



**CHRYSOS
CORPORATION**
Assays at the speed of light

Technical Note TN-401
QAQC Procedures for Chrysos
PhotonAssay™ Systems

Executive Summary

This technical note provides a high-level overview of the procedures used to monitor data quality during routine operation of a Chrysos PhotonAssay™ unit. The differences between PhotonAssay and conventional laboratory techniques are discussed, as PhotonAssay has some distinct QAQC requirements compared to traditional destructive methods such as fire-assay.

The PhotonAssay QAQC procedures include three main components:

- low-level instrument and measurement checks that are performed automatically during normal operation
- regular validation of system performance by periodically running reference samples of accurately known composition
- periodic review of longer-term reference sample results

Whilst the low-level instrument checks run without operator intervention, the preparation and insertion of reference samples requires user intervention. The accuracy and precision of results released by the PhotonAssay™ unit are dependent on consistent application of the recommended QAQC procedures and practices. Appropriate selection, use and ongoing care of monitoring standards will ensure consistent and reliable performance.

Other related publications

TN-403 Chrysos PhotonAssay™ Monitoring Standard Recommendations

TN-001 Chrysos PhotonAssay™ Measurement Performance for Gold on Certified Reference Materials

WI-023 Work Instruction document titled Re-jarring CRMs



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1. Introduction

Quality Assurance and Quality Control (QAQC) is an essential part of any laboratory analysis process, providing confidence that fit-for-purpose analytical results are being produced.

The generally accepted procedure for QAQC involves inserting specially prepared standards with known concentrations (including blanks) into the sample stream of unknown materials to validate the analytical method. Analytical results for the standards are then compared with their accepted grades. Plotting results on a control chart allows biases, trends or unacceptable variances to be readily detected. The inclusion of repeat and duplicate samples provides another important control check on both the analytical method and sampling errors.

PhotonAssay™ is a fully automated and non-destructive technique for the rapid analysis of gold and other elements. Unlike chemical methods such as fire-assay, which rely on the extraction of gold from the sample before it can be measured, PhotonAssay™ directly measures gold atoms inside a bulk ore sample. This can lead to small but systematic differences in the average grades measured using PhotonAssay™ and traditional methods for some standard materials. The instrument precisions of PhotonAssay and fire-assay also vary differently with gold grade. Together, these factors make it important to use method-appropriate mean grade values and control limits for any standards used in a quality control program.

As an automated technique, PhotonAssay™ provides additional layers of internal QAQC that involve more than just quality control checks using standards. First, the unit continuously monitors the quality of the raw data, with the internal software checking that the X-ray source, automation, robotics and detector systems are running correctly. Running a reference disc with every sample allows the detection and correction of most machine issues that might occur on a cycle-by-cycle basis. Second, the software continuously checks the monitoring standards and blanks that are run as part of the sample stream feeding the unit. These checks are part of an automated process that detects problems and flags batches for human review if required. This review by Chrycos QAQC staff forms the third and final layer of QAQC.

2. Monitoring standards and certified reference materials

A common type of monitoring standard used for QAQC purposes are certified reference materials (CRMs). CRMs are materials that have been carefully prepared by a commercial manufacturer to be highly homogeneous or uniform. The grades and associated uncertainty range of key elements are certified by the manufacturer for specific analytical methods such as fire assay or PhotonAssay™. These grades are typically determined through a round-robin, where the samples are sent to multiple reputable laboratories and results statistically combined.

If a material does not have a certified value for the analytical method of choice, then a known concentration can be determined by repeated analysis of the material by the laboratory using that analytical method. The variance or standard deviation in the measured values can also be determined at the same time, providing input into the setting of control limits for that material.

PhotonAssay™ and fire-assay are different analytical methods. PhotonAssay™ is intrinsically insensitive to material type, so matrix and composition does not impact the measured gold grade, whereas fire-assay is a matrix-dependent method for gold analysis. For example, materials that are particularly refractory may have less than 100% gold recovery via fire-assay, whereas analysis via PhotonAssay™ is unaffected. Conversely, materials with high uranium



or thorium content (background elements for PhotonAssay™) may show higher grade variance than when measured via fire-assay.

