# ACLASS Practice Assessment Lab Comparison Summaries for PREPA – June, 2012

SUMMARY --

Four laboratories in Puerto Rico, all related to fuel oil testing arranged by the Puerto Rico Electric Power Authority (PREPA), were contracted by PREPA to perform Practice Assessments by ACLASS in early June 2012. There were at least 3 key objectives in the contract. First, we were asked to perform at each lab a 2.5-day practice assessment to ISO 17025, related to a submitted identical accreditation scope of 13 fuel oil tests. Second, we were asked to report on the relative readiness of each of the four laboratories for accreditation to the ISO 17025 standard for this testing, including a report to PREPA only of those assessments. (This report represents that summary and comparison.) Third, we were asked to coordinate a Proficiency Testing comparison of the technical capabilities of the 4 labs, in comparison with an identified US accredited testing laboratory for most or all of those 13 scope line item tests. (The full data submissions from that study are not yet in place, so that third section is excluded from this summary.)

The Practice Assessments were all performed during the week of June 4th to 8th, 2012 by Dr. Bill Hirt, the Director of Accreditation for ACLASS, and Michelle Negreros, a contract chemical assessor for ACLASS. Each assessor performed two assessments, singly, that week. One handled Altol and Prepa labs. The other handled Saybolt and Inspectorate labs. There was at least one or two PREPA representatives attending each of the four Practice Assessments, in addition to the ACLASS assessor. In fact, these representatives hand-carried a fuel oil sample to each assessment. It had been collected, composited and sub-divided into aliquots by PREPA staff prior to the assessments. It was designed and agreed by ACLASS and PREPA to use these aliquots as the primary sample to use for analysis during the assessment and method witnessing. It was also designed and agreed that each of the four labs would be asked to sample the large, local fuel oil storage tanks to create a second sample. Each of their two samples would then be analyzed per the drafted accreditation scope, and the data submitted as part of the PT comparison study (which was then outlined to each of the labs at their opening meeting for the assessment.)

PREPA actually arranged for an addition to the typical ISO 17025 Practice Assessments, where ACLASS uses its Form 1 checklist to cover all the specific requirement elements from the international standard. They submitted to ACLASS the month before the visit an additional 10 questions or issues to be evaluated. The official notes for these added elements are seen in table form below for each laboratory. These notes are referred to as "PREPA PA Extra Checklist" results. In addition, from the four labs, all ISO 17025-related assessment findings were drafted and reviewed with each lab respectively at a closing meeting as each assessment completed.

To summarize our observations and conclusions, we found that, of the four laboratories, Altol Enterprises was the most familiar with ISO 17025 and was found to hold accreditation for its environmental testing services, but not for its fuel oil testing. None of the other three laboratories were accredited to ISO 17025. All seemed to be very familiar with or certified to ISO 9001 for their management systems. Not all of the laboratories however even had a management system that included a formal Quality Manual. Despite the familiarity with ISO 17025, the conclusion was made that Altol was not the one closest to readiness for ISO 17025 accreditation for the fuel oil testing. That determination was made for Inspectorate Laboratory. As a final conclusion, we were impressed with the commitment and effort by PREPA to undertake this study and project. We hope it is not seen as anything but relative judgment, but it seemed that the internal PREPA lab, of the four that we assessed, was the farthest from readiness for ISO 17025 accreditation. It was also the laboratory that was not currently equipped for many of the fuel oil tests on the scope of accreditation as

submitted. This underscores why PREPA used a combination of the other laboratories to subcontract many of those fuel oil tests.

We refer readers of this report to follow the summary notes here and compare in addition the attachments at the end of the report. This includes the summary paragraph of each assessment visit and the Extra Checklist notes plus the findings drafted from each visit. We would include notes now, in sequence of relative readiness for ISO 17025 accreditation, for each of the labs, starting with the most ready.

The Inspectorate Laboratories assessment was a bit unusual since the lab director was away unavoidably, and in his place we found a representative from the regional and from the corporate headquarters for the laboratory as hosts. Interestingly, this difficulty highlighted a great strength and support of the laboratory to understand their appreciation of the ISO standard, technical issues and support to perform gap analysis and preparedness for accreditation. When we reviewed the extra checklist elements, we found most of them scored as excellent status or close to readiness. Their number and severity of findings was the smallest of the 4 laboratories. They had only 4 major findings, 4 minors and 2 OFIs. We had a good sense that they may be ready for accreditation within a few short months.

The Altol Laboratory was ranked as second-most ready for accreditation. They were much farther ahead in their environmental program than their fuel oil testing program but still very competent. Their management system documentation did not include the fuel oil program, but hopefully could reasonably be expanded to do so. Some of their standards including chemical and physical standards were not found to be sourced from accredited organizations, but again, this can be remedied, and their understanding from their environmental operations should serve them well in the expansion of the fuel oil testing. The Altol lab had 6 majors, 11 minors, and 5 OFIs as findings. This was more than 50% more than Inspectorate.

