MINERALOGICAL BALANCES BY DISSOLUTION METHODOLOGY

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1. INTRODUCTION

Mineralogy has traditionally concentrated to identify minerals, search for new species, study of mineral structures and investigate the chemical composition of minerals. Mineral quantification has been an ignored subject not only in mineralogy and petrology but even in economic geology, thought the economic value of a mineral deposit is almost invariably controlled by the mass proportions of the minerals it contains, rather than by its overall chemical composition.

Whether a mineral deposit can be exploited commercially depends on several factors, some of which (e.g. political, economic and strategic conditions) are beyond the control of *the project team* (including at least geologist, mine engineer, mineralogist, process engineer, and metallurgist). However, frequently the major factor is the technical ability of *the team* to produce salable products at a socially acceptable cost. In this task the availability of reliable mineralogical data becomes crucial. In each of the stages from the evaluation of the ore deposit, via process selection and development to plant design following high-quality mineralogical information is required (modified after Jones 1987): (1) Sample composition (chemistry), (2) Mineral (or phase) identity, (3) Mineral composition, (4) Mineral proportion, (5) Element distribution, (6) Grain-size distribution, (7) Mineral properties, (8) Ore texture, (9) Particle-size distribution, (10) Particle shape, (11) Particle composition, and (12) Particle texture.

Information on mineral proportions, coupled with data on mineral compositions, enables *the team* to determine how the valuable elements and the penalty-incurring elements are distributed among the various minerals in an ore. This information makes it possible to determine which of these minerals should be recovered or extracted in a mineral processing plant. Such information can also indicate what processes are likely to be needed to collect these minerals or the valuable elements they contain into products of different qualities.

This paper introduces a method for mineral quantification using mass balance calculations and mineral phase analyses, i.e. selective leaching methods. Procedure has been used in Outokumpu group as a routine tool for ca. a decade in the studies of (sulfidic) Ni, Cu, Zn, Pb, and Au ores. Theoretical and descriptive part of the paper is based on manuscripts of P. Hautala (1992), who unfortunately never had the opportunity to publish his wide manuscripts on applied mineralogy.

An approach for mineral quantification used in metamorphic petrology is rather similar, but the main interest there has been a determination of stabile mineral paragenesis rather than an exact quantification of minerals (see Fisher 1989, 1993; Greenwood 1967; Thompson 1982; Nicholls 1977). Also norm calculations (Cross et al. 1903, Niggli 1936) resemble somehow

the procedure to be described, but the former are chain calculations rather than solving a set of linear algebraic equations.

2. THEORY

2.1 *Gibbs' Phase Rule*

Gibbs' Phase Rule, which is usually written in the following form:

$$f = c - p + 2,$$

presents for a system in heterogeneous equilibrium[1] the relation between the number of degrees of freedom (or variance) f, the number of chemical substances (number of components) c, and the number of phases (number of different homogeneous parts p).

Phase is homogenous, physically distinct and mechanically separable portion, while component is a constituent the concentration of which can undergo independent variation in the different phases. Component refers to the minimum number of chemical constituents that are necessary and sufficient to describe the composition of all phases within the system. Degrees of freedom can be defined as the minimum number of variables that must be fixed in order to define perfectly a particular condition in the system.

In mineralogical samples phases are minerals, but components can be selected in many different ways depending on problem. Usually in silicate and oxide system oxides are selected for components. In sulfide systems chemical elements and in solid solutions end members are commonly selected as components, respectively.

The number of degrees of freedom can be defined as

$$f = M - N$$

where M is the number of quantities (pressure, temperature and mole fractions) necessary to describe the system and N is the number of them that cannot be varied independently.

Because the term f gives the minimum number of variables that must be fixed to define the condition of the system, the number must be either positive or zero. The maximum number of stable coexistent phases in a system will be present when f=0. This argument can be applied to multicomponent systems; for example, it can be immediately deduced that maximum number of stabile coexisting phases in a 12-component system is 14.

When pressure and temperature are fixed, i.e. isobaric and isotermic system, then Gibbs' Phase Rule deduces to a form

$$f = c - p$$

which equals to

$$p = c - f.$$

While f must be greater or equal to zero, it follows that

$$p \leq c.$$

When system is in equilibrium and temperature and pressure are fixed, the number of phases is equal or smaller than the number of components.

2.2 *Mathematics*

We shall deal here mineralogical systems and samples and use minerals as phases and elements (or oxides) as components. Notations p (phase) and c (component) will be replaced

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[1] Heterogeneous equilibrium is equilibrium between different, physically distinct parts.

by m (mineral) and e (element), respectively. We shall use notations familiar to matrix calculations.

Let:
w=weight fraction
$a_{e,m}$=weight fraction of component e in phase m
b_e=weight fraction of component e (in a sample)
x_m=weight fraction of mineral m (in sample)

In an ideal case following closure conditions prevail:

$$\sum a_{e,m} = 1 \quad (0 \le a_{e,m} \le 1)$$

$$\sum b_e = 1 \quad (0 \le b_e \le 1)$$

$$\sum x_m = 1 \quad (0 \le x_m \le 1)$$

A set of linear algebraic equations can be written for a system consisting of P mineral phases (m=a,b,c,...,P) and C components (e=1,2,3,...,C) as follows:

$a_{1,a}x_a + a_{1,b}x_b + a_{1,c}x_c + ... + a_{1,P}x_P = b_1$
$a_{2,a}x_a + a_{2,b}x_b + a_{2,c}x_c + ... + a_{2,P}x_P = b_2$
$a_{3,a}x_a + a_{3,b}x_b + a_{3,c}x_c + ... + a_{3,P}x_P = b_3$
.....
$a_{C,a}x_a + a_{C,b}x_b + a_{C,c}x_c + ... + a_{C,P}x_P = b_C$

Here the minerals, P unknowns x_m, m=1,2,3,...,P, are related to C equations. The weight fraction of the elements in the minerals (i.e. chemical compositions of the minerals), $a_{e,m}$ with e=1,2,3,...,C and m=a,b,c,...,P, are known numbers, as the right-hand side weight fraction of the elements in the sample (i.e. chemical composition of the sample) b_e, e=1,2,3,...,C.

This can be written in terms of matrix calculations. Let us write three different matrices. First for chemical composition of minerals, or more strictly the weight fraction elements in minerals (matrix A), second for chemical composition of the sample, or more definitely said weight fraction of elements in the sample (vector b), and third for mineral[2] composition of the sample, or weight fraction of minerals in sample (vector x).