Importantly, extensive testing of the PhotonAssay™ technology on numerous CRMs that have been certified via fire-assay (more than 200 to date from multiple manufacturers) has demonstrated that PhotonAssay™ and fire-assay grades agree closely and show no systematic normalisation differences versus grade (see Figure 1; refer also to technical note TN-001 for further details). To ensure consistency with the current accepted industry standard gold analysis method of fire-assay, and to provide data continuity between analyses performed using the two methods, the overall normalization of the PhotonAssay™ method was originally established using a suite of fire-assay certified-materials.

Although PhotonAssay™ and fire-assay grades agree closely overall, gold results for individual CRMs can differ between the two methods. Typically, these variations are less than 5%, although a small number of materials show larger differences. The overall normalisation of PhotonAssay and fire-assay grades for materials produced by the manufacturers CDN, Geostats, OREAS and Rocklabs agree within 1%, while the materials from AMIS and RRM show 2.4% and 4.8% positive normalisation differences respectively for PhotonAssay compared to fire-assay. There is no observable systematic trend with grade, and these differences appear to reflect variations in the materials used for CRM preparation.

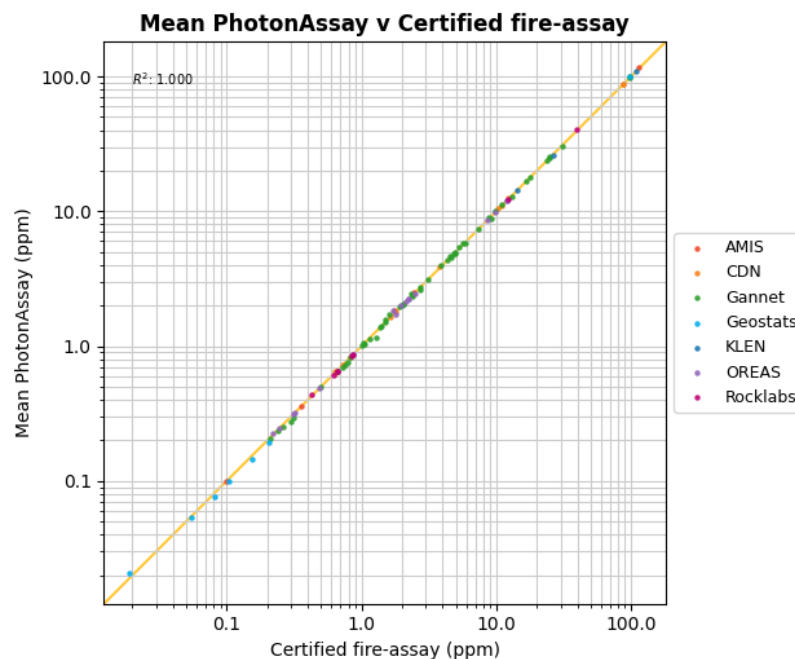


Figure 1. Comparison of PhotonAssay™ and fire-assay mean gold grades for a suite of 114 materials from seven manufactures.

When using a CRM for QAQC, apart from knowing the expected mean grade, it is also essential to understand the expected measurement precision. For a well-performing instrument, the measured grade will show a Gaussian or normal distribution about the true grade characterised by a certain standard deviation. The smaller the standard deviation, the more precise the measurement. Knowledge of the standard deviation is essential for setting 'control limits' which are bounds used to discriminate between acceptable and unacceptable deviations from the mean.



It is important to note that the between lab-standard deviations reported on CRM manufacturer’s data sheets are not intended to be used to set control limits in this way. Excerpts from the material data sheets provided by the manufacturer OREAS¹ and Rocklabs² highlight this:

- OREAS: “In the application of standard deviation (SD) in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.”
- Rocklabs: “Our suggested best practice would be to accumulate a history of the test results obtained and plot them on a control chart to determine any laboratory bias and variability. The appropriate centre line and control limits for this chart should be based on the average level and variation exhibited in the laboratory’s own data. This chart will provide a clear picture of the long-term stability or otherwise of the laboratory testing process, providing good clues as to the causes of any problems.”