Saybolt Laboratories was ranked third. This laboratory seemed to have a corporate support structure, but it did not seem to be as knowledgeable or attentive to laboratory support as Inspectorate. The lab equipment was older and less well maintained. In fact, for the sediment test method that required temperature controlled centrifugation of the fuel oil, the centrifuge in use was very old, loud and vibrating and had no temperature control at all. We considered most of their methods as adequate with reasonable QA and QC, but it would need more improvement to have the quality assurance and confidence comparable to the two subcontracted labs. Officially we wrote 9 major findings and 5 minors for Saybolt.

Lastly, the PREPA laboratory seemed to rely very heavily on its subcontracted laboratories for internationally verifiable competence in this fuel oil testing work. They had not seemed to have made a formal commitment, outside of this project, to have their management system ready for international recognition and accreditation. Prepa did not have a quality manual or formal set of second level documents, which we often call SOPs for many of its management system areas. The technical staff had a very good competence technically but were not aware of the international standard or many of the related quality management concerns in an accredited lab. At the assessment we found 19 major findings and 8 minors. This lab would clearly require more training and development than the other three labs in this study. These issues could all be managed and overcome of course, and we have seen it with many organizations that are now accredited.

The other layer of comparison and verification of competence would involve the review of each of the laboratories' proficiency testing results in the study here. Unfortunately this report is not yet complete. It will be completed and sent to PREPA as soon as possible. These results will hopefully add to the confidence in the use of these labs, unless there were any spurious results found.

We greatly appreciated the opportunity to work with PREPA on this undertaking and look forward to working with them in the future for accreditation services and training.

*The formally filed Practice Assessment Visit summaries are attached below here for each of the labs. These represent the summaries filed in our EQM location for each laboratory. →*

The PREPA laboratory Practice Assessment took place for 2.5 days from June 6th to 8th with a partial review of the requested PREPA scope of accreditation. Sampling was considered a method in the scope. Only six of the requested scope items were offered by PREPA lab, and five of these methods were witnessed during this practice assessment: 1- Sampling ASTM D4057, 2- Gravity, API Degree ASTM D-52002, 3- Viscosity ASTM D-88, 4- Sulfur ASTM D4294, 6- Asphaltenes ASTM D6560, 7- Vanadium ASTM D-1548. As a result, the findings drafted at the visit were shared with PREPA, but no corrective actions were warranted or expected. A report of findings was handed out to the management of PREPA during the closing meeting. Each finding was categorized as an opportunity for improvement and there was a note indicating, when relevant, that if it was an actual accreditation assessment, which kind of non conformance it would be (major or minor). In total, 26 findings were written for the practice assessment. All findings are filed officially as OFI's so the CAR process would not be required by EQM. The primary purpose of the visit was to assess, for PREPA, the gap analysis or degree of readiness of each of 3 of PREPA's Puerto Rican subcontracted test laboratories for their #6 Fuel Oil testing for ISO 17025 laboratory accreditation. PREPA stated they intend for their internal laboratory to seek such accreditation and may require accreditation as well for their subcontracted labs in the near future. They want to be familiar with their relative readiness. The activity this week would also include a comparative Fuel Oil PT study that ACLASS would organize. The PREPA lab showed good technical competence, but the quality system under the ISO 17025 standard was not yet established nor implemented in the laboratory. This positions the laboratory in a relative disadvantage compared with its subcontracted test laboratories.

The Inspectorate laboratory Practice Assessment took place for 2.5 days from June 6th to 8th with a full review of the requested Prepa scope of accreditation. All of the requested scope items were offered by Inspectorate and witnessed. The arrangement for the visit was by Prepa and not Inspectorate. As a result, the findings drafted at the visit were shared with Inspectorate, but no corrective actions were warranted or expected. the report per se will only be accessible by Prepa and not by Inspectorate. This visit was also unique because the lab manager was not present. He had to be gone on vacation. As a result, the regional manager and the corporate QA manager (from Houston) were there for assistance. The primary purpose of the visit was to assess, for Prepa, the gap analysis or degree of readiness of each of 3 of Prepa's Puerto Rican subcontracted test laboratories for their #6 Fuel Oil testing . . . for ISO 17025 laboratory accreditation. Prepa stated they intend for their internal laboratory to seek such accreditation and may require accreditation as well for their subcontracted labs in the near future. They want to be familiar with their relative readiness. The activity this week would also include a comparative Fuel Oil PT study that ACLASS would organize. The Inspectorate lab showed good technical competence and impressive management system resources to have very modest needs for readiness for accreditation. It was clear, however, that their documents and training were not yet directly addressing ISO 17025 elements, but with the regional and corporate representatives on site this visit demonstrated good awareness of those capabilities and the ability to implement them in a very short timeframe. Roughly a dozen findings were written. In this case, they may have wording to show they would be written as majors or minors in a real assessment, but here all were filed officially as OFI's so the CAR process would not be required by EQM of ACLASS.