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[2] Often a term "mineralogical composition of the sample" is used, but here for simplicity "mineral composition of the sample" is applied

$$A = \begin{pmatrix} a_{1a} & a_{1b} & .. & a_{1P} \\ a_{2a} & a_{2b} & .. & a_{2P} \\ .. & .. & .. & .. \\ a_{Ca} & a_{Cb} & .. & a_{CP} \end{pmatrix}$$

$$b = \begin{pmatrix} b_1 \\ b_2 \\ .. \\ b_C \end{pmatrix}$$

$$x = \begin{pmatrix} x_a \\ x_b \\ .. \\ x_P \end{pmatrix}$$

where C is a number of elements (components) and P is a number of minerals (phases). In ideal case A is square matrix (C=P).

Equation above can be written now in matrix form as A•x=b, i.e.

$$\begin{pmatrix} a_{1a} & a_{1b} & .. & a_{1P} \\ a_{2a} & a_{2b} & .. & a_{2P} \\ .. & .. & .. & .. \\ a_{Ca} & a_{Cb} & .. & a_{CP} \end{pmatrix} \bullet \begin{pmatrix} x_a \\ x_b \\ .. \\ x_P \end{pmatrix} = \begin{pmatrix} b_1 \\ b_2 \\ .. \\ b_C \end{pmatrix}$$

Here the raised dot denotes matrix multiplication, A is a matrix of coefficients, x is a column vector of unknowns, and b is right-hand side written as column vector.

As an example (later referred as example 1), we want to know the weight fraction of sulfides from a sample[3] consisting of chalcopyrite, pyrrhotite, sphalerite, quartz, magnetite, plagioclase, graphite and other minor gangues, which do not contain copper, sulphur nor zinc. Bulk chemical analysis by XRF gave S=6.13 wt.%, Cu=3.66 wt.%, and Zn=1.00 wt%. Chemical composition of minerals was determined by electron microprobe, and the averages of the analyses are as follows: sphalerite: S=33.12 wt.%, Cu=0.26 wt.%, Zn=59.03 wt.%; chalcopyrite (stoichiometric): S=34.94 wt.%, Cu=34.64 wt.%, Zn=0.00 wt.%; and pyrrhotite: S=39.54 wt.%, Cu=0.01 wt.%, Zn=0.00 wt.%.

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[3] Sample derives from Viscaria Cu-deposit (Martinsson 1991) and represents one ore type mined

Phases of interest are sphalerite (sp), chalcopyrite (ccp) and pyrrhotite (po). Components are Zn, Cu and S, respectively. Now the matrices A, b and c can be written:

		sp	ccp	po
	Zn	0.5903	0.0000	0.0000
A=	Cu	0.0026	0.3464	0.0001
	S	0.3312	0.3494	0.3954

	sp	x_{sp}
x=	ccp	x_{ccp}
	po	x_{py}

	Zn	0.0100
b=	Cu	0.0366
	S	0.0613

Task is to find the solution for an unknown vector x. This is $x=A^{-1}b$, where A^{-1} is the matrix inverse of a square matrix A, i.e. $A \bullet A^{-1} = 1$, where 1 is identity matrix (all zeros except for ones on the diagonal).

When P=C equation is exactly determined (as many equations than unknowns). When C<P, then equation is underdetermined (fewer equations than unknowns), and when P<C, then overdetermined (more equations than unknowns), respectively. In two first cases there can be one or no solution. In the overdetermined cases there is, in general, no solution vector. It happens frequently that the best compromise solution is sought, the one that comes closest to satisfying all equations simultaneously.

Several methods of numerical mathematics exists for finding the solution for x. These include Gauss-Jordan elimination, LU decomposition and Singular Value Decomposition (SVD). The description of these methods is out of scope of this paper, and reader can find them in any textbook of numerical mathematics (e.g. Press et al. 1996).

The case in example 1 is exactly determined, and there exists one solution, which using singular value decomposition is

	sp	0.0169
x=	ccp	0.1055
	po	0.0476

This is in weight percentages: sphalerite 1.69 wt.%, chalcopyrite 10.55 wt.%, and pyrrhotite 4.76 wt.%.

2.3 *Real cases*
Real cases of nature are seldom ideal or thermodynamically in equilibrium. The number of minerals can be greater than the number of elements (e<m). Minerals tend to deviate from their stoichimetric compositions, and their compositions are not constant even in one sample. If a wide spectrum of elements has been analysed, the number of elements can be greater than the number of minerals (m<<e). Then A is not a square matrix, and the number of exactly right answers is zero (or in ideal case one).

Since weight fraction of mineral can not be negative (i.e. in given m $x_m \geq 0$), the task is to find the solution for x, which minimizes the residual vector R

$$R = |b - A \bullet x|$$

subject to

$$x_m \geq 0,$$

for all m.

Method for finding the solution in this case is called non-negative least square (Lawson & Hanson 1974).

2.4 *Analytical uncertainties*
Each single analysis or a group of analyses is subject to deviation and error. There are also other uncertainties related to knowns in matrix A and vector b. The mineral composition of the sample is not solved ambitiously, before this has been taken into account.

If the uncertainties are connected to vector b only, then a procedure called weighted least square can be applied (Lawson & Hanson 1974). As a systematic scheme for assigning the weights suppose that one can associate with each component, b_i, of the data vector b a positive number, ρ_I, indicating the approximate size of the uncertainty in b_i. If one has the appropriate statistical information about b, it would be common to take ρ_I to be the standard deviation of the uncertainty in b_i. then it would be usual to define weights by

$$g_{ii} = \frac{1}{\sigma_i}$$

or equivalently

$$w_{ii} = \frac{1}{\sigma_i^2}$$

Note that with this scaling all the components of the modified vector b* defined by

$$b_i^* = g_{ii} b_i = \frac{b_i}{\sigma_i}$$

have unit standard deviation.

Now the task is to solve x from the equation A•x=b*.

If the uncertainties and errors are connected to both matrix A and vector b (as it always in the nature is), then *Monte Carlo simulation* is the only way to make the estimation (Press et al. 1989). If the actual distribution law of measurement errors is known, synthetic data sets can be subjected to the actual set of equations. The procedure is to draw random numbers from appropriate distribution so as to mimic the best understanding of the measurement errors in matrix A and vector b. Knowns A and b are generated as many times as desired, and the solution for x from the equation $R = |b - A \bullet x|$ is searched each time separately. This way the probability distribution of vector x is mapped.

Rather than present all details of the probability distribution of errors in mineral quantities, it is a common practice to summarize the distribution in the form of confidence limits. In example 1, when the uncertainties related to knowns are considered, the correct way to report the mineral quantities is: mineral weight percentages are with 2σ (95.4%) probability as follows: sphalerite 1.72±0.04 wt.%, chalcopyrite 10.60±0.12 wt.%, and pyrrhotite 4.8±0.2 wt.%.