PhotonAssay™ and fire-assay are distinct methods and have precisions that vary differently with gold grade. For example, the PhotonAssay™ instrument error is generally better than fire-assay at grades above 2-3 ppm, whereas in the range of 0.3-3 ppm the precision of the two methods is comparable, and at grades below about 0.3 ppm fire-assay has higher precision³.

Table 1 Typical performance of PhotonAssay™ and fire-assay on certified reference materials. For fire-assay, detection limits are from (Hoffman, Clark, & Yeager, 1999); accuracy ranges estimated from inter-laboratory standard deviation values.

Gold assay performance	Value for fire-assay	Value for 2-cycle PhotonAssay
Detection limit (2σ)	1 ppb (ICP) 5-10 ppb (AAS) 20-30 ppb (grav.)	10 ppb (blanks) 15–30 ppb (typical ore)
Grade at which 10% precision (1 SD) is achieved	0.1-0.2 ppm	0.2-0.3 ppm
Precision @ 0.35 ppm	5-8%	7%
Precision @ 1 ppm	3-7%	4%
Precision @ >10 ppm	2.5-3.5%	2%

The intrinsic differences between the two methods means that using fire-assay certified mean grades and standard-deviation values for PhotonAssay™ QAQC is not acceptable and may result in apparent QAQC fails. The use of method-appropriate mean grades and selecting control limits that correctly reflect the precision of the method is vital for appropriate QAQC.

Increasingly, manufacturers are now providing CRMs that include PhotonAssay™ certified mean grades and standard deviations and these values should be used for QAQC when available .

¹ <https://www.oreas.com/downloads/?fileId=1491>

² <https://www.scottautomation.com/assets/Reference-Materials/30-11-20/OxE166-signed.pdf>

³ When it comes to total measurement error (combined sampling and instrument errors) on real-world samples, PhotonAssay™ is generally more precise than fire-assay down to gold grades of 0.1 ppm.



3. Selection and Use of Monitoring Standards for PhotonAssay™

3.1. Standards selection

During routine operation of a PhotonAssay™ unit, registered monitoring standards must be included in the sample stream. These registered monitoring standards are used by Chrysos' automated QAQC processes to monitor instrument performance and to ensure consistent high-quality analysis. Additionally, laboratories and clients may include 'blind' standards at their discretion.

Chrysos provides a recommended list of monitoring standards and blanks for use with each PhotonAssay™ service. These monitoring standards have recommended mean grade and standard deviation values that should be used for ongoing internal quality control (QC). As PhotonAssay™ is intrinsically insensitive to material type and independent of particle size, matrix matching is not required. For example, the recommended monitoring standard suite for the standard gold service (PAAU02) includes a blank, two low grade, two medium grade and one high grade material; details are provided in Technical Note TN-403 (Chrysos PhotonAssay™ Monitoring Standard Recommendations). These standards have been selected for the following reasons:

- They span the measurable grade range for each service
- Many have been certified externally for the PhotonAssay™ method or have had their performance internally determined by Chrysos
- The lower-grade materials have low levels of uranium, thorium and barium, improving measurement precision and providing more statistically useful results for assessing instrument performance
- Low grade standards (< 0.6 ppm) are less reliable for tracking unit normalisation due to the lower instrument precision at these grades

When selecting monitoring standards that are not part of the recommended list in TN-403, it is recommended to contact Chrysos to discuss requirements and assess the suitability of the selected materials.

3.2. Blanks

Blanks are measured as part of the monitoring standard suite and are treated the same by the QAQC software on the PhotonAssay™ unit. These blanks are analysed to calculate detection limits and to check that the unit is only detecting measurable concentrations in materials with grades above the detection limit. The addition of blanks also provides a source of data for long-term tracking of unit operation.

Chrysos recommends the use of 1 blank material as part of the monitoring standard suite for each service (refer to TN-403). These blanks should be free of interfering elements (minimal U or Th content, ideally < 1 ppm) as elevated interfering element grades will increase the detection limit. A clean white sand (e.g., play-pit sand) is a readily available and low-cost material that typically meets these requirements.

3.3. Quantity

Enough of each monitoring standard should be initially purchased to allow 5-6 jars of each material to be prepared. A 2.5 kg tub of material should be sufficient in most cases. The monitoring standard jars must be kept topped up as material settles or is lost during jar



mishandling (see section 3.5), so spare material from the same batch from the manufacturer needs to be available.

