OPTIMIZING THE BALANCE OF QUALITY AND TURNAROUND TIME FOR A PROCESS CONTROL XRF LABORATORY

Suzanne W. Bowe

Kennecott Utah Copper, Magna, Utah

ABSTRACT

Kennecott Utah Copper, outside Salt Lake City, Utah, is one of the largest copper producers in the world. A major part of this operation is the Smelter, where many of the processes are controlled by wavelength dispersive X-ray fluorescence (WDXRF). Because of the increasing value of the products and increasing production costs, and uses of data outside of process control, the quality of the Smelter Lab assays has become more important. A program was initiated to improve the quality of results while still maintaining acceptable turnaround time. This paper describes how these goals were achieved.

INTRODUCTION

Kennecott Utah Copper’s Bingham Canyon Mine outside Salt Lake City, Utah, is one of the largest copper mines in the world. The operation also includes a concentrator, smelter, and refinery, and takes the copper from about half a percent in the ore to 99.99% pure in the finished product. The company is also a major producer of gold, silver, molybdenum and sulfuric acid, and sells other metals depending on markets and recovery technology.

The operation includes a central lab as well as smaller labs in the various plants to do control analyses, final product purity assays, and analyses for metals accounting, research, and environmental and industrial hygiene monitoring. The Smelter has the largest of the satellite labs; this lab operates continuously, using WDXRF to control many of the Smelter processes. There are sixteen routine sample types, which can mean as many as 260 samples a day (although usually fewer), plus QC’s and special project samples.

Table 1. Typical 24-hour routine process control sample load for the Smelter XRF Laboratory.

<table>
<thead>
<tr>
<th>Category</th>
<th>Samples</th>
<th>Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrates (Smelter feed)</td>
<td>Three types, with different additives (e.g., flux, coolant)</td>
<td>58</td>
</tr>
<tr>
<td>Mattes (approx. 70% Cu)</td>
<td>Three types, with different physical characteristics</td>
<td>42</td>
</tr>
<tr>
<td>Silica Slag</td>
<td>One type</td>
<td>20</td>
</tr>
<tr>
<td>Calcia Slag</td>
<td>One type</td>
<td>28</td>
</tr>
<tr>
<td>Miscellaneous Materials (for temp. control and recycling)</td>
<td>&quot;40Bin&quot; (mix of slags and dirty matte)</td>
<td>16</td>
</tr>
<tr>
<td>Boiler and Electrostatic Precipitator Dusts (four types)</td>
<td></td>
<td>8</td>
</tr>
<tr>
<td>Silica Slag Mill Products</td>
<td>Heads, Tails, and Concentrates</td>
<td>12</td>
</tr>
<tr>
<td><strong>Total Samples</strong></td>
<td></td>
<td><strong>184</strong></td>
</tr>
</tbody>
</table>
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The analytical process starts with drying for wet samples (moisture may also be reported) and crushing for furnace products. Dried or crushed material is milled, and some samples require a magnetite analysis on a milled portion. Then, powdered sample is mixed by weight with wax, pressed, and analyzed on either of two instruments (PANalytical Axios and 2400). Parameters such as grinding time, sample and wax weights, and counting time vary by sample type.

The lab is staffed by one analyst at a time, who also has some non-X-ray work. This leaves little time for method development or troubleshooting problems, so over time, some problems have accumulated. And because of the increasing value of the products and increasing production costs, it has become more important than ever to control Smelter processes more tightly. In addition, some of the analyses are used for metals accounting or research projects. Therefore, high-quality assays are very important.

With this in mind, Smelter technical management initiated a project to improve the quality of the XRF analyses. The requirements were:

- all new, more accurate, analytical methods, with particular attention to matrix variation,
- improved precision,
- a full QA system,
- specific quality targets, and
- the lab must still function with only one analyst at a time.

As the work progressed, questions from data users most often concerned variation—had an analysis procedure changed? But with all the attention on quality, turnaround time requirements did not change; operations still expected results as quickly as before.

This paper will describe how the competing needs for speed and quality were addressed.

**PROGRAM OVERVIEW**

To provide the needed quality without slowing down operations, four points were key:

- Customer communication
- Accurate analytical method setup
- Minimization of variation
- Quality and efficiency improvements
CUSTOMER COMMUNICATION

Communication with customers was essential; by determining what the customers required, wasting resources on what was not important to them could be avoided. In the case of this project, the customers were the Smelter Technical Department and operations personnel. There are no regulatory requirements for how the lab produces data, so the system could be set up with the sole focus on providing the best data to control Smelter processes within acceptable time limits.

