike and laboratory control sample replicates were well below the target MQO (average RSD of 3.6% and 0.51%, respectively). No qualifiers were added.

Average selenium concentrations were compared to the average RMP EEPS cormorant and tern egg concentrations from previous years (2002 - 2012) and were similar and comparable (cormorant: 3.82 versus 3.80 ug/g dw; tern: 3.87 versus 3.896 ug/g dw).

MDLs sensitivity
MDLs were sufficient with no non-detects reported for selenium in any of the composites.

QB averages (procedural, field blank)
Selenium was not measured in the method blanks at levels above the method detection limits (all non-detects).

Accuracy (using a variety of SRMs or Matrix spike QRECs)
Accuracy was evaluated using the matrix spike samples with the average error of 6.62% being well below the target MQO of 35%. Laboratory control sample accuracy was also below the target MQO (average %error of 4.48%). No qualifiers were necessary.

Average precision from replicate field sample
Precision was evaluated for selenium using the laboratory replicates of the composites. The average RSD of 23.1% was below the 35% target MQO. The precision of the matrix spike and laboratory control sample replicates were well below the target MQO (average RSD of 3.6% and 0.51%, respectively). No qualifiers were added.

Comparison of dissolved and total phases
Not Applicable

Comparison to previous years
Average selenium concentrations were compared to the average RMP EEPS cormorant and tern egg concentrations from previous years (2002 - 2012) and were similar and comparable (cormorant: 3.82 versus 3.80 ug/g dw; tern: 3.87 versus 3.896 ug/g dw).

Ratio Checking Summary
Not Applicable

SGS-AXYS

Cormorant Eggs

PFCs and Moisture

QA Issues for Project Manager to Review

Hold time review (especially desired by stormwater programs)

All samples were analyzed for PFCs between 52 and 106 days after collection. No hold time is listed in the RMP QAPP.

QA Review

Dataset completeness

Results were reported for thirteen PFCs, lipid, and moisture in 9 cormorant egg composites (Total) analyzed in 1 lab batch. A lab replicate, matrix spike/matrix spike replicates (MS/MSDs), a certified reference material (CRM: NIST 1947), laboratory control sample (LCS), and method blank were also analyzed. All results were submitted not blank corrected.

Overall acceptability

Overall the data were acceptable. MDLs were generally sufficient with non-detects reported for 54% (7 out of 13 PFCs); six of them having extensive non-detects (ND> 50%): Perfluorobutanesulfonate, Perfluoroheptanoate, Perfluorohexanoate, Perfluoropentanoate (100%), Perfluorobutanoate (90%), and Perfluorooctanesulfonamide (70%).

No PFCs were measured in the method blanks at levels above the method detection limits. Accuracy was evaluated using the matrix spikes, except for Perfluorodecanoate, Perfluorododecanoate, and Perfluorooctanesulfonate which were evaluated using the laboratory control spikes. The certified reference material was not used for Perfluorooctanesulfonate as the certificate value is not certified (is a reference value). The average %error ranged from 0.50% to 16.54% well below the target MQO of 35%. Laboratory control spike accuracy for the rest of the PFCs was examined and was below the target MQO (ranged from 3% to 18.5%). No qualifiers were necessary.

Precision was evaluated using the laboratory replicates of the composites, except for Perfluoropentanoate, Perfluorooctanesulfonamide, Perfluorohexanoate, Perfluoroheptanoate, Perfluorobutanoate, and Perfluorobutanesulfonate which were evaluated using the matrix spike replicates. The average RSD ranged from 0% to 16.39% of 1.17% all below the 35% target MQO. The precision of the other matrix spike replicates for the remaining PFCs was also well below the target MQO (ranged from 0.43% to 10.43%). No qualifiers were necessary.
Average PFC concentrations were compared to the average RMP EEPS concentrations for Cormorant eggs from previous years (2006 and 2009) and ranged from 29.5% to 194% (0.3x to ~2x greater), depending on the specific compound.