As PhotonAssay™ is a non-destructive method, the same monitoring standard jar will be re-used multiple times over a period of days or weeks. Having multiple jars in circulation at any one time reduces the impact of errors from any given jar, for example if material is lost.

3.4. Measurement

Monitoring standards for PhotonAssay™ QAQC should be spread evenly throughout the sample jars at a rate of 1 monitoring standard for every 20 client sample jars. This enables a monitoring standard to be run approximately every 20 minutes. Monitoring standard jars must be registered in advance within the Chrysos database, as this will allow their observed grades to be used for automatic QAQC tracking. The same monitoring standards are also used during commissioning for the initial confirmation of system performance.

The required rate for standard insertion enables Chrysos' automated QAQC software to consistently track data quality and instrument performance. If monitoring standard rates are too low, or the monitoring standards are not evenly spaced throughout the batch, the unit may be running 'blind' for long periods of time. This can increase the likelihood that the automatic QAQC checks will fail once an entire batch is completed. If the automatic QAQC checks fail, the data will need to be reviewed by the Chrysos Physics QAQC team before they can be released. Overall, higher monitoring standards throughput will provide better tracking of the instrument performance, ensure data consistency, and facilitate faster data turnaround. Additional monitoring standards can be added blind to the sample stream, enabling the customer to conduct independent QAQC checks.

3.5. Ongoing care and maintenance of standards

As PhotonAssay™ is a non-destructive method and the same monitoring standard jars are used repeatedly before being replaced, it is important that these are properly managed and maintained.

Correct knowledge of the weight and fill-level is important for accurate PhotonAssay™ results to be obtained, therefore it is essential to check and prevent changes in jar weight or fill. To prevent settling or material loss from affecting results, monitoring standard jars should be kept well-packed with a 100% fill level. Jars are prone to decrease in fill over time, from the possible loss of material during mishandling and the settling of material within the jar as it loses entrained air. Some materials are more prone to settling than others. The jar fill is only measured when a monitoring standard jar is first registered onto the system⁴, so settling over time can result in the fill being overestimated as the monitoring standards are continuously re-run. When a monitoring standard is initially prepared, the correct packing process should prevent any substantial decrease in jar fill from occurring during its lifetime. Topping up and re-registering of monitoring standards should occur if a jar's fill decreases for any reason.

Monitoring standards should be re-jarred before a jar reaches 130 cycles (15s cycles) or 60 (40s cycles) of operation. Typically, this will occur every 1-2 months depending on overall unit utilisation. For the full re-jarring process, refer to the Chrysos Work Instruction document WI-023. Re-jarring is required as the plastic jars become brittle after multiple passes through the machine, increasing the chance of jars cracking and losing material. Chrysos' automatic

⁴ When jars are run repeatedly through the system, their bases can become scuffed and marked, which affects the performance of the optical fill measurement system.



software retires a monitoring standard jar after it has gone through the maximum number cycles. This means that the material cannot be re-used until it has been re-jarred and provided with a new barcode.

4. Repeat and duplicate measurements

Repeat measurements involve multiple measurements on the same portion of material, while duplicate measurements involve analysing two separate portions of material prepared from the same sample.

For fire-assay, repeat measurements of the same aliquot are impossible as the material is consumed during the analysis. Therefore, method precision can only be inferred by performing duplicate measurements on different aliquots of suitably homogeneous monitoring standards.

The PhotonAssay™ repeatability is dominated by the statistical errors associated with counting individual gamma-rays emitted by the sample. The PhotonAssay™ precision for a given sample depends on the grade, the presence of other background elements such as uranium, thorium and barium, and sample heterogeneity. The analysis software used to calculate a sample grade also uses conventional error propagation techniques to calculate the expected instrument precision for each reported grade value and includes this in the results file in the 'Error' column. The calculated precision is also used to determine the lower detection limit (LDL) for each element in the sample.

As the instrument precision is reported for each sample, there is no direct need to perform repeat measurements to determine the PhotonAssay™ precision. However, as PhotonAssay™ is a non-destructive technique, repeatability can be checked if required by simply re-running a sample as many times as required and comparing the reported results. For highly heterogeneous samples the observed repeatability may exceed the reported instrument precision due to movement of gold within the jar.