The Saybolt laboratory Practice Assessment took place for 2.5 days from June 4th to 6th with a full review of the requested Prepa scope of accreditation. All of the requested scope items were offered by Saybolt and witnessed. The arrangement for the visit was by Prepa and not Saybolt. As a result, the findings drafted at the visit were shared with Saybolt, but no corrective actions were warranted or expected. The report per se will only be accessible by Prepa and not by Saybolt. The primary purpose of the visit was to assess, for Prepa, the gap analysis or degree of readiness of each of 3 of Prepa's Puerto Rican subcontracted test laboratories for their #6 Fuel Oil testing . . . for ISO 17025 laboratory accreditation. Prepa stated they intend for their internal laboratory to seek such accreditation and may require accreditation as well for their subcontracted labs in the near future. They want to be familiar with their relative readiness. The activity this week would also include a comparative Fuel Oil PT study that ACLASS would organize. The Saybolt lab showed good technical competence and management system resources to have relatively modest needs for readiness for accreditation. It was clear, however, that their documents and training were not yet directly addressing ISO 17025 elements, so this is required. Roughly a dozen findings were written. In this case, they may have wording to show they would be written as majors or minors in a real assessment, but here all are filed officially as OFI's so the CAR process would not be required by EQM.

The ALTOL laboratory Practice Assessment took place for 2.5 days from June 4th to 6th with a full review of the requested Prepa scope of accreditation. All of the requested scope items were offered by ALTOL, seven methods out of thirteen were witnessed during this practice assessment: 1- Sampling: ASTM D-4057, 2- Gravity, API Degree at 60oF ASTM D287-92 (2006), 3- Viscosity ASTM: D-445, 4- Sulfur ASTM D4294-10, 5- Ash ASTM D-482, 6- Asphaltenes ASTM: D3279-07, 7- Vanadium ASTM D-5863-B. For the rest, a visual inspection of the equipment was made. The arrangement for the visit was by PREPA and not ALTOL. As a result, the findings drafted at the visit were shared with Altol, but no corrective actions were warranted or expected. The report per se will only be accessible by Prepa and not by ALTOL. A report of findings was handed out to the management of ALTOL during the closing meeting. Each finding was categorized as an opportunity for improvement and there is a note indicating, when relevant, that if it was an actual accreditation assessment, which kind of non-conformance it would be (major or minor). In total, 22 findings were written for the practice assessment. All findings are filed officially as OFI's so the CAR process would not be required by EQM. The primary purpose of the visit was to assess, for PREPA, the gap analysis or degree of readiness of each of 3 of Prepa's Puerto Rican subcontracted test laboratories for their #6 Fuel Oil testing for ISO 17025 laboratory accreditation. PREPA stated they intend for their internal laboratory to seek such accreditation and may require accreditation as well for their subcontracted labs in the near future. They want to be familiar with their relative readiness. The activity this week would also include a comparative Fuel Oil PT study that ACLASS would organize. The ALTOL lab showed good technical competence and impressive management system resources to have very modest needs for readiness for accreditation. ALTOL lab is managing an integrated system, having been working on ISO 9001 from the year 2009 and being accredited on ISO 17025 for their environmental tests. However, it is clear that the system has not covered yet the Oil testing area, but the commitment of the management to do so is very evident, so they will need to inject the resources needed.

*Below here are the individual laboratory Extra Checklist notes as recorded at the Practice Assessments and the respective finding in a table. Note that, at the PA visits and closing meeting, it was explained that the official findings were all recorded as Opportunities for Improvement or OFIs. This was to avoid difficulties with the database system for ACLASS to attempt to manage resolution of all findings from a normal assessment. We took the liberty here, however, to put minor and Major finding designations on each of the finding to help understand the significance of corrective actions needed for accreditation readiness.*

PREPA Practice Assessment Extra Checklist -- Lab Assessed -- INSPECTORATE Date - June 8th, 2012

| Assessment Check Item | Assessed (Y/N) ? | Comments / Evaluation |
|---|---|---|
| 1. Interpretation of bias in sulfur method ASTM D4294 | Y | No attention paid to ASTM repeatability or bias studies. Worry about strictness of EPA standard and limit, overly strict implementation, but limited awareness of uncertainty or bias in their measurements. |
| 2. If other accreditations or certifications are presented by labs being audited, to ensure that they apply to satellite facilities, like here in Puerto Rico | Y | No connection by Inspectorate to sister labs for subcontracting or calibration services, but excellent corporate and regional management of equipment resources. High commitment to upgrade any as needed. Strong commitment and attention already to resolve traceability and uncertainty shortcomings. Corporate QA in Houston has good 17025 awareness but not yet commitment. |
| 3. Proper handling and date validations for reference standards and written certificates (not expired) | Y | Chemical traceability excellent. NIST and ASTM. No specific awareness or attention to ISO guide 34 traceabilty but could train local management and accomplish quickly. |
| 4. Checks for validity of calibration standards or certified test samples (i.e., fidelity plots) | Y | Excellent SPC control chart plots maintained daily and posted on lab wall near equipment. |
| 5. Proper use of calibration curves (not expired) | Y | Strong and strict attention to QA limits and plotting of QA/ QC samples. No tolerance for expired curves. |
| 6. Existence of Quality Control Procedures, Quality Management Manuals and lab SOP's | Y | ISO 9001-based documentation in place. Mention 17025 in QManual but not diligently followed as yet. Lab SOP's for PREPA in place. |
| 7. LIMS and chain of custody procedures | Y | Chain of custody forms and process in place. Good records. Excellent LIMS with bar coding and sample and QC sample processing. |
| 8. Recommended interpretation of blanks or LCS samples used during consecutive sample runs | Y | Additional layers of QC from corporate and regional management. Excellent adherence to QC. No large sample processing to note its use. |
| 9. Calculations software | Y | Analyzer software in place, e.g. PerkinElmer for AA excellent. At accreditation time, we would assure each verified by supervisor for use and function. |
| 10. Precision and Bias | Y | Available in published methods, but no particular application made of it. Precision only for repeatability as needed. Tracked by regional and corporate management. No local understanding noted as yet of 17025 traceability or uncertainty, but corporate support could overcome this. |
| | | |