Communication went both ways; when major changes were considered, the customers had to make the final decision, but needed complete information from lab personnel to do so. Regular contact also provided opportunities to educate the customers about the lab; as they became more comfortable with the lab and more knowledgeable about the analyses, they were less likely to assume the lab was the source of problems.

Communication with customers was both formal (regular project meetings, quarterly QA reports, documents on company network, etc.) and informal, and included encouraging analysts to add written comments to data in the case of unusual samples, etc.

ACCURATE METHOD SETUP

The guiding principle in setting up new analytical methods was to take the time and effort to address very thoroughly all aspects of the analysis up front. Although this results in long method-development time (some new methods are yet to be completed), this policy helps avoid customer dissatisfaction and saves time in the long run by eliminating the need for problem-solving and rework.

During method development, there were many instances where speed and quality conflicted. Advantages and disadvantages had to be weighed in each situation to make the compromise that best suited the customer. In many cases, the decision was made in the lab based on knowledge of the customer’s needs, but major decisions were made by the customer after considering information provided by the lab.

The general method development procedure and some examples are described below.

First, customer requirements for each sample type (turnaround time, elements needed, quality requirements, etc.) were determined.

Then the sample preparation procedure was developed. Initially there was an interest in fusion, but management decided to see what improvements could be made without it; their concern was time, both in terms of elapsed time from sample receipt to data reporting, and the increased workload for the analyst. As setup of methods progressed, fusion was reconsidered for a sample type that included additives of variable mineralogy, but again was declined. However, while fusion was not found suitable for routine analysis, it was determined to be useful for research on varying sample matrices. Pressed powder continued to be used for real samples, but most grind
times were increased to improve precision. Separate methods were set up for each sample type to minimize matrix effects.

Because pressed powder was used, and there were no CRM’s for our specific sample types, real samples were used as standards. Samples were collected over a long enough period of time to provide wide concentration ranges of each element and appropriate relative variation for good line overlap calculations where needed. Enough standards were made to be able to take advantage of any of the software’s calculation options, and enough volume of each was collected to allow for repeated assays, particle size determinations, extra material for testing in case of any questions in the future, and large enough splits for both the Smelter and Central Labs to make multiple pressed pellets (methods were also calibrated at the Central Lab in case both Smelter instruments are out of service at the same time, unlikely but possible). Standards were prepared in the same way that real samples were to be prepared. Multiple analyses were done to characterize each standard, using both the Kennecott Central Lab and outside services.

Time was taken to carefully choose all instrument parameters when setting up the method, and to process the calibration data after running the standards.

Drift correction procedures were carefully considered for each sample type. In many cases the same channels, using standards of high and low concentrations to determine slope and intercept, were used for multiple methods. However, for some sample types, individual channels that were drift-corrected differently were set up for specific elements. The drift-correction method possibilities vary by instrument manufacturer, and are worth investigating and optimizing to obtain consistent results without recalibration.

Each kind of sample had its own challenges. For example, one sample type has about one or two tenths of a percent of silicon in about 68% copper. The silicon is a very important control element and was a frequent target of customer questions in the past. To optimize low silicon analysis, InSb crystals were installed in both instruments. This crystal is only used for silicon
but it gives much better sensitivity than what would otherwise be used. The drift correction was
done a little differently than for most elements, and more QC’s are run for this sample type than
any other to give the data users the assurance that previous problems with silicon have been
corrected.

Once a method was set up, QC samples were run both immediately after calibration to
demonstrate accuracy, and on an ongoing basis to demonstrate continued consistent
performance. In general, QC’s are set up to address customer concerns; most ongoing QC’s are
run to verify precision, since that has been the most frequent concern of data users. QC’s to
demonstrate ability of the method to handle varying matrices within a sample type were run
initially, and if a sample type changes, new samples are collected and characterized to see how
well the method handles them. In some cases, they may be added to the calibration curves.

Before any new method was implemented, one or more real samples were run daily for at least a
month by both old and new methods to give data users a good idea of what changes to expect. A
data package consisting of these old-vs.-new comparisons, QC results (initial accuracy, several
precision comparisons, short-term drift), schedule of QC’s to be run, and discussion of possible
matrix issues was given to technical management.