5. Chrysos' automated and manual QAQC processes

PhotonAssay™ units perform extensive automated QAQC checks, which are further analysed offline by the Chrysos QAQC team. This section summarises the various quality checks that are performed before batch results are reported to clients.

5.1. Instrument QAQC

The PhotonAssay™ software completes numerous low-level checks before the start of normal operation and during sample analysis. At the start of each day, calibration ('k-cal') jars are run through the unit to provide an automated review of the operating conditions of the key PhotonAssay™ components and a detailed check of the calibration. During operation, the condition of the X-ray source, gamma-ray detectors, automation system and safety systems are continually monitored. Measurements failing any of the quality checks are automatically re-run. If any significant and sustained deviation from normal operating conditions is detected, the system automatically enters 'safe mode' and prevents any more assays from being run.

Factors that may initiate safe mode include:

- Problem with the detector performance or cryogenic cooling system
- X-ray source operating outside of required energy or power settings
- Reference discs placed in the wrong locations in their presentation tray
- Automation issues resulting in unusual transfer or measurement times
- Monitoring standards deviating substantially from their expected grades



Once safe mode is triggered, the unit can be re-started by the user. However if the unit enters safe mode a second time, this indicates that there may be an issue requiring a maintenance engineer's intervention. In this case the user should alert the Chrysos maintenance team.

5.2. QAQC performed on standards

Chrysos' automated software tracks monitoring standards that are registered on the PhotonAssay™ unit to facilitate routine QAQC processes. These include:

- Tracking of system performance on a batch and a monitoring standard level
- Automatic generation of alerts on failure of any QAQC check
- Long-term stability monitoring
- Automatic generation of QAQC charts for selected monitoring standards

During routine operation, running a monitoring standard triggers a series of automated QAQC checks. Most reports of sample grades are automatically exported once a batch with no QAQC triggers has been completed. If the QAQC conditions are not met, the software flags the batch and prevents the report from being directly exported to the customer, holding it up for review by the Chrysos QAQC team (see section 5.3).

The QAQC checks that lead to data being held include:

- The average weighted ratio of the monitoring standards in the time-period including the batch falls outside 2 SD from the registered monitoring standard values for 95% of monitoring standards
- A monitoring standard jar is outside of 3 SD from the expected grade
- Two consecutive measurements of a monitoring standard fall outside 2 SD from the expected grade
- There is a jar cycle mismatch. This occurs when the automation has cycled an incorrect jar for the sequence. As a result, both jars (the expected and the measured) will be flagged as ERR with no result given. The report would be issued, and the client informed that there are jars without results which can then be re-run in another batch

The monitoring standard checks are the final step in the automated QAQC process. Batch QAQC is only performed once a batch is completed, that is when all jars finish their required measurement cycles. If all the export conditions are met, then the batch report is exported to the customer.

5.3. Chrysos QC Team Review

Any batch which is not automatically reported is directed automatically to the Chrysos QAQC team for review. This team receives notifications of any problems for a range of different metrics: raw data quality, detector, and LINAC performance etc. The physics team reviews the underlying cause, with the reporting of results blocked until the review is completed. On rare occasions, samples may need to be re-run, and this will be communicated directly to the customer's QAQC team.

The team provides 24/7 coverage for QAQC and customer support. There is a #QC slack channel that alerts staff to any QAQC-failed batches, an email address (support@chrysosecorp.com), and a web-based portal to meet different support requirements.



6. QAQC reporting

Daily, weekly and monthly reports are sent out via email and Slack at 6:00 am client local time. These reports provide a summary of the daily, weekly or monthly performance to the customer, and provide continuous feedback on a unit's output.

The daily reports provide a summary of the batches, the number of samples and number of monitoring standards run through the unit that day, and QAQC plots for each of these monitoring standards. The report also details any monitoring standard jars that have imminent retirement and those that have expired. Any monitoring standard jars that have expired must be removed from circulation and replaced with new jars.