MDLs sensitivity

MDLs were generally sufficient with non-detects reported for 54% (7 out of 13 PFCs); six of them having extensive non-detects (ND> 50%). Perfluorobutanesulfonate (100% NDs), Perfluoroheptanoate (100%), Perfluorohexanoate (100%), Perfluoropentanoate (100%), Perfluorobutanoate (90%), and Perfluorooctanesulfonamide (70%).

QB averages (procedural, field blank)
No PFCs were measured in the method blanks at levels above the method detection limits.

Accuracy (using a variety of SRMs or Matrix spike QRECs)
Accuracy was evaluated using the matrix spikes, except for Perfluorodecanoate, Perfluorododecanoate, and Perfluorooctanesulfonate which were evaluated using the laboratory control spikes. The certified reference material was not used for Perfluorooctanesulfonate as the certificate value is not certified (is a reference value). The average %error ranged from 0.50% to 16.54% well below the target MQO of 35%. Laboratory control spike accuracy for the rest of the PFCs was examined and was below the target MQO (ranged from 3% to 18.5%). No qualifiers were necessary.

Average precision from replicate field sample
Precision was evaluated using the laboratory replicates of the composites, except for Perfluoropentanoate, Perfluorooctanesulfonamide, Perfluorohexanoate, Perfluoroheptanoate, Perfluorobutanoate, and Perfluorobutanesulfonate which were evaluated using the matrix spike replicates. The average RSD ranged from 0% to 16.39% of 1.17% all below the 35% target MQO. The precision of the other matrix spike replicates for the remaining PFCs was also well below the target MQO (ranged from 0.43% to 10.43%). No qualifiers were necessary.

Comparison of dissolved and total phases
Not Applicable

Comparison to previous years
Average PFC concentrations were compared to the average RMP EEPS concentrations for Cormorant eggs from previous years (2006 and 2009) and ranged from 29.5% to 194% (0.3x to ~2x greater), depending on the specific compound.

<u>Ratio Checking Summary & Lab Follow-up</u>

The following email thread documents issues raised and resolved during the ratio review.

> <u>Email from Meg Sedlak</u>
>
> Hi John and Amy,
>
> The PFAS data looks good with one possible exception. PFOSA, a PFOS precursor, was detected at relatively high concentrations in one Central Bay sample (1.34); it was not detected in the other two CB samples. We haven't seen PFOSA in seal samples in the

past and it's a bit odd that it would appear in the Richmond CB site which has lower concentrations of PFAS (particularly PFOS) than the SB sites. You may want to ask the lab to confirm that detection. That said, PFOSA was detected twice in the lower SB samples (although one detect is near the MDL and it is not detected in the corresponding duplicate). Its possible the MDLs have come down since the prior work.

The RPDs are good ranging from 4.3 to 26 %. PFAS were not detected in the blanks. All good.

The data are very interesting. PFOS concentrations have increased in the SB from ~380 in 2012 to ~600 in 2016 (geometric means). We saw significant declines in PFOS in the 2012 samples as compared to the prior work (2006/2009). I am not sure why the concentrations went back up but it is believable to me given the vagaries of biological samples. Off the top of my head, we might attribute one of the factors being the drought with a higher level of effluent in the SB. It would be interesting to know whether the other contaminants in birds increased as well (at least those associated with effluent).

Concentrations of some of the longer chained PFASs such as PFDoDA (C12) and PFUnDA (C11) also increased which may suggest the degradation of some of the longer-chained precursors such as the fluoroteleomer alcohols as the market shifts to alternatives. I will need to look into this. Short-chained compounds such as PFBA and PFBS were not detected which is consistent with our prior work.

All in all, lots of interesting things to think about. I think it could make for a great poster at SETAC this year.

I really appreciate all the hard work that you do to maintain a robust and accurate data base. Again my apologies for the slowness in my review.

Regards,

Meg

PS Please let me know what AXYS say about the PFOSA detect. It could be right.....

4/11/2017 email from AXYS

Hi John,

I asked our lead chemist to review ALL analytical data for quality and possible errors for this sample. No indication of an error could be found and there was no evidence of sample to sample carryover suggested for the result reported. The data was re-verified as correct and the result deemed to be valid.

Andrew Porat

4/13/2017 emails

Meg,

Just want to confirm you are okay with reporting the data as is?

