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ABSTRACT

This report looks at two ways to estimate the bulk mineralogy of the rocks for assay intervals. The aim is to find an efficient indicator of the most common minerals in the rock. Phase (modal) analysis has traditionally been done using visual methods such as point counting and image analysis. A modern version of this process is the X-ray point counting routine using the SEM-EDS based software. These methods are too slow and expensive for routine analysis of bulk sample mineralogy at the normal assay spacing.

Two sources of data were considered that provide information that can be used to determine the mineral abundance in assay samples. The most widely applied method is (semi-) quantitative X-ray diffraction (QXRD). The QXRD method is most applicable to major minerals and has limited application to minerals at low abundance. The nominal detection limit is 0.5%. Values below 5% have large errors. A second, less common, method is calculation of mineralogy from chemical assay data. Conversion of chemical analyses to mineralogical analyses depends on the unique chemical composition of each mineral. Elements only found in one mineral are easily accounted for, but many compositions are ambiguous. Deciding on the actual mineralogy is not simple. Recalculation of mineral mode from chemical analyses is more accurate than QXRD when the correct minerals, and mineral compositions, are known.

Where only a few QXRD analyses are available they can be used to setup a standard for calculation of mineralogy from assay data. We found linear programming works well in this environment. The best results are obtained when both H₂O and CO₂ are directly measured. LOI should be included if these are not available.

Where both QXRD and chemical analysis are available for all samples, the best results are obtained using the least squared method to merge the datasets assuming QXRD errors have much higher analytical errors than chemical assays. The combined method provides more robust results because the high abundance minerals are controlled by the QXRD measurements while the chemical assays improve the precision for low abundance minerals.

Keywords: QXRD, XRF, linear programming, non-negative least squares methods

INTRODUCTION

Mining operations worldwide excavate billions of tons of rock annually. Throughout the operations, the rock mineralogy is a key parameter in predicting performance characteristics such as mill throughput, concentrator efficiency, loss to tailings and acid generation/neutralisation potential. An efficient measurement of mineral abundance in assay samples would improve predictions of mine performance. There are a number of methods that can be used to calculate modal mineralogy from the chemical assay. We have tested two methods that have potential application in the minerals industry. These methods have been applied to a number of deposit types but the results reported here are from a porphyry Cu deposit.

METHODOLOGY

Combined XRF/XRD results

It is possible to calculate the mineralogy of rock directly from the chemical assay using a least squares approach (e.g. Herrmann and Berry 2002). This method requires knowledge of the composition of the minerals. Mineral composition data is often available from existing petrology reports or can be measured directly on the samples using EPMA. In this example, mineral compositions were found in existing unpublished reports or typical compositions were used from various literature sources including Deer et al. (1993). Better results would have been achieved if variable phases such as biotite and chlorite were measured directly on the samples.

The least squares method requires the number of elements analysed to exceed the number of minerals calculated. In most mines this is a major limitation since there are commonly more than 10 significant minerals. One way to overcome this problem is by measuring the mineralogy with (semi-) quantitative X-ray diffraction (QXRD). With the addition of a QXRD result we have many more constraints and can carry out the calculation with a full list of minerals. In the example reported here, we calculated the abundance of 25 minerals with 38 constraints. It is very important when setting up the least squares problem to have a column for elements that have not been analysed and a closure constraint (minerals total to $100 \pm 1\%$).

The QXRD method is most applicable to major minerals and has limited application to minerals at low abundance. In complex samples QXRD has poor precision. For example, Omotoso et al (2006) reported the efforts of three high quality XRD laboratories and showed the typical measurement errors were 10% and analyses have high detection limits with minerals below 0.5% commonly not detected.

The QXRD analyses were carried out at the University of Ballarat. X-ray diffraction traces were obtained from the assay pulps with Siemens D500 and D501 diffractometers using Fe-filtered $\text{CoK}\alpha$ radiation, operating conditions were 40kV/30mA. Quantitative XRD results were obtained using SiroQuantTM 3.0 and refinement of the most suitable mineral structures available in the current software package databank. SiroQuantTM 3.0 uses the Rietveld method which is a standardless, full pattern approach to quantitative phase analysis using X-ray powder diffraction data. For this test the chemical assays were determined using XRF analysis at the University of Tasmania. The samples were analysed with the ORE2 method, which uses two stage ignition

and a special flux designed to retain S, C and H were measured by Element Analyser at the CSL, University of Tasmania.