MINIMIZING VARIATION

Even before new analytical methods were developed, some procedures were set up or modified
to improve precision; these improvements are often much quicker to initiate, and are especially
helpful in process control, where adjustments are made based on analytical trends. Additional
precision improvements came as new methods were set up. The goal was to create a system that
runs consistently enough that there are no surprises, either for the analysts or the data users.

Some examples of procedures used to decrease variation were longer grinding and counting
times (which of course had to be balanced against turnaround time requirements), optimized drift
correction, and good support equipment maintenance, such as increasing the frequency of
polishing mold caps for the presses.

QUALITY AND EFFICIENCY IMPROVEMENTS

Whenever possible, improvements were put in place to increase speed without lowering quality,
or to improve quality without delaying analysis. Sometimes new equipment or a new procedure
improved both at once. Continuous improvement is always possible and the lab still looks for
opportunities. Some examples that have helped are:

The lab has three grinding mills. The previous procedure was to set each of the three timers for
the longest time used on the mill, then rather than take the time to reset the display when a
sample with a shorter grind time was prepared, to push the start button, and mentally count the
time or watch the clock, then push the stop button. Second timers were added to the mills,
allowing enough preset times for all sample types. This saves time and improves repeatability.
An effort was made to improve scheduling of maintenance, drift correction, and research samples for times when the lab had fewer samples to run, which was normally during planned repairs of sections of the Smelter. Included in the scheduling improvement was to plan instrument preventive maintenance visits, changing P-10, and drift correction all for the same time whenever possible; the new P-10 flows in while the instrument warms up after PM parts replacement, and drift correction which may be necessary after the changes to the instrument is done when it was scheduled anyway.

As part of the setup and optimization of the quality program, several control procedures such as drift correction were done by a single person outside the normal analytical flow. Having these procedures done by the same person has had the effect of keeping the quality more consistent, while at the same time helping keep the routine analyses on schedule because the analyst on shift can concentrate on that work. This is not practical for all labs, but is worth considering where there is a heavy workload.

QC samples were scheduled to determine two things: first, to let the analysts know whether the system is operating within specifications and can be used, and second, to let the data users know the level of quality of sample results. Even a relatively small number of QC samples can at times add stress to the workload, so a strong effort was made to be sure that only samples that are truly informative are run; no QC’s were assigned just for the sake of running a certain number of checks. The schedule was also made somewhat flexible to avoid interfering with process control analyses.

A QC sample developed for the program is the “Daily Check,” a glass standard run on a method set up individually for each instrument, which consists of one element for every crystal-detector combination in use. This sample is run daily and after repairs or other downtime, and has proven to be very useful in troubleshooting, helping to determine when the instruments are warmed up after downtime, and generally getting a good feel for how the instruments operate. Data from this standard was also helpful in setting the schedule for drift correction. This is the only QC that is run every day.

Several items that were already in place when this project was started are worth mentioning because of their contribution to efficiency:
Years ago Kennecott decided to get a second wavelength dispersive instrument for the Smelter because the existing backup was a portable with limited performance. This eliminated the backup problem and also helped with an increasing workload. Having two instruments also helps maintain quality by making it possible to take an instrument out of service when needed for maintenance or drift correction, which would be much more difficult if it were the only good instrument. Of course this isn’t practical or even necessary for everyone, but for a high-throughput, round-the-clock process control laboratory, our experience has been that it is very helpful.

The lab is very efficiently organized with respect to equipment placement, the result of good suggestions by the employees. The employees also work very well together. Kennecott has benefitted greatly from the involvement and teamwork of the analysts.

The lab also has a very efficient sample logging/data reporting system, which requires very little analyst time. The system includes a warning feature that prompts review of data with abnormal concentrations before allowing it to be sent out. This helps avoid giving the control room incorrect data, for example if samples are accidentally switched.

**RESULTS**

When new methods replaced procedures with known biases, “fudge factors” were taken out of the furnace control model. When biases weren’t as well understood or integrated into the model, QC data or new vs. old method comparisons have helped management to better understand Smelter processes.

Because of more consistent performance and a better understanding of the lab, there are fewer complaints from data users.

And finally, a major part of the analysts’ quarterly bonus is based on a turnaround time score. Despite increases in grinding and counting times, and the addition of QC and method development samples to the workload, because of efficiency improvements and procedures to minimize problems, the turnaround time score has remained at its previous level.

**CONCLUSION**

By using customer needs as a guide, working carefully to develop accurate methods, putting consistent procedures in place to minimize variation, and implementing efficiency improvements, it was possible to increase data quality to a level acceptable to management, without increasing turnaround time.