John

Yes. Thank you.

Meg

DFG-WPCL

Cormorant Eggs

PCB

QA Issues for Project Manager to Review
The lab reported in the batch comment:
Diln. Coeln. Portion of L-490-16-09-DUP sample lost during ASE extraction procedure due to cell leakage resulting in low surr %R, hi RPD. Not enough sample remaining to re-extract. Hi concentrations of target analytes in MS/D, some %R, RPD not calculable.

For the time being, results for that entire duplicate sample are labeled as “Rej” (rejected).

Reporting Issues for Lab to Review

Formatting Issues for Data Manager to Review

Hold time review (especially desired by stormwater programs)
All samples were analyzed within less than 200 days, well within the 1 year target hold time.

QA Review
Dataset completeness
Results were reported for 10 samples (3 sites x 3 composites + 1 lab replicate) for 54 PCBs including some coeluters, moisture, and lipids.

Percent usable (non-reject) field data
90% of the data were reportable, but a large percentage (>80%) of results were qualified, due to variability resulting from some extraction issues.

Overall acceptability
Overall the data are marginally acceptable, primarily due to poor precision resulting from extraction problems..

MDLs sensitivity
The method was sensitive enough to get detects in at least 50% of samples other than for 9 of the PCBs.

QB averages (procedural, field blank)
None of the PCB congeners were detected in the method blank.

Average precision from replicate field sample
Precision on lab replicates was marginal, with many analytes showing RSDs over the target 35% and many in excess of 50%. The variation among field replicates (composites within a site from different sets of eggs) further exacerbated this variability. Similar variation was found in PBDEs, but was less notable due to the smaller number of target analytes and lower concentrations overall. The affected analytes were flagged VIL, but not censored. The lab noted some problems in the analysis of the dupe leading to overcorrection for surrogate loss, so all PCB analytes for that sample were flagged as estimated, and high biased (VJ,VHB). Even after rejecting the lab dupe, intrasite variation is pretty high among the composites

Accuracy (using a variety of SRMs or Matrix spike QRECs)
Recoveries on matrix spike and CRM samples was generally good for PCBs, with recovery errors typically around 20-30%, with only 5 PCBs with recovery errors between 35% and 50%, flagged (VIU) but not censored.

Comparison of dissolved and total phases
Not applicable

Comparison to previous years
For the most part concentrations were within a factor of 2 or 3 from 2012 or 2009 results, other than PCBs 087 to 095 which were about 4 to 9x the previous years' averages. Concentrations on those congeners are generally low so it might just be indicative of noise on a low signal.

Ratio Checking Summary

The following email thread documents issues raised and resolved during the ratio review.

4/11/2017 email from Jay Davis

Hi John;
The congener ratios look good. Nothing suspicious there.
The only thing anomalous is the lower concentration replicate of SBComp_3_L25358-9, which is hopefully the one with the extraction problem.
Thanks,
Jay

4/13/2017 email from Don Yee

The one with the problem was

SampleID	LabSampleID

SBComp_3_L25358-9	L-490-16-09 DUP

For the most part it had lower conc on a few congeners I spot checked.
Don

4/17/2017 emails from Jay Davis
On Mon, Apr 17, 2017 at 7:13 AM, Jay Davis <jay@sfei.org> wrote:
Hi John;
Yes.
I would prefer that data that are flagged as rejected (like for this sample) are not by default downloaded with the rest of the dataset, or that somehow data users are warned not to just take these values and blindly include them in their analyses. Do we have a mechanism in place to prevent that?
Thanks,
Jay

On Mon, Apr 17, 2017 at 7:41 AM, John Ross <johnr@sfei.org> wrote:
Jay,
Only if we flag each individual rejected result so it is not included in a CD3 download. For CEDEN we might have to flag as do not export.
John

I think we should take those steps. The data for that sample are not reliable.
Thanks,
Jay

DFG-WPCL

Cormorant Eggs

PBDE

QA Issues for Project Manager to Review
The lab reported in the batch note:
Diln. Coeln. Portion of L-490-16-09-DUP sample lost during ASE extraction procedure due to cell leakage resulting in low surr %R, hi RPD. Not enough sample remaining to re-extract. Hi concentrations of target analytes in MS/D, some %R, RPD not calculable.