The method used to combine the chemical assays and QXRD results was a weighted least squares calculation using the NNLS subroutine of Lawson & Hanson (1995). This algorithm includes a forced non-negativity result. The least squares model was weighted by the estimate of measurement error. As a result, the misfit (X^2) can be used to identify problem samples (Press et al 1986) where the mineral compositions used are wrong or there is a mineral in the sample that is not in the calculation list. For the XRF results the measurement error was estimated as 3% relative plus 0.02% absolute. The errors for the QXRD results are based on repetitions of samples, the error estimates reported in the literature and consideration of the fit errors achieved. For XRD results the errors were set at 10% relative plus 1% absolute. At this weighting the problem can be stated as finding a modal mineralogy consistent with the chemical analysis which is as close as possible to the QXRD results.

Linear programming and the Simplex Method

The first method of calculating the modal mineralogy from chemical analyses requires QXRD results. Where there is no additional information one option is using linear programming and the simplex method (Press et al 1986). Linear programming is a mature method with application to real world quantities that cannot be negative. Again we need to know the mineral compositions that may be present however an extended list of minerals can be provided and in this example 45 minerals including 5 different chlorite compositions were considered. Linear programming maximises a linear objective function based on the proportional sum of a “property” of each mineral in the list. These “property” values need to be optimized for the common minerals. In this context the objective functions are a proxy for how likely the mineral is to occur in the deposit. Very likely minerals are given higher values. Knowledge from petrology reports and possibly some QXRD results are required to set these properties correctly. In this example, the QXRD results from half the samples were used to find the best value for each of the minerals. The linear programming allows fast calculation and there are no practical limits to the size of the mineral list to be considered.

For the tests carried out here the calculations have been done using the linear programming routine in Press et al. (1986). The “objective function” or mineral “probability” of quartz and barite was set to 5. Common minerals had values between 2 and 5. Unlikely minerals were set to 0 (talc, pyrolusite) or -2 (corundum, native Cu). Less common minerals not detected in the QXRD analyses were given objective functions of 1 (pyrophyllite, paragonite, chalcocite, rutile). Finding the correct values took several hours but once set these values can be used across the deposit.

RESULTS

Using combined QXRD/XRF analyses the mineralogy must match the rock composition closely. For major minerals this is generally a small correction compared to the QXRD alone. For example K feldspar hardly changes from the QXRD value (Fig. 1). In contrast, mineral estimates below 5% are improved. Apatite is the

dominant phosphate in these rocks and the combined QRD/XRF result (Fig. 2) represents a maximum value. The QXRD results for apatite were unreliable at 1% apatite. The original QXRD results had a recognised interference which meant the chalcopyrite was overestimated in many samples. These high estimates are inconsistent with the Cu content. Thus the combined QXRD/XRF value is either the same or less than the QXRD value (Fig. 3). The combined technique honours the reliable QXRD results and improves precision for other minerals where the assay data provides a more robust constraint.

The linear programming method provides a similar estimate of mineralogy for the most common minerals Quartz and feldspar match extremely well (e.g. Fig. 4). It also provides a robust estimate where an element is largely contained in one mineral (e.g. Cu in chalcopyrite: Fig. 5). There is a reasonable correlation of chlorite content (Fig. 6). However, there is a compositional ambiguity between biotite, chlorite and muscovite. With the parameters set to successfully calculate chlorite, the muscovite is variable (Fig. 7). Biotite content is general less than 10% in these samples (based on QXRD) and the linear programming method dramatically underestimated this mineral in all but one sample as Fe and Mg are allocated to chlorite first.

The model used here accurately estimated carbonate contents of the rocks. Surprisingly a second calculation where only loss-on-ignition was used also calculated carbonate contents similar to the combined QXRD/XRF values. It appears that measuring the CO₂ and H₂O⁺ content makes only a small difference in the ability to estimate the carbonate content of the samples.