3. DISSOLUTION METHODOLOGIES

Chemical analyses can be classified in this context on the basis of their ability to discriminate different minerals as follows: 1) bulk analyses, 2) partial analyses, and 3) selective analyses. To determine these, new variable is introduced, $d_{e,m}^{s}$, which is the recovery of element e in mineral m by analysis method s, and $(0 \leq d_{e,m}^{s} \leq 1)$. In bulk analysis $d_{e,m}^{s} = 1$ for each e and m. In partial analysis $d_{e,m}^{s}$ is between zero and one for each e and m, but either it is in several cases not close to zero nor one, or it can vary from one case to another, or for several minerals and elements it is not exactly known. In selective analyses $d_{e,m}^{s}$ is close to zero or one for each e and m, and when deviating it is exactly known in each case.

3.1 *Bulk analyses*

There are only a few, often trivial instances, when it is possible to estimate accurately the mineralogical composition (or shorter mineral composition) of a material from the results of a bulk chemical analysis. It is clear that as samples become mineralogically complex it becomes more and more difficult to deduce their mineral compositions from sets of bulk chemical analyses. When bulk analyses are applied, the number of phases which can be solved can not be greater than the number of analysed elements (i.e. components).

3.2 *Selective dissolution methodologies*

Usually when the mineralogy of the base metal ores is studied samples are dissolved in mineral acids (e.g. nitric acid) and the concentration of elements of interest is determined by AAS or ICP. The dissolution of minerals, or more specifically, the dissolution of each element in mineral varies, then $b_{e}^{s} \leq b_{e}$, where superscript s refers to dissolution method.

When the effect of dissolution methods is considered, each $a_{e,m}$ has to be replaced by $c_{e,m}$ which is defined

$$c_{e,m} = a_{e,m} \bullet d_{e,m}^{s} \ ,$$

where $d_{e,m}^{s}$ is the recovery of element e in mineral m by dissolution method s and $(0 \leq d_{e,m}^{s} \leq 1)$. In matrix form task is to solve x from the equation C•x=b.

When several different dissolution methods are used, the number of components increases, because an element divides into several components. In favorable cases this means that the number of minerals, which can be calculated, increases, as well. If selective methods are highly selective (i.e. for each e and m $d_{e,m}^{s}$ is either zero or one), calculation becomes significantly more accurate.

Chemical phase analysis can be simple one-stage dissolution or multistage cascade dissolution, where more than one dissolution methods are applied stepwise (see later).

Development of selective dissolution methods requires a co-operation of chemist and mineralogist, and therefore the main work in this area has been done in the extractive metallurgy. The possibilities of selective dissolution methods have been overlooked during recent years, because of the progress of more sophisticated methods, i.e. image analysis. Therefore the most comprehensive summary of selective dissolution methods *Young's Chemical Phase Analysis* is relatively old, from 1970 (look also Young 1974). But still Young's procedures are actual, because they have been developed for rapid routine work in the laboratories of the mineral industry.

Some selective dissolution methodologies for porphyry copper and sulfidic deposits are represented in chapter 7.

3.3 *Combination of several selective techniques*
Mineral balancing is not limited to be done on the basis of chemical analyses only. Whatever parameter, which is a function of weight fraction of minerals (e.g. density, magnetic susceptibility, whiteness or XRD-peak intensities), can be used as a component. Also if the weight fraction of some mineral or mineral group is known from other sources, this can be taken as an additional equation. Even inaccurate numbers can be used, when the accuracy is considered and Monte Carlo simulation is applied in solving the equations. By mass balancing analysis values of different kind can be combined and used in solving the mineral composition of the sample. It is also possible, that there are some additional constraints besides $x_m \geq 0$.

When bulk chemical analysis, selective dissolution methods, quantitative diffraction, image analysis and possibly some other selective techniques are combined and equations are written, the number components (i.e. equations) becomes much greater than the number of minerals, which means that case is overdetermined, and most probably the solution becomes more accurate if the uncertainties related to each parameter are considered.

4. DERIVATIVES
Reliable mineral quantification enables to calculate some additional parameters from the samples, as well. These include distribution of given element, composition of given fraction and density of the sample.

4.1 *Distribution of elements*
In applied mineralogy one of the key numbers besides the weight fraction of minerals is the distribution of valuable element between the minerals present. Weight fraction of element e carried or hosted by mineral m, $h_{e,m}$, can be calculated from equation

$$h_{e,m} = \frac{x_m \cdot a_{e,m}}{\sum\limits_{m=1}^{P} (x_m \cdot a_{e,m})}$$

If the residual of the component e is zero, then nominator can be replaced by b_e. Weight fraction can be changed to weight percentage by multiplying $h_{e,m}$ by 100.

In example 1 the distribution of copper between minerals is: sphalerite 0.1%, chalcopyrite 99.9% and pyrrhotite 0.0%. Sulphur is distributed as follows: sphalerite 9.2%, chalcopyrite 60.2%, and pyrrhotite 30.7%.

4.2 Composition of given fraction

In extractive metallurgy it is often useful to know the mineral and chemical composition of given mineral fraction. Weight fraction of element e1 in mineral fraction m=1,...,n is

$$f_{e1,1-...-n} = \frac{\sum\limits_{m=1}^{n} (a_{e1,m} \cdot x_m)}{\sum\limits_{e=1}^{C} \sum\limits_{m=1}^{n} (a_{e,m} \cdot x_m)}$$

Weight fraction of mineral m1 in mineral fraction m=1,...,n is

$$f_{m1,1-...-n} = \frac{x_m}{\sum\limits_{m=1}^{n} x_m}$$

respectively.

In example one process engineer or metallurgist would be interested in knowing the chemical composition of hypothetical concentrate if sulfides were recovered in concentrate and all gangues were depressed into tail. Question can be answered, when mineral and chemical composition of the sulfide fraction is calculated. The result in weight percentages given below.

$f_{sp,sp\text{-}ccp\text{-}po}$ (wt.%)	10.0		$f_{Zn,sp\text{-}ccp\text{-}po}$ (wt.%)	5.9
$f_{ccp,sp\text{-}ccp\text{-}po}$ (wt.%)	62.1		$f_{Cu,sp\text{-}ccp\text{-}po}$ (wt.%)	21.5
$f_{po,sp\text{-}ccp\text{-}po}$ (wt.%)	28.0		$f_{Fe,sp\text{-}ccp\text{-}po}$ (wt.%)	36.0
Sum	100.0		$f_{S,sp\text{-}ccp\text{-}po}$ (wt.%)	36.0
			Sum	99.5

Note that the sum of elements does not correspond to 100. This is because the sum of the columns in matrix A does not correspond to 100.

For process engineer calculated result tells, that he or she has to depress pyrrhotite but especially sphalerite effectively and take care of adequate liberation of chalcopyrite to produce a salable copper concentrate. Also the production of separate zinc concentrate has to be considered.