# NC's found, though officially all OFI's in the draft report for EQM

| NC # | Element # | minor/Maj | Finding (INSPECTORATE) |
|---|---|---|---|
| 1 | 4.12.1 | Minor | System listed in quality manual but no example found with CAR-type form and root cause. |
| 2 | 4.14.1 | Major | Internal audit not outlined yet to cover ISO 17025 elements, traceabilility, all technical methods, reference materials etc. (major) |
| 3 | 4.15.1 | OFI | Suggest lab here in PR establish semi-annual or annual review of improvement activities measures and issues. Monthly BMI reviews very valuable but give a very different perspective. |
| 4 | 4.3.1 | Minor | Procedures are mature for corporate documents but not defined for local forms |
| 5 | 4.3.2.1 | Major | The Master document list provided showed some corporate documents and others obsoleted, but it was not clear what the official local quality manual would include. The system also has QPs and SOPs. Forms were found often uncontrolled and kept in a hard copy binder with no master |
| 6 | 4.6.4 | Minor | The vendor list was not in place as a list, only pages in a binder, and several vendors were at least slightly overdue for evaluation. |
| 7 | 5.3.1 | OFI | Suggest lab acquire programmable or more accurate settings for oven temperatures |
| 8 | 5.4.6.1 | Major | No uncertainty procedure was found for the methods as yet. Lab does not have yet any accreditation body guidance or practice for uncertainties either. We acknowledge that, due to our schedule, the lab manager was not present for this Practice Assessment. He may understand better than others here. |
| 9 | 5.5.10 | Minor | No intermediate checks done to top loader balance, also only 1 weight used on analytical balance. |
| 10 | 5.6.2.1.1 | Major | Only one chemical traceabilty provider and one mechanical calibration provider were found accredited to ISO17025. JR Biomedical calibrated balances, most thermometers and other devices but had deficient certificates and no accreditation. |
| | | | |

PREPA Practice Assessment Extra Checklist -- Laboratory Assessed -- ALTOL date assessed -- June, 4 – 6, 2012

| Assessment Check Item | Assessed (Y/N) ? | Comments / Evaluation |
|---|---|---|
| 1. Interpretation of bias in sulfur method ASTM D4294 | N | |
| 2. If other accreditations or certifications are presented by labs being audited, to ensure that they apply to satellite facilities, like here in Puerto Rico | Y | The laboratory has accredited methods in the environmental area under the ISO 17025 standard.<br>The laboratory is certified ISO 9001.<br>The laboratory does not have satellite facilities. |
| 3. Proper handling and date validations for reference standards and written certificates (not expired) | Y | Sampling: the master tape used to verify the metric tape is verified every five years by an external supplier. Calibration laboratory is not an accredited one.<br>Sulfur: the checking standard used was found to be out the time frame recommended by the manufacturer. The manufacturer was not an accredited reference material producer.<br>Vanadium: the reference material is from an accredited manufacturer.<br>Viscosity: thermometer used to verify temperature is calibrated every six months. Calibration laboratory not accredited.<br>API: hydrometers used are calibrated every year.<br>Weight set: calibrated every year. Calibration laboratory is not an accredited one. |
| 4. Checks for validity of calibration standards or certified test samples (i.e., fidelity plots) | Y | See comments in issue #3. |
| 5. Proper use of calibration curves (not expired) | Y | Sulfur analysis: a curve built in March 2011 was found to be in use.<br>Vanadium analysis: a fresh curve was prepared for the analysis. |
| 6. Existence of Quality Control Procedures, Quality Management Manuals and lab SOP's | Y | There is a QM that is not yet covering the fuel testing activities. It is oriented to the environmental analyses.<br>There is no QA/QC procedure in place for the fuel testing activities.<br>The laboratory uses the ASTM documents as they are, the laboratory does not have its own SOP's for these activities. |
| 7. LIMS and chain of custody procedures | N | |
| 8. Recommended interpretation of blanks used during consecutive sample runs | Y | Except for the vanadium calibration curve, that includes a blank as the 0 of the curve, no blank are used. |
| 9. Calculations software | Y | Calculations are done with a manual calculator for API, viscosity, ash. The equipments do calculations for sulfur and vanadium. |
| 10. Precision and Bias | N | |