For the time being, results for that entire duplicate sample are labeled as “Rej” (rejected).

Reporting Issues for Lab to Review

Formatting Issues for Data Manager to Review

Hold time review (especially desired by stormwater programs)
All samples were analyzed within ~200 days or less, well within recommended hold time of 1 year for EPA 1614.

QA Review
Dataset completeness

The dataset included resutls for 10 field samples (including one lab replicate), and a blank, CRM, matrix spikes, and an LCS sample, reported for 27 BDEs, and moisture.

Percent usable (non-reject) field data
83% of the results were reportable, with the rejected results being blank contamination greater than a third of the field sample results, as well as one lab rep with extraction problems for all PBDEs.

Overall acceptability
Overall the data are acceptable, although they showed some inconsistency in blank contamination, recovery, and precision.

MDLs sensitivity
MDLs were sufficient for most of the dominant congeners, but about half the target analytes (12 of 27 BDEs) were not detected in all samples.

QB averages (procedural, field blank)
BDEs 17, 28, and 47 were found in blanks, with 17 and 28 sometimes constituting ⅓ or more of the field sample result, and censored (VRIP flag) as a result.

Average precision from replicate field sample
Precision on the lab replicate was higher than desirable (RSD > target of 35% and sometimes over 50%), so results on those congeners were flagged (VIL) but not censored. This was likely in part due to an analytical problem in the extraction.

Accuracy (using a variety of SRMs or Matrix spike QRECs)
Recoveries on matrix spikes for some of the major congeners also deviated >35% from their target values, so we flagged (VIU) but not censored.

Comparison of dissolved and total phases
Not applicable.

Comparison to previous years
Results were in a similar range as previous years, and. Mean concentration of BDE 47 was around 15 ng/g ww vs around 21 for 2012, and averaging 6 and 7 ng/g ww for BDE 99 and 100, vs 9 and 12 ng/g ww for 2012.

Ratio Checking Summary

The following email thread documents issues raised and resolved during the ratio review.

> 4/11/2017 email from Rebecca Sutton
> Hi all - I've reviewed the egg dataset. The values generally make sense (see below). However, there are a bunch of flags I don't normally see so broadly on this type of dataset, mostly RPD exceeding control limits (IL/VIL). Don, based on your more technical review, do we need to be concerned?
>
> Meanwhile, here is a draft summary:
> 2016 bird egg dataset: Acceptable

Blank contamination noted, but not at levels of concern for dominant congeners. Excellent lab duplicate agreement.
Two highest concentrations subjected to secondary dilution for analysis. VIL flags indicating RPD exceeds control limit for dominant congeners.

Overall congener fingerprint comparable to 2012 cormorant egg data. Outlier ratios (2stdev) only noted for the most and least contaminated sample. Median PBDE sum ~1/2 of 2012 levels (21 vs 41); overall range broader (107-9 vs 83-18). Higher levels in 2016 consistently observed at Richmond Bridge site. (All comparisons made based on wet weight, not lipid weight!)

4/11/2017 email from Don Yee

Hi Becky,
The lab is no longer analyzing tissue for anyone (or at least not outside clients) and they didn't have enough material left to reanalyze a lab rep when somehow they messed up the extraction or final blowdown on the one lab rep.
So the RPD is not what we would want to see for sure. RPD on site reps is not great although it's pretty variable in general since each is a composite of 7 eggs and individual variance is luck of the draw which egg ends up in which composite. Not the labs fault entirely, likely natural variation, but inseparable since we have no separate evaluation of lab reps.
My inclination is leave them flagged as is. Jay was suggesting going into the archives to get a lab rep but I'm not sure the value of that (vs using it for future synoptic trend analysis, or CECs or whatever) especially since the lab is shutting down to outside work at least.
Don

4/11/2017 email from Rebecca Sutton

Hey Don - Thanks for the context! I had forgotten the lab shut-down thing (I had thought I was looking at AXYS data).
I'm fine with current flags and data as-is.
John, am I done now?

Rebecca,
Yes this is now completed.
John