4.3 Density
The density of the rock ρ can be calculated from the following equation

$$\frac{1}{\rho} = \sum_{m=1}^{P} \left(\frac{x_m}{\rho_m} \right)$$

when the weight fraction (x_m) and density (ρ_m) of each mineral is known. Note that the porosity has not been taken into account, and its' volume is assumed to be 0.

The density of sulfide fraction in example 1 is 4.43 g/cm^3. If the rest of the sample besides sulfides composes of material having the density of quartz, then the density of the sample is 2.84 g/cm^3.

5. COMPUTER AIDED CALCULATION
There exists several commercial and freeware programs or program libraries, which can be used to solve linear algebraic equations (e.g. MATLAB$^{®}$). It might be, however, more convenient to build up tailored package by linking freeware subroutines. For this purpose several good libraries are available: LINPACK, LAPACK, IMSL and NAG libraries, as examples. Press et al. (1989)[4] give several C- and FORTRAN programs for computational linear algebra. Furthermore they describe the theory behind the programs and guide for their proper use. Lawson and Hanson (1974) give FORTRAN routine for solving non-negative least square problems.

6. COMPARISON OF DESCRIBED TECHNIQUE TO OTHER RELATED TECHNIQUES
Above described method for mineral quantification is rapid, simple, cheap, and accurate. There are some problematic cases, where other quantification methods frequently fail to give accurate result, but selective dissolution methods work. Methods, which are based on physical separation (magnetic separation, heavy media separation) or optical identification

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[4] Numerical Recipes home page http://cfata2.harvard.edu/nr/nrhome.html

(point counting, image analysis) of minerals do not work in fine grain sizes (<5 λm). Fine fractions are important in many cases, because they regularly are the most problematic ones in mineral processing (e.g. in flotation), and accurate mineralogical data is required to minimize the losses.

Poorly crystalline phases (e.g. chrysocolla) can not be quantified by X-ray diffraction. Image analysis, which is based on electron beam techniques (EDS spectrum, back scattered electrons image) meets difficulties, when element of interest is distributed between several minerals, and especially when some of the main carriers contains variable trace quantities of the valuable element. When dealing with precious metals, detection limit and precision can cause problems in several methods.

Mineral balancing by selective dissolution methods works in most of the above described problematic cases. The accuracy depends on selectivity of the dissolution techniques. When the techniques are highly selective, precision of the quantification is almost as good as that of the chemical analysis itself. Precision is not dependent on grain size, and detection limits are low enough for trace and precious metals. Multiple distribution can be solved, if several dissolution methods and elements are combined. Described method gives mineral quantities directly as weight percentages, and therefore distribution of any given element, composition of given mineral fraction, and density can be calculated directly from the result.

Major disadvantage of the method based on dissolution techniques is the fact that it requires continuous co-operation with chemical and mineralogical laboratories. Although one can find several selective dissolution techniques from handbooks, the utilization of any method requires time demanding research and testing with known mineral samples before being a routine tool. Several parameters (e.g. leaching time, temperature, solution concentrations, solid-liquid separation technique) has to be fixed. If a suitable selective dissolution technique can not be found from the literature, the development of a new technique requires knowledge on mineral behavior in numerous "mineral acids" and mineral stability's as a function of pH.

7. CASE STUDIES

Following two examples illustrate the importance of reliable mineral quantification and effectiveness of mineral balancing by dissolution techniques. In both cases mineralogy has played a major role in process selection and development.

7.1 *Copper: Zaldivar porphyry copper ore*

The Zaldivar porphyry copper ore is located in northern Chile, about 1,400 km north of Santiago and 175 km east of Antofagasta at an altitude of 3100 m above sea level (Lamberg et al. 1996). Mineral resources (excluding the underlying hypogene material) include 425 million tons grading 0.78% TCu (total copper) and 0.30% ASCu (acid soluble copper) above a cut-off grade of 0.30 TCu. Mine was opened in late 1995 and during 19 years mine life 1.9 million tons of cathode copper will be produced by sulfide heap leaching, oxide heap leaching and dump leaching.

Zaldivar deposit can be divided roughly in four subhorizontal zones similarily as most of the supergene enriched porphyry copper deposits (e.g. Guibert & Park, 1986). Zones are from the surface: leached cap, oxide zone, supergene sulfide zone, and protore. Protore consists of varying amount of pyrite, chalcopyrite, molybdenite and bornite mainly as disseminations and in lesser degree in quartz veinlets. Cu grade is 0.2-0.4 wt.%.

Steady lowering of the water level produced the secondary copper oxide and chalcocite enrichment blanket. The leached and enrichment columns in two different orebodies of Zaldivar called the Main Zone and the Pinta Verde Zone are quite different. In the Main Zone high pyrite/chalcopyrite ratio (>2) and low neutralizing capacity of rhyolite produced thick leached cap and supergene enrichment blanket. The exchange of Cu in descending waters and Fe in pyrite produced thin (1-300 mm) "sooty chalcocite" (including chalcocite sensu stricto, djurleite, and digenite) coating on pyrite in the water table level. Some chalcocite precipitated also as veinlets.

In Pinta Verde Zone descending Cu rich solutions met reactive gangue minerals (i.e. plagioclase, chlorite, biotite, apatite), and as a consequence of released Si, Al, Fe, P, and Cl the precipitation of chrysocolla, pseudomalachite, libethenite, turquoise-chalcosiderite and atacamite took place. Cu oxide minerals occur mainly as fine grained (<5 mm) crusting on silicate and apatite grains as well as filling in the microcracs. Clay minerals like Mn oxide ("Cu wad"), Fe hydroxides and smectites contain typically >15, 1.7 and 2 wt.% absorbed Cu, respectively.

Leached cap is from few meters (Pinta Verde Zone) up to 250 m (Main Zone) thick with typical assemblage of iron hydroxide minerals in argillically altered rocks. The thickness of enrichment blanket varies from few meters to 250 m.

After the formation of the supergene enrichment blanket the groundwater table decented to a level below the blanket, and at the same time arid conditions did not provided enough precipitation to move the whole blanket downward to a new location at the deeper water table. Chalcocite blanket transformed to brochantite (and in places to antlerite, cuprite and other Cu oxides similar to Pinta Verde Zone) along the shear zones and in the upper part of the Main Zone supergene blanket.

Complex history has produced a diverse copper mineralogy in Zaldivar deposit. Therefore it has been important to determine reliably the distribution of Cu in different parts of the ore, and to find out how the Cu minerals behave in number of process alternatives considered. Different phases of Cu are usually examined by optical microscopy, image analysis, X-ray diffraction, heavy media separation, or other physical means. In Zaldivar these techniques faced serious problems, because of variable mineralogy of Cu (due to existence of mixed sulfide-oxide ores), frequently found poorly crystalline Cu phases, and the occurrence of copper in variable contents in clay minerals.