# NC's found, though officially all OFI's in the draft report for EQM

| NC # | Element # | minor/Maj | Finding (ALTOL) |
|---|---|---|---|
| 1 | 4.11.1 | OFI | The information recorded in the corrective preventive action form does not have all the details needed to evidence the follow up activities carried out to evaluate effectiveness of the actions. |
| 2 | 4.13.2.2 | Minor | The technician identified a reading as non appropriate, this reading and the explanation of why was it not appropriate was not written in the log book. |
| 3 | 4.13.2.2 | Minor | The technician wrote the readings of the hydrometer for the API test in his glove, then it was written in the log book. |
| 4 | 4.14.1 | OFI | It was not easy to identify the links among the records for the components of the audit report: checklists, and corrective/preventive actions. Moreover, it was not easy to relate non conformance in form F-12 to the findings in the checklist, it took some time from the quality manager to do so. |
| 5 | 4.14.1 | Minor | Records for the internal audits do not show coverage of the fuel testing activities. |
| 6 | 4.14.1 | Major | The checklist used only includes the elements of the section 5 of the standard. |
| 7 | 4.14.1 | Minor | Lead auditor does not have the training for ISO 17025. However, he has been trained for ISO 9001 and there is another auditor with the ISO 17025 training. |
| 8 | 4.15.2 | Major | Records of the management reviews do not show discussion on issues related to the fuel testing area |
| 9 | 4.6.2 | Minor | The acetylene used for the vanadium test was not AA quality. The documentation for the purchase showed that such specification was requested. A signature in the reception of the product indicates compliance, but the information in the actual cylinder could not prove so |
| 10 | 5.10.3 | Major | The test report does not have all the elements required by this standard. There is no title and the procedure used for sampling is not included. There is no indication of what is the end of the report and there are more documents as part of it. |
| 11 | 5.10.3.1c | OFI | The uncertainty is not expressed in the report, even though, the result is going to be compared with a specifications due to local regulation. |
| 12 | 5.2.1 | Minor | The competence assurance program has not been applied to the fuel testing personnel. |
| 13 | 5.4.1 | OFI | It is not easy for the personnel to find the documents for the activities or procedures at the side of the main activity. They may be in other documents or not being documented at all, but they do not know immediately. |
| 14 | 5.4.6.3 a12 | Major | The current uncertainty procedure has not applied to the fuel testing area.<br>No uncertainty budgets have been generated for such tests. |
| 15 | 5.5.10 | Major | The certified viscosity reference standard in use in the laboratory (lot 09301) to verify the viscometer shows "use before: 12 31 2011". |
| 16 | 5.5.10 | Minor | There is no defined procedures for the verification of equipment. Examples seen: balances, viscometers. NOTE: in an actual accreditation assessment it would be a minor non conformance. |
| 17 | 5.5.11 | OFI | There are no instructions for the use of correction factors. There is confusion in the concept of what a correction factor is and the applications that it involves; but more important, the impact that it may have not to apply them. |
| 18 | 5.5.2 | Minor | The laboratory verifies the competence of the calibration provider only by checking the traceability of the standards used and the credentials of the person that performs the calibration. See 5.6.2.1.1 in the standard ISO 17025 for the definition of a competent calibration laboratory.<br>It is not enough to assure that the calibration laboratory is competent.<br>NOTE: if it was an actual accreditation assessment, it would be a minor non conformance. |
| 19 | 5.6.1 | Minor | The criteria to accept or reject a calibration curve in the test for vanadium are not properly defined. Only the correlation factor of the curve was used. However, the method also gives a criteria for the use of a checking standard and this was not used accordingly by the technician.<br>Note: if this was an actual accreditation assessment, this would be a minor non conformance. |
| 20 | 5.6.1 | Major | The calibration curve for sulfur analysis was built on March 2011. No documented reference was found to suggest that such a curve was valid after such a long time.<br>Note: if this was an actual accreditation assessment, this would be a major non conformance. |
| 21 | 5.6.3.2 | Minor | The standard used for verifying the calibration curve in the sulfur test was found to be out of the time frame indicated by the manufacturer of the material. It says that the product is stable one year after the date it is opened and was opened, (assumed because there is no record) on March 2011, when the curve was built. The technician explained that the product was stable, but there is no evidence that the material was checked to demonstrate that its properties are still as originally.<br>NOTE: if it was an actual accreditation assessment, it would be a minor non conformance. |
| 22 | 5.9.1 | Major | In general, there is no QA program established in the laboratory for the tests in the scope. There are some tools being used, such as reference materials for checking a calibration curve in sulfur.<br>Note: in an actual accreditation assessment, this would be a major non conformance. |
| | | | |