CONCLUSIONS

QXRD is a suitable method to measure the abundance of the major minerals in the minerals industry. It is very effective for mineral above 10%, but many of the economic minerals are at too low an abundance to be accurately measured by QXRD. The precision of modal mineralogy estimate is significantly improved by combining the QXRD results with the chemical assay using a weighted least squares calculation.

Where QXRD is not available it is possible to calculate modal mineralogy using linear programming. This provides similar levels of accuracy to the combined QXRD/chemical assay method for most samples but a small number of samples will have large errors.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

Figure 1: Comparison of K feldspar content estimated by QXRD with the result after combining QXRD and chemical assay using weighted least squares method.

Figure 2: Comparison of apatite content estimated by QXRD with the result after combining QXRD and chemical assay using weighted least squares method.

Figure 3: Comparison of chalcopyrite content estimated by QXRD with the result after combining QXRD and chemical assay using weighted least squares method.

Figure 4: Comparison of K feldspar content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

Figure 5: Comparison of chalcopyrite content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

Figure 6: Comparison of chlorite content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

Figure 7: Comparison of muscovite content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

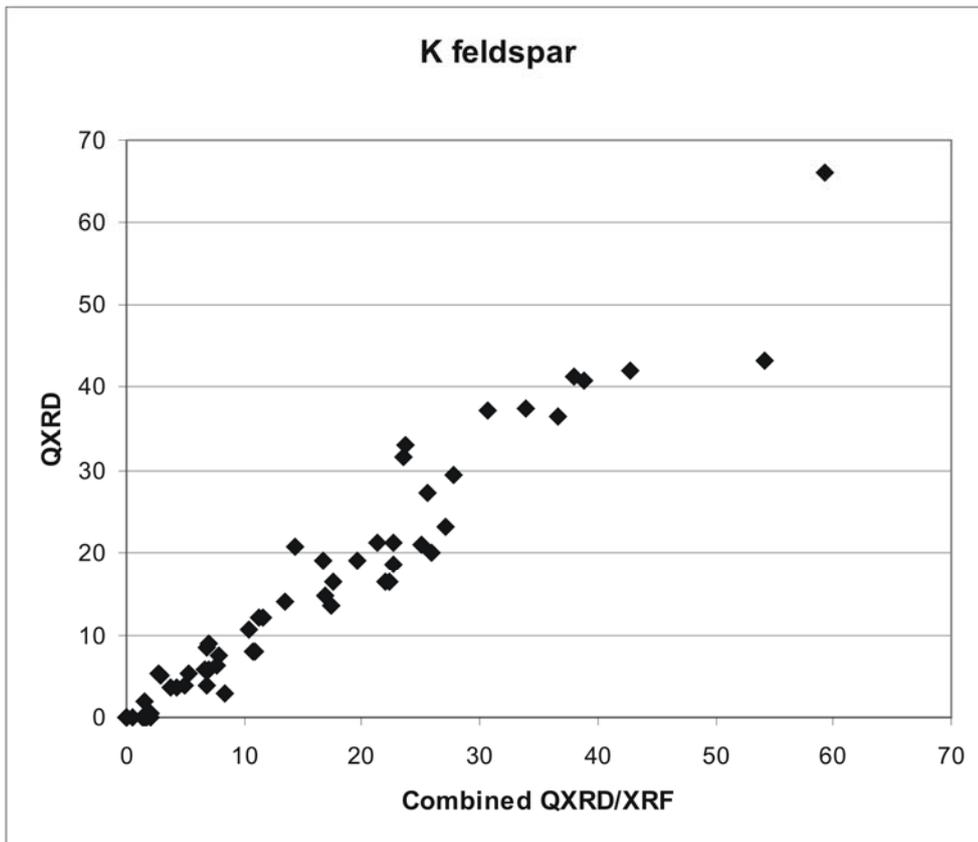


Figure 1: Comparison of K feldspar content estimated by QXRD with the result after combining QXRD and chemical assay using weighted least squares method.