The need of rapid and reliable quantitative determination of the copper phases present led to the development of chemical procedures, based principally on selective dissolution. Two different sequential copper phases analyses were developed to quantify the Cu mineral composition in ore and process samples.

Sequential *Copper Phases Analysis* modified after Young (1974) aims to distinguish water soluble Cu phases, oxidic Cu minerals, secondary Cu sulfides and primary Cu sulfides. Cu Phase Analysis composes four successive dissolutions after each the filtrated residue continues to the next step and solution is analysed by AAS with respect to copper. 2 g of ground sample is first dissolved in water, after which the residue is leached in sulfuric acid. This is followed by potassium cyanide dissolution, and the last step is nitric acid decomposition. Scheme is highly selective for most of the Cu phases, as it can be seen in Fig.

1, which shows the extraction of copper from the most common copper minerals in each stage.

The exact amount of chrysocolla ($Cu_2H_2(Si_2O_5)(OH)_4$) and the content of copper it carries is problematic to estimate by any means. This is, however, extremely important because chrysocolla is not amenable to normal flotation processes (e.g. Aplan & Fuerstenau 1984, Raghavan et al. 1984) but it is easily leached. The density of chrysocolla is low (2.0-2.4 g/cm^3) and therefore if any classification takes place in sample preparation, analysed sample will be biased. Chrysocolla is brittle (hardness 2-4) and it easily breaks down when preparing a polished (thin) section. Optical identification is difficult, because it's optical properties varies widely (e.g. color from green and blue to black). The structure of chrysocolla is still poorly known (Van Oosterwyck-Gastuche and Gregoire 1971, Chukrov et al. 1968, Sun 1963, Newberg 1967) and it gives very faint peaks in X-ray diffraction due to its cryptocrystalline texture. Copper and H_2O content of chrysocolla varies widely. Special attention is needed when analysed by electron microprobe, because chrysocolla damages seriously under the electron bombardment.

To have reliable estimate on the amount of chrysocolla in Zaldivar ore samples, sequential *Oxidic Copper Phase Analysis* was developed. The method is highly selective for chrysocolla and Cu sulphates and oxychlorides, as shown in Fig. 2.

Chemical analysis of representative ore samples from the Main Zone are given in Table 1. Detected copper minerals and their Cu contents are given in Table 2. The mineral calculation matrixes for copper minerals are given in Table 3 and the result of the whole calculation in Table 4. Distribution of copper is illustrated in Fig. 3.

In Zaldivar ore types chrysocolla content varies from 0.04 to 2.03 wt.% (Table 4). Copper content of chrysocolla varies from 15.4 to 43.5 wt.% in analysed 23 grains in five samples average being 31.18 wt.% Cu. When this variation is taken into account and Monte Carlo simulation is used to define the chrysocolla content, for example in sample Oxide Andesite estimated chrysocolla content changes from 1.60 wt.% to 2.3±3.7 wt.% (actually to 2±4!) with 68.3% (1σ) probability. This would mean, that chrysocolla carries in that specific sample 0-100% of copper with 68.3% probability, which is far from the required accuracy or precision. Actually we are eager to know exactly the distribution of copper, and those numbers we can give with much better precision than the mineral weight percentages themselves. This is because *Copper Phase Analysis* and *Oxidic Copper Phase Analysis* give the copper content carried by each phase directly as weight percentages, which can be transferred directly to percentages. When this is considered, the result has to be expressed so that the weight percentage of *average chrysocolla* (which Cu content is 31.18 wt.% Cu) in sample Oxide Andesite is 1.60±0.02 wt.% with 68.3% (1σ) probability, and it carries 30.1% of the copper content of the sample. The result with this precision is very difficult to attain by any other available method.

7.2 *Nickel: Montcalm Ni deposit*
Montcalm Ni deposit is located in the Western Abitibi Subprovince, Ontario, Canada, some 50 kilometers west from Timmins (Barrie & Naldrett 1988). Semimassive ore is hosted by gabbroic body, which has undergone a high grade metamorphism and currently the most common mineral paragenesis in the ore hosting rocks is plagioclase (An_{78}), amphibole (actinolite - actionolitic hornblende) and chlorite with minor orhtopyroxene, phlogopite, calcite, and magnetite. Ore is semimassive and currently the reserves are 6.9 million tonnages

grading 1.6 wt.% Ni and 0.7 wt.% Cu. Chemical composition of typical ore sample is given in Table 5.

Sulfide mineral paragenesis in Montcalm Ni-Cu ore is pyrrhotite, pentlandite, chalcopyrite and pyrite. Pentlandite occurs in two ways: (1) as euhedral grains average 20-40 μm in diameter bound by pyrrhotite, and (2) as exsolution flames in pyrrhotite. Pentlandite, as other sulfides too, is practically fresh.

For determination of sulfidic nickel several methods have been (Czamanske and Ingamells 1970, Penttinen et al. 1977, Karapetyan 1968, Lynch 1971, Katsnel'son and Osipova 1960). Klock et al. (1986) tested six published analytical techniques for the selective chemical dissolution of sulfide-bound nickel. They recommended the bromine-methanol method (Penttinen et al. 1974) for assaying sulfide-Ni deposits when Ni is also present in silicate phases.

Mineral composition of typical ore sample, distribution of nickel and composition of the sulfide phase are given in Table 6. Calculation matrixes are shown in Table 7.

In Montcalm the product of high-grade Ni concentrate has been subject to investigations. The production of over 12 wt.% Ni for the ore given in Tables 5-6 requires the depression of pyrite. Pyrite and pentlandite can be separated in the flotation by controlling carefully the electrochemical condition on the mineral surfaces. The optimization requires a series of tests, the successfulness of which can not be estimated solely on the basis of Ni grade and recovery. The exact mineral balances are required for each of the sulfides, and therefore the application of mineral balancing by selective bromine methanol dissolution has been the key tool in the process development. As an example the results of one flotation test are shown in Fig. 4.

8. CONCLUDING REMARKS

Reliable mineral quantification is just one step in the mineralogical studies of ore deposit. On the basis of mineral weight fractions one can find boundary conditions for several questions, such as "How much of valuable metal can be recovered if these and these minerals are recovered, but those are depressed?", and "What will be the content of this harmful element if we fail to separate this and this mineral?" Exact answers require knowledge on particles to be processed, knowledge on their size distributions, compositions, and textures (see list in chapter 1) but in all the cases also some experimental work.