PREPA Practice Assessment Extra Checklist -- Lab -- SAYBOLT Date Assessed -- June 6th, 2012

| Assessment Check Item | Assessed (Y/N) ? | Comments / Evaluation |
|---|---|---|
| 1. Interpretation of bias in sulfur method ASTM D4294 | Y | No attention paid to ASTM repeatability or bias studies. Worry about strictness of EPA standard and limit, overly strict implementation, but limited awareness of uncertainty or bias in their measurements. |
| 2. If other accreditations or certifications are presented by labs being audited, to ensure that they apply to satellite facilities, like here in Puerto Rico | Y | No connection by Saybolt to sister labs for subcontracting or calibration services. Parent provides very helpful admin support but not 17025-related services. |
| 3. Proper handling and date validations for reference standards and written certificates (not expired) | Y | Chemical traceability a problem. No specific awareness or attention to ISO guide 34 or NIST traceabilty consistently. Some ASTM standards well in place. |
| 4. Checks for validity of calibration standards or certified test samples (i.e., fidelity plots) | Y | Not fidelity plots but run unexpired standards with appropriate frequency in analysis. |
| 5. Proper use of calibration curves (not expired) | Y | Seen as adequate. Example seen was annual creation and use of vanadium curve per uv spectrophotometer. |
| 6. Existence of Quality Control Procedures, Quality Management Manuals and lab SOP's | Y | ISO 9001-based documentation in place. Mention 17025 once in QManual but not again. Lab SOP's for PREPA in place. |
| 7. LIMS and chain of custody procedures | Y | Chain of custody forms and process in place. Good records. |
| 8. Recommended interpretation of blanks or LCS samples used during consecutive sample runs | Y | Reasonable use at this witnessing. No large sample processing to note its use. |
| 9. Calculations software | Y | Examples used as needed for methods. At accreditation time, we would assure each verified by supervisor for use and function. |
| 10. Precision and Bias | Not directly | Calculated as needed for methods, but no particular application made of it. Precision only for repeatability as needed. |
| | | |

# NC's found, though officially all OFI's in the draft report for EQM

| NC # | Element # | minor/Maj | Finding (SAYBOLT) |
|---|---|---|---|
| 1 | 4.10 | Minor | No formal improvement targets were found in the system |
| 2 | 4.12.1 | Major | No Preventive Actions were found in the system. This is a major finding, though not a critical issue. |
| 3 | 4.14.1 | Major | Internal audits were performed with corporate support, but not all ISO 17025 elements were tracked |
| 4 | 4.15.1 | Major | Management review was done out of corporate. Not meeting all the 17025 elements. No local management review done, though corporate does send a summary of PT issues, internal audit summary and other data. |
| 5 | 4.2.3 | Major | Dozens of requirements for ISO 17025 were not defined in the Quality documents of Saybolt or verified as yet in the system. This includes management review, internal auditing, subcontracting, purchasing, vendor approvals, complaints, corrective actions, improvement activities, non-conforming work, and other elements. Corporate support out of Houston, TX has high quality services including internal audit visits and reports and summary of some metrics. They are simply not yet directly addressing all the ISO standard requirements to assure compliance. |
| 6 | 4.5.1 | Major | Would be Major finding. Subcontracted laboratories for potentially accredited work are not accredited or verified for full 17025 compliance |
| 7 | 4.5.4 | Minor | JCL is the primary ISO 17025 accredited lab used to calibrate hydrometers and thermometers, but the scope retained in the file for them is expired 3 years. |
| 8 | 4.7.2 | Minor | No system was acknowledged in the document system for customer feedback. Lab only uses compliments and complaints tracking. This would be at least a minor finding |
| 9 | 5.10.8 | Minor | The Lab Template for #6 Fuel Oil reporting form was used in the laboratory as the reporting template for those samples. Multiple forms were seen not following the instruction that was very prominent on the form to Cross Out tests NOT requested. Instead the lab practice was to simply check on the forms those tests that WLRL requested. None were crossed out. |
| 10 | 5.4.1 | Major | Method ASTM D1548 for ashing samples prior to vanadium analysis requires careful attention to assuring the ashing temperature never exceeds 550 F. During the method witnessing, technicians set the temperature to 550 F instead of the recommended 525 F in the method. Inspection of all prior calibrations of the ashing furnace showed 10 to 20 F increases in temperature over the year for each set temperature. In addition the API method ASTM D287 requests temperature be extrapolated from the temperature to 0.25 F but all temperatures recorded in the log have been 0.5 F which is the resolution of the thermometers. |
| 11 | 5.4.1 | Major | The centrifuge step in ASTM D1796 requires tight temperature control, but the age and condition of the lab centrifuge used did not provide this control. |
| 12 | 5.4.6.2 | Major | No formal procedure was found in place for managing uncertainties for the accredited scope (major) |
| 13 | 5.5.2 | Minor | The primary reference thermometer in the lab was found to be a wide range liquid-in-glass thermometer with 1.0 F accuracy / readability. It was reportedly used to verify many other temperature devices in the lab. One in particular noted a 0.1 F error on its attached label. The accuracy of the reference thermometer could not explain this extra accuracy of error. |
| 14 | 5.6.2.1.1 | Major | The calibration provider for balances and a few of the laboratory thermometers, Precision Control, was not accredited and provided no reliable uncertainties on their certificates. |
| | | | |