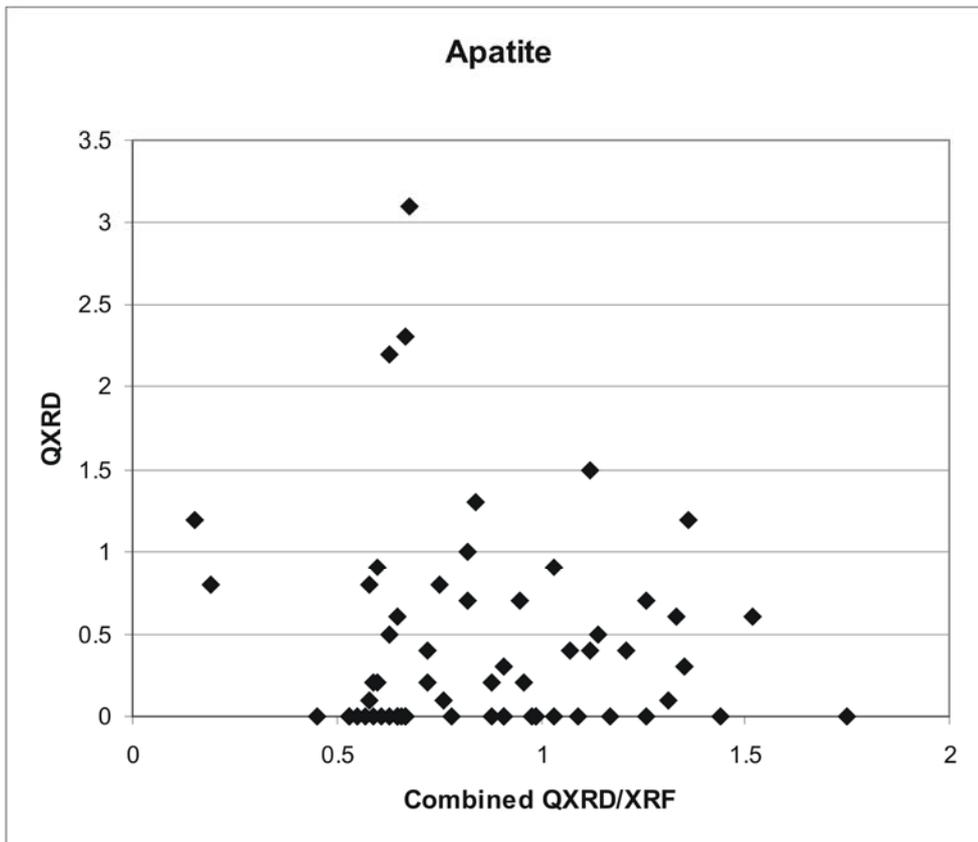


Figure 2: Comparison of apatite content estimated by QXRD with the result after combining QXRD and chemical assay using weighted least squares method.