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10. ACKNOWLEDGEMENTS

11. NOTATIONS

ρ = density

$a_{e,m}$=weight fraction of component e in phase m

b_e=weight fraction of component e (in a sample)

$$c_{e,m} = a_{e,m} \bullet d_{e,m}^s$$ =weight fraction of component e in phase m when determined by method s

$d_{e,m}^s$ = recovery of element e in mineral m by analysis method s

$f_{e,m1\text{-}m2\text{-}m3\text{-}...\text{-}mn}$ = weight fraction of element e in mineral fraction m1-m2-m3-...-mn

$f_{m,m1\text{-}m2\text{-}m3\text{-}...\text{-}mn}$ = weight fraction of mineral m in mineral fraction m1-m2-m3-...-mn

$h_{e,m}$=weight fraction of element e carried (hosted) by mineral m

w=weight fraction

x_m=weight fraction of mineral m (in sample)

12. TABLE CAPTION

1. Chemical analysis of representative ore samples from the Main Zone of Zaldivar porphyry copper ore
2. Detected copper minerals of Zaldivar porphyry copper ore and their Cu contents.
3. The mineral calculation matrixes of Zaldivar
4. Calculated mineral compositoin of Zaldivar ore types
5. Chemical composition of typical ore sample from Montcalm Ni-Cu ore, Canada.
6. Calcualted mineral composition, composition of sulfide fraction and distribution of nickel of typical ore sample from Montcalm Ni-Cu ore.
7. Calculation matrixes of Montcalm ore sample

13. FIGURE CAPTION

1. Extraction of most common copper minerals in Copper Phase Analysis
2. Extraction of some copper minerals in Oxidic Copper Phase Analysis
3. Distribution of copper in ore types of Zaldivar porphyry copper deposit, Chile
4. Extraction of most common Ni bearing minerals in nitric acid, hydrochlorid acid and bromine-methanol dissolution methods.
5. Results of one representative flotation test presented as Ni grade vs. mineral recoveries.

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chc=chalchantite
ma=malachite
az=azurite
bro=brochantite
ata=atacamite
cco=chrysocolla
CuO=CuO
cup=cuprite
cc=chalcocite
cv=covellite
bn=bornite
cp=chalcopyrite
Cu=copper

Fig. 1. Extraction of copper from most common copper minerals in the Cu Phase Analysis

bro=brochantite
ata=atacamite
cco=chrysocolla
cup=cuprite
cc=chalcocite
cp=chalcopyrite
Cu=copper
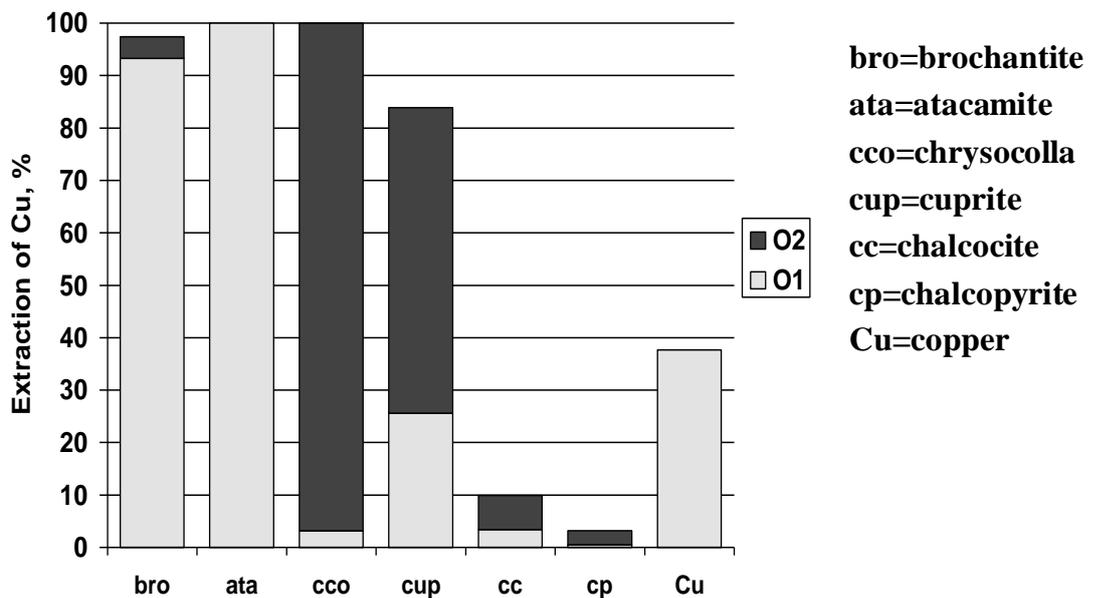
Fig. 2. Extraction of copper from most common copper minerals in the Oxidic Cu Phase Analysis
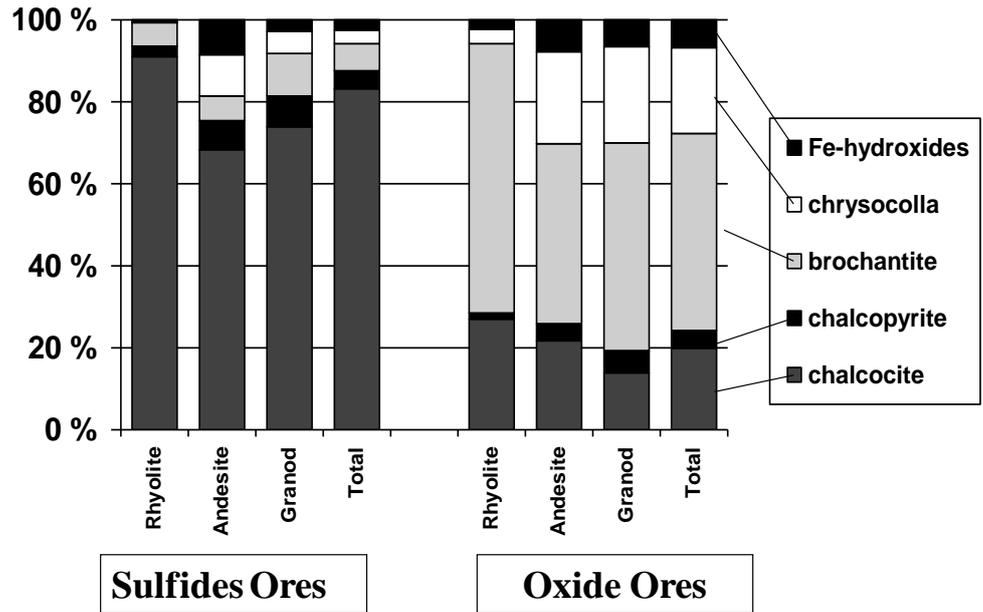
Fig. 3 Distribution of copper in ore types of Zaldivar porphyry copper deposit, Chile.
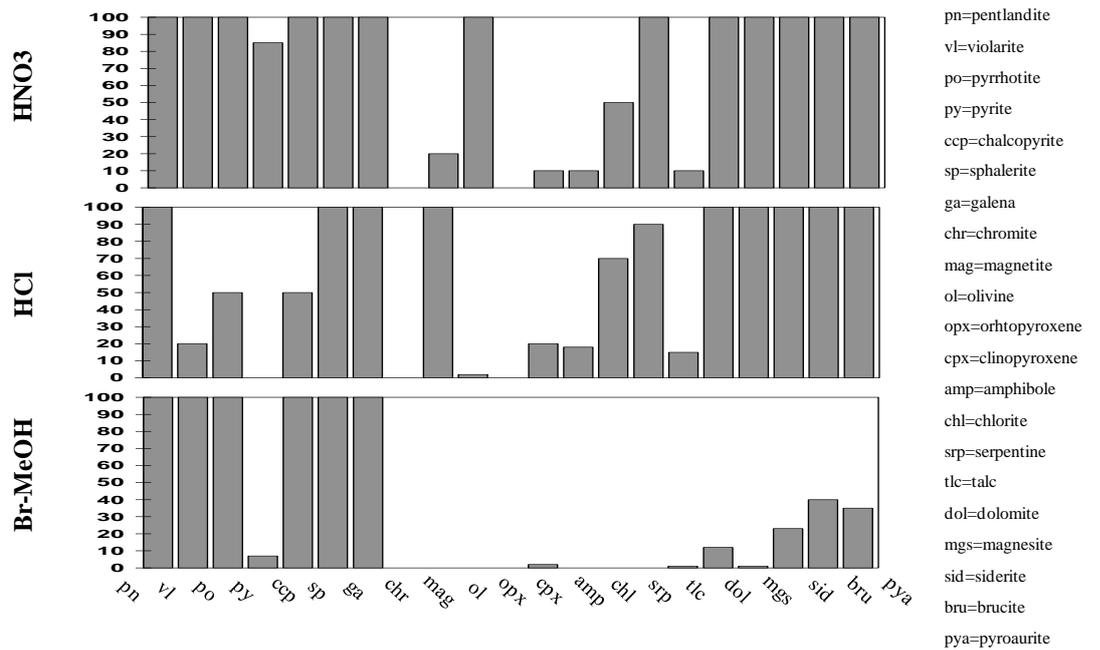
Fig. 4. Extraction of nickel from the most common minerals of Ni sulfide deposits in three different dissolution methods
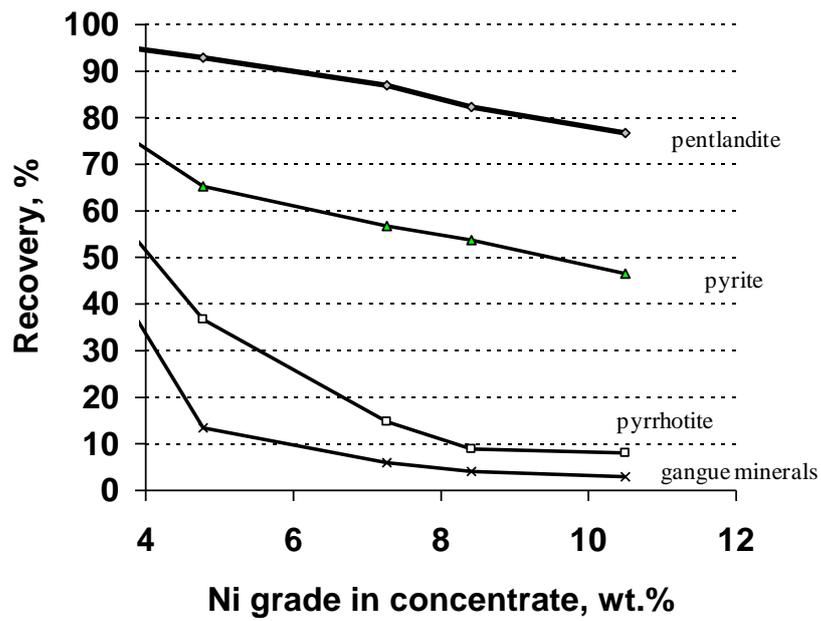
Fig. 5. Ni grade vs. mineral recovery (%) curves of one representative froth flotation test of Montcalm.