PREPA Practice Assessment Extra Checklist -- Laboratory Assessed -- PREPA -- Date Assessed -- June 6-8, 2012

| Assessment Check Item | Assessed (Y/N) ? | Comments / Evaluation |
|---|---|---|
| 1. Interpretation of bias in sulfur method ASTM D4294 | N | |
| 2. If other accreditations or certifications are presented by labs being audited, to ensure that they apply to satellite facilities, like here in Puerto Rico | Y | There are no other accreditations or certifications. There is an agreement with EPA.<br>The laboratory does not have satellite facilities. |
| 3. Proper handling and date validations for reference standards and written certificates (not expired) | Y | Sampling: metric tape used is not verified.<br>Sulfur: checking standard used to verify the calibration curve. Control charts not updated. Criteria for accepting or not the results are not well defined.<br>Vanadium: no checking standard used to verify the calibration curve. However, the standard to do so is in the lab.<br>Viscosity: Orifices of viscometer calibrated every 6 months according to ASTM D88-07. Correction factors are updated then.<br>API: Verification is with demineralized and boiled water. No control chart or info about the water quality or characteristics.<br>Weight set: Balance verification only with 100g weight. A correction is made using a table OIML R111 which is for weight calibration.<br>In general: The basic quality control is a duplicate every 20 samples. The calculations are not done every time the duplicate is done. No control chart. No control of nonconforming work. |
| 4. Checks for validity of calibration standards or certified test samples (i.e., fidelity plots) | Y | See comments in issue #3. |
| 5. Proper use of calibration curves (not expired) | Y | Sulfur analysis: a fresh curve is prepared every six months.<br>Vanadium analysis: a fresh curve is prepared every six months. No checking standard is used. |
| 6. Existence of Quality Control Procedures, Quality Management Manuals and lab SOP's | Y | There is no QM.<br>There is no complete QA/QC procedure in place.<br>The laboratory uses its own SOP based on the ASTM documents. |
| 7. LIMS and chain of custody procedures | N | |
| 8. Recommended interpretation of blanks used during consecutive sample runs | Y | Except for the vanadium analysis, no blank are used. |
| 9. Calculations software | Y | Calculations are done with a manual calculator, except for the case of sulfur and API degrees, for the equipments do the calculations. |
| 10. Precision and Bias | N | |

# NC's found, though officially all OFI's in the draft report for EQM

| NC # | Element # | minor/Maj | Finding (PREPA) |
|---|---|---|---|
| 1 | 4.13.2.2 | Minor | The calculations for the height/depth of the spots where the fuel oil sample is to be taken are not written in the log book right away, instead, on a piece of paper. |
| 2 | 4.14.1 | Major | There was no procedure, no schedule and no trained auditors to perform internal audits to ISO 17025 at the lab. |
| 3 | 4.14.1 | Major | The internal audits at the lab were not found to cover all elements of ISO 17025 |
| 4 | 4.2.1 | Major | There is no quality system established as yet for the lab. The 17025 standard has not been introduced in the activities of the lab. |
| 5 | 4.2.2 | Major | There is no quality manual that describes the quality system for the lab. |
| 6 | 4.3.2.1 | Major | There is no master document list used in the lab |
| 7 | 4.9.1 | Minor | The protocol for results out of limits does not cover other types of non-conforming work during the testing process. |
| 8 | 5.10.2c | Minor | The report format does not display page numbers and total pages. |
| 9 | 5.10.2h | Minor | Sampling plan or sampling is not indicated on the report. Moreover, the scope indicates sampling as a test method. |
| 10 | 5.10.2k | Major | The report does not include any note that the results relate only to the samples tested. |
| 11 | 5.10.3 | Minor | Compliance with specifications is included in the report, but uncertainty of the measurement is not considered for the comparison. |
| 12 | 5.10.3 | Minor | Information of the sampling done is not included in the report of the results. |
| 13 | 5.2.1 | Major | There is no evidence of current competence assurance and evaluation of technical competence of the personnel who perform the tests. Training is not specific for the technical competence in the fuel testing area. |
| 14 | 5.2.4 | Major | The most recent approved job description for the lab chemists was in October 1997. The last approval of competence was at hiring as part of initial training. |
| 15 | 5.3.1 | Major | According to the chemist, the humidity of the lab is high, and the fog can be noticed on the lab equipment. There are no humidity records found, however, to evaluate it. At this PA, the humidity problem was not observed. |
| 16 | 5.3.1 | Major | The appropriate environmental conditions were not documented and monitored in the lab |
| 17 | 5.4.1 | Minor | The chemist did not transfer the full solution volume to the volumetric flask when washing the sample. It was an inappropriate quantitative transfer. |
| 18 | 5.4.6.2 | Minor | The results of the tests are to be compared with specifications of local regulations, but the results were not accompanied by the measurement uncertainties. |
| 19 | 5.4.6.2 | Major | The lab was not found to have an understanding of ISO 17025 uncertainty. It needs to be reinforced. |
| 20 | 5.4.6.2 | Major | No procedure was found in the lab for measurement uncertainty. The lab has not yet identified the sources of uncertainty nor calculated any values. |
| 21 | 5.5.10 | Major | Equipment is not being verified or is not correctly verified. No verifications were found for the metric tape used to measure the height of the oil sampling, for the 1L volumetric flasks for the samples. The balance verifications used 100 g weight, although the common weights measured were 2 g to 4 g. There was no verification of the calibration curve for vanadium, although there was a reference standard for vanadium in the lab. |
| 22 | 5.5.1 | Major | It was observed that the personnel did not use safety equipment in the laboratory when appropriate including lab coats, goggles or gloves. When handling hot liquids samples and moving them to multiple lab areas, only tongs were used with no base of support. It was reported that individual chemists work alone in the lab (with no safety backup personnel). Strong acids and bases are used with no base of support. |
| 23 | 5.6.3 | Major | The density analyzer used for the API grading test was verified with demineralized and boiling water following the SOP, but no records of this were kept. |
| 24 | 5.8.2 | Major | The sample identification code of the vanadium test was not recorded, nor was the ID of the blank sample. This was also noted for the asphaltene evaporation cups for this test. |
| 25 | 5.9.1 | Major | For every test in Prepa, the corresponding SOP includes the criteria for repeatability and reproducibility as parameters for QC. There is also the use of a standard control in the sulfur test. However, there is no integrated QC program with pre-defined criteria to evaluate the results of the controls and no guidelines for actions to be taken when results are not as expected. |
| 26 | 5.9.1 | Major | The control chart data for sulfur analyses has not been recently updated. The last update record was made in January 2012. The January record also did not include the analyst name or the control used with its properties. Also, duplicates are tested every 20 samples and recorded in the log book, but no evaluation was found to show be done e.g. to look for data trends. |
| 27 | 5.9.2 | Major | Test data was found to be out of control limits but no corrective action evidence was found. For example sulfur data from May 25 and June 7th was out of specification, and the only action taken was to repeat the analysis. |