The weekly report provides more precise information for each standard, including whether the weekly mean weighted element ratio is within plus or minus 2 SD and 1% of the expected grade of the monitoring standard. Any individual jars with mean grades that are outside of these limits are highlighted in red. These monitoring standards are plotted on a control chart to show the overall normalisation. Figures 2 and 3 illustrate typical automatically generated control charts for individual and multiple monitoring standards respectively over different time periods.

Finally, the monthly report provides a high-level overview of the batches, the number of samples and number of monitoring standards run through the unit that month and provides QAQC plots for each of these standards.

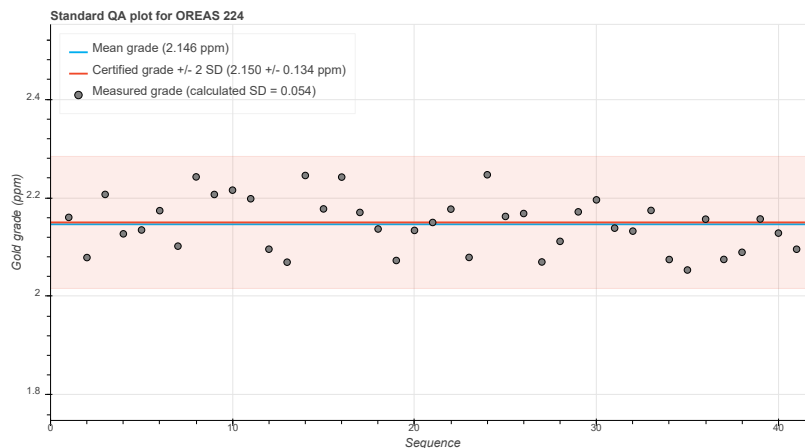


Figure 2 Example of automatically generated QAQC chart for a monitoring standard measured over a 10-day period.

CRM RATIO PLOT:

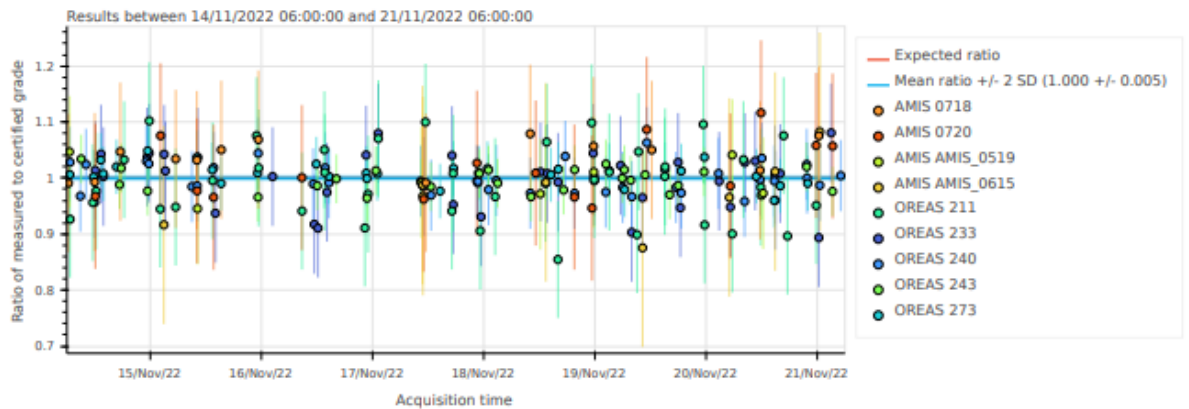


Figure 3 Example of automatically generated grade ratio plot for multiple monitoring standards over a 1-week period.

7. Summary

Most of the day-to-day QAQC is handled by the automated software installed on each PhotonAssay™ unit, with numerous checks being performed for every measurement cycle to ensure consistent data quality. Monitoring standards are an important part of the QAQC process. The selection, use and care of monitoring standards are important aspects of ongoing QAQC that require customer input. Inclusion of registered standards at the recommended rate of 5% (1 monitoring standard per 20 samples) will ensure optimal data quality and rapid and automated reporting of results.

8. References

Tickner et al., 2017. Improving the sensitivity and accuracy of gamma activation analysis for the rapid determination of gold in mineral ores. Applied Radiation and Isotopes 122 (2017) 28-36