Table 1.   Chemical analyses of representative ore samples from the Main Zone of Zaldivar porphyry copper ore (wt.%).

Ore type	Oxide ores				Sulfide ores			
	Andesite	**Granod.**	**Rhyolite**	**TOT**	**Andesite**	**Granod.**	**Rhyolite**	**TOT**
SiO2	64.9	65.9	72.6	66.0	65.3	68.3	75.8	72.3
TiO2	0.494	0.482	0.280	0.469	0.507	0.430	0.183	0.297
Al2O3	17.66	16.70	14.18	17.02	17.46	16.31	13.09	14.63
FeOtot	4.29	4.39	2.65	4.16	4.90	4.17	2.70	3.38
MnO	0.040	0.066	0.023	0.046	0.048	0.049	0.021	0.031
MgO	1.25	0.89	0.35	1.05	1.50	0.71	0.25	0.59
CaO	0.61	0.59	0.09	0.55	0.80	0.39	0.09	0.28
Na2O	2.18	1.70	0.18	1.84	2.73	1.33	0.34	1.03
K2O	3.61	3.96	4.00	3.75	3.56	4.48	4.01	4.00
P2O5	0.173	0.216	0.109	0.179	0.184	0.113	0.075	0.105
CO2	0.41	0.31	0.46	0.38	0.29	0.37	0.36	0.35
Ni	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
Co	0.005	0.003	0.004	0.004	0.008	0.006	0.009	0.008
Zn	0.016	0.043	0.006	0.023	0.043	0.052	0.009	0.023
Pb	0.020	0.040	0.021	0.026	0.017	0.040	0.011	0.018
S	0.787	0.822	0.845	0.803	1.192	2.265	2.072	1.897
Mo	0.0066	0.0092	0.0842	0.0151	0.0085	0.0113	0.0135	0.0119
Cu XRF	1.742	2.103	2.093	1.885	0.972	1.342	1.324	1.239
Cu P1	0.005	0.000	0.008	0.004	0.000	0.000	0.000	0.000
Cu P2	1.100	1.570	1.495	1.281	0.218	0.271	0.148	0.183
Cu P3	0.503	0.350	0.505	0.457	0.678	1.009	1.205	1.046
Cu P4	0.049	0.066	0.027	0.052	0.050	0.065	0.030	0.040
Cu PSum	1.657	1.987	2.035	1.794	0.946	1.345	1.382	1.268
Cu AS	1.105	1.570	1.503	1.284	0.218	0.271	0.148	0.183
Cu sulf	0.552	0.417	0.532	0.509	0.728	1.074	1.234	1.086
Cu O1	0.60	0.94	1.36	0.78	0.04	0.14	0.13	0.11
Cu O2	0.60	0.64	0.27	0.58	0.26	0.23	0.17	0.20
Cu OSum	1.20	1.58	1.63	1.36	0.29	0.37	0.30	0.31