## DRAFT SCOPE OF TESTING

| | Cost per test / volume needed per test (ml) | Items, materials or products tested | Specific tests or properties measured | Specification, Standard method or technique used | *Detection limit/range/equipment |
|---|---|---|---|---|---|
| 1 | | Fuel Oil No. 6 | Sampling | ASTM D 4057<br>3 levels<br>(UML) composite | Bacon bomb<br>Zone sampler |
| 2 | | Fuel Oil No. 6 | °F, PMCT<br>Flash point | ASTM D 93<br>No lower than 150°F | DL: N/A<br>Range: 40-350°F<br>Eqp: PMA4 |
| 3 | | Fuel Oil No. 6 | Heat of combustion, Btu/lb<br>Btu/gal at 60°F | ASTM D 240<br>No lower than 150.000<br>Btu/gal at 60°F | Eqp: parr 6200 |
| 4 | | Fuel Oil No. 6 | API gravity<br>API degrees at 60°F | ASTM<br>ASTM D 287<br>No lower than 10.5°<br>but no greater than 18° | Range: 9-21<br>Eqp: thermo-hydrometer |
| 5 | | Fuel Oil No. 6 | Viscosity<br>Saybolt at 122°F<br>SFS at 122°F | ASTM D 445 or D88<br>Less than 350 SFS at 122°F | Range: 70-450°F<br>Eqp: Universal Saybolt viscometer |
| 6 | | Fuel Oil No. 6 | Asphaltene content, Ppm by weight<br>% by weight | IP 143 or<br>ASTM D3279<br>No greater than 8% by weight | Det. Limit: 0.50%<br>Range: 0.5%/30.0%<br>Eqp: Analytical Balance |
| 7 | | Fuel Oil No. 6 | Sulfur content<br>% by weight | ASTM D 4294<br>No greater than 1.5% by weight | DI: <5ppm<br>=< 0.0005%<br>Range: 0 – 9.999%<br>Eqp: SLFA 2800 |
| 8 | | Fuel Oil No. 6 | Vanadium content<br>Ppm by weight | ASTM D 1548<br>No greater than 150 ppm by weight | Range 190-110 nm<br>Eqp: Genesys 10 UV-VIS |
| 9 | | Fuel Oil No. 6 | Sodium plus potassium<br>Ppm by weight | ASTM D 1318<br>or IP 288<br>No greater than 25 ppm by weight | As per ASTM<br>(needs verification) |
| 10 | | Fuel Oil No. 6 | Calcium<br>Ppm by weight | IP 288<br>No greater than 10 ppm by weight | As per ASTM<br>(needs verification) |
| 11 | | Fuel Oil No. 6 | Ash<br>% by weight | ASTM D 482<br>No greater than 0.1% by weight | As per ASTM<br>(needs verification) |
| 12 | | Fuel Oil No. 6 | Water and sediment<br>Sediment in crude oil, % by volume | ASTM D 1796<br>ASTM D 473<br>No greater than 1% by volume | Det: 0% vol. eqp; universal centrifuge |
| 13 | | Fuel Oil No. 6 | Pour point<br>Temperature, °F | D97<br>No greater than 75°F | As per ASTM<br>(needs verification) |