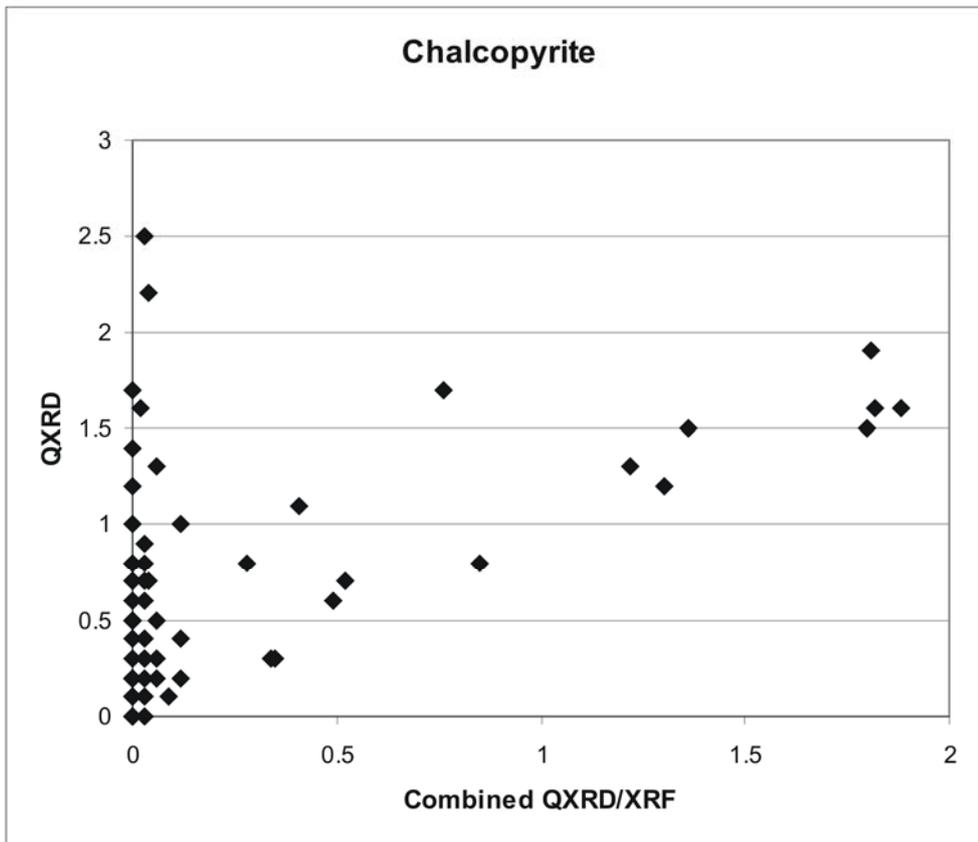


Figure 3: Comparison of chalcopyrite content estimated by QXRD with the result after combining QXRD and chemical assay using weighted least squares method.

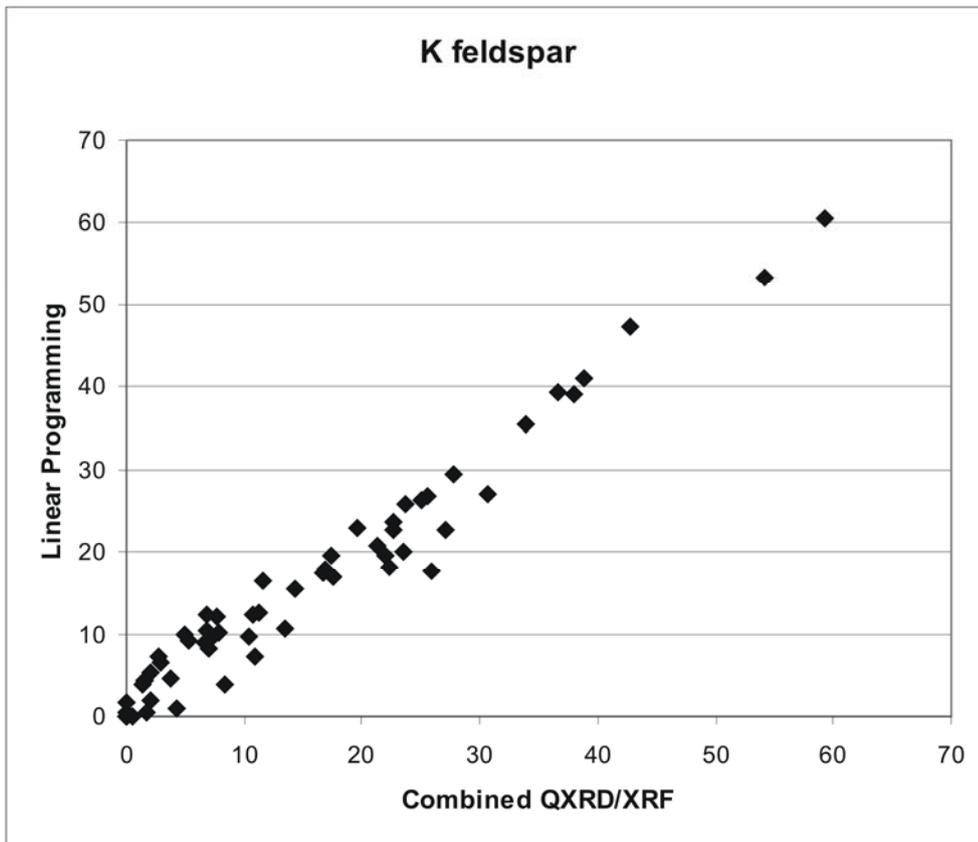


Figure 4: Comparison of K feldspar content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

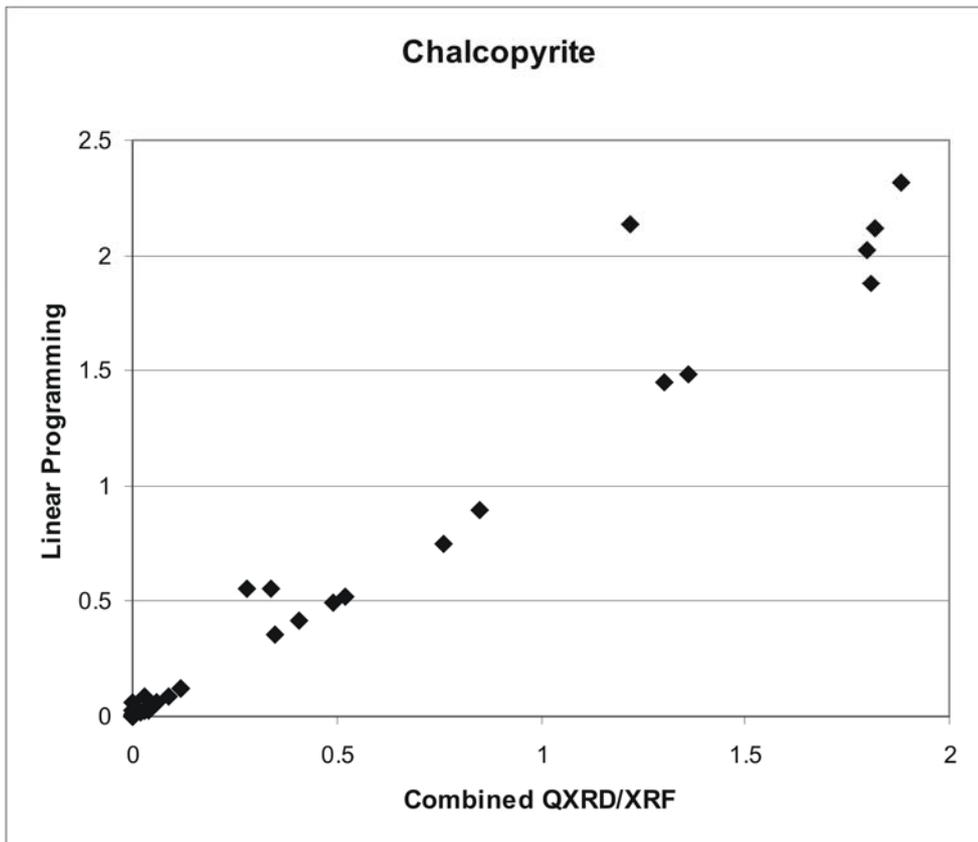


Figure 5: Comparison of chalcopyrite content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

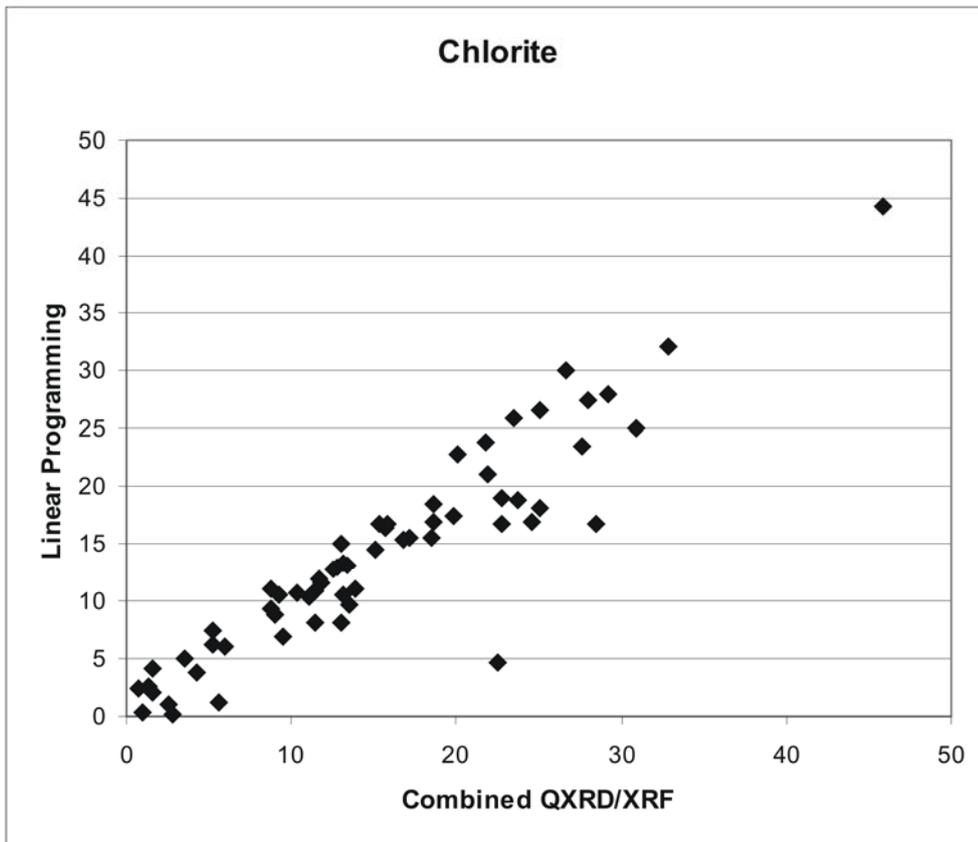


Figure 6: Comparison of chlorite content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.

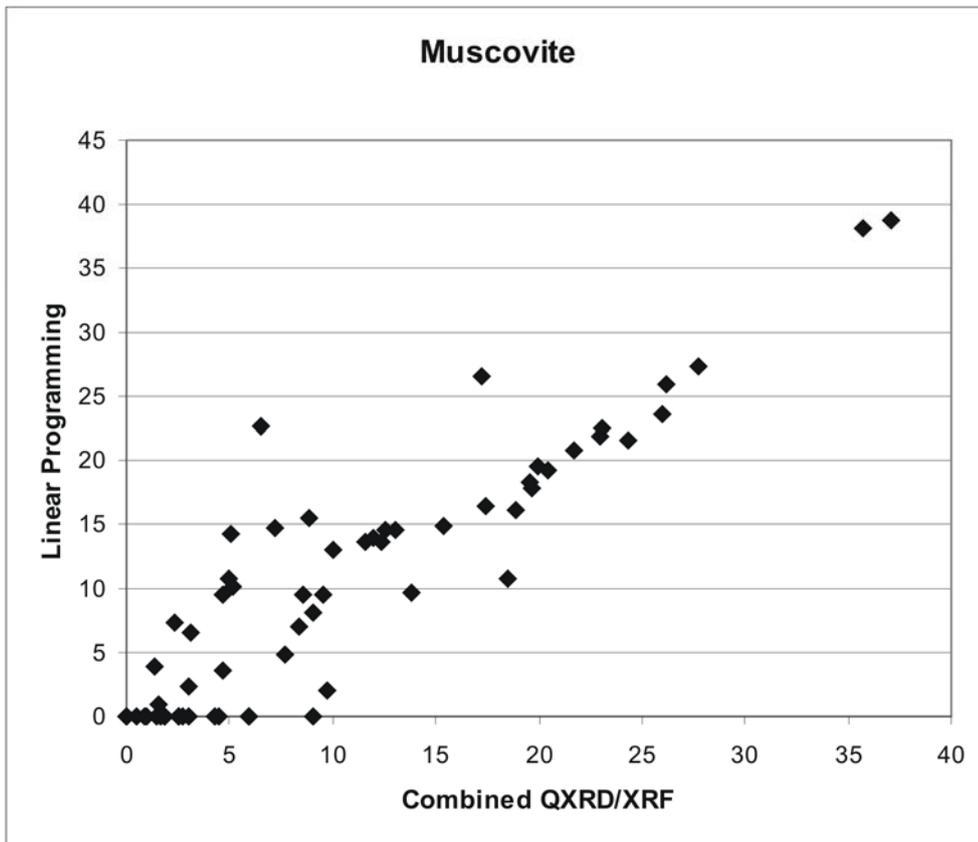


Figure 7: Comparison of muscovite content estimated by linear programming with the result after combining QXRD and chemical assay using weighted least squares method.