By XRF if not otherwise indicated; P1, P2, P3 and P4 = copper phase analysis steps 1 to 4; O1,O2 = Oxidic copper phase analysis; AS="acid soluble"; sulf="sulfidic"

Table 2.   **Copper bearing minerals of Zaldivar porphyry copper ore and their Cu contents.**

Mineral	Abbrev.	Formula	Copper, wt.%	
			avg	stoich

Main Zone

Mineral	Abbrev.	Formula	avg	stoich
Chalcocite	*cc*	*Cu2S*		*79.86*
Covellite	cv	CuS		66.47
Chalcopyrite	*ccp*	*CuFeS2*		*34.64*
Brochantite	*bro*	*Cu4(SO4)(OH)6*	*57.13*	*56.2*
Antlerite	ant	Cu3SO4(OH)4	53.52	53.74
Chrysocolla	*cco*	*CuSiO3.nH2O*	*31.18*	*38.13*
Cuprite	cup	Cu2O		88.9
Chalchantite	chc			
Wad	wad	aMnxOy.bCuO.cFe2O3	17.17	0
Fe-hydroxides	goe	FeOOH.nH2O	1.68	0
Chlorite-smectite	chl		3.19	0
Biotite-hydrobiotite	bt		2.09	0
Muscovite-smectite	ms		0.4	0
Kaolinite-smectite	kln		0.89	0

Additionally in Pinta Verde

Mineral	Abbrev.	Formula	avg	stoich
Pseudomalachite	pma	Cu5(PO4)2(OH)4H2O	53.39	53.51
Libethenite	lib	Cu2PO4(OH)	50.67	53.16
Turquoise	tq	Cu(Al,Fe)6(PO4)4(OH)4-8.1-4H2O	6.41	6.44-7.81

Main phases in bold and talic

Table 3.   Calculation matrixes for copper minerals.

		chc	cco	bro	cc	ccp
	Cu P1	0.2545	0.0000	0.0000	0.0000	0.0000
	Cu P2	0.0000	0.3118	0.5713	0.0000	0.0000
A =	Cu O1	0.0000	0.0000	0.5713	0.0000	0.0000
	Cu P3	0.0000	0.0000	0.0000	0.7986	0.0000
	Cu P4	0.0000	0.0000	0.0000	0.0000	0.3460

		OA		OL	OZ	O	SA	SL	SO	SZ	S
	Cu P1	0.005		0.000	0.008	0.004	0.000	0.000	0.000	0.000	0.000
	Cu P2	1.100		1.570	1.495	1.281	0.218	0.271	0.142	0.148	0.183
b =	Cu O1	0.602		0.936	1.361	0.778	0.038	0.144	0.060	0.134	0.114
	Cu P3	0.503		0.350	0.505	0.457	0.678	1.009	0.537	1.205	1.046
	Cu P4	0.049		0.066	0.027	0.052	0.050	0.065	0.027	0.030	0.040

		OA		OL	OZ	O	SA	SL	SO	SZ	S
	chc	0.0196		0.0000	0.0314	0.0149	0.0000	0.0000	0.0000	0.0000	0.0000
	cco	1.5972		2.0343	0.4298	1.6116	0.5773	0.4096	0.2620	0.0449	0.2200
x =	bro	1.0537		1.6378	2.3823	1.3618	0.0665	0.2512	0.1050	0.2340	0.1995
	cc	0.6299		0.4386	0.6324	0.5728	0.8490	1.2640	0.6721	1.5085	1.3099
	cp	0.1416		0.1916	0.0780	0.1503	0.1445	0.1870	0.0772	0.0858	0.1145

Note that b and x are in weight percentages and A in weight fractions.
Because the mineral composition is calculated for many samples using the same A matrix
b goes through from samples OA to S and the set of equations is solved each time separately.

Table 4.   Estimated mineral composition of Zaldivar ore types, wt.%.

Mineral / Ore type		Oxide ores				Sulfide ores			
		Andesite	**Granod.**	**Rhyolite**	**TOT**	**Andesite**	**Granod.**	**Rhyolite**	**TOT**
Chalchantite	chc	0.02	0.00	0.03	0.01	0.00	0.00	0.00	0.00
Chrysocolla	cco	1.60	2.03	0.43	1.61	0.58	0.41	0.04	0.22
Brochantite	bro	1.05	1.64	2.38	1.36	0.07	0.25	0.23	0.20
Chalcocite	cc	0.63	0.44	0.63	0.57	0.85	1.26	1.51	1.31
Chalcopyrite	ccp	0.14	0.19	0.08	0.15	0.14	0.19	0.09	0.11
Pyrite	py	0.89	0.82	0.74	0.86	1.63	3.37	2.89	2.68
Molybdenite	mb	0.011	0.015	0.141	0.025	0.014	0.019	0.023	0.020
Calcite	cal	0.93	0.71	1.05	0.87	0.66	0.85	0.81	0.79
Albite	ab	17.7	13.6	0.8	14.8	21.9	9.8	1.7	7.8
Anorthite	an	0.2	0.8	0.0	0.1	1.7	0.0	0.0	0.0
Muscovite	ms	37	41	41	39	36	45	40	41
Chlorite	chl	4.7	2.1	0.0	3.3	6.4	0.2	0.0	0.0
Kaolinite	kln	4	1	3	4	0	0	0	0
Quartz	qtz	31	35	52	35	31	41	56	48
Rutile	ru	0.4	0.4	0.2	0.4	0.4	0.4	0.1	0.2
Goethite	goe	3	4	2	3	3	2	0	1
Zircon	zir	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Sum		104	104	104	104	104	104	104	104

Table 5.   **Chemical composition of typical ore sample from Montcalm (wt.%). Analysis by XRF if not otherwise stated.**

SiO_2	32.0
TiO_2	0.122
Al_2O_3	10.6
Cr_2O_3	0.0651
V_2O_3	0.0082
FeOtot	29.2
MnO	0.136
MgO	7.85
CaO	5.88
Na_2O	0.92
K_2O	0.197
P_2O_5	0.017
CO_2 (Leco)	0.40
Cu	0.703
Ni	1.440
Co	0.064
Zn	0.010
Pb	0.001
S (Leco)	14.5
As	0.001
Fe (BrMeOH)	15.70
Cu (BrMeOH)	0.675
Ni (BrMeOH)	1.355
Co (BrMeOH)	0.033

Table 6.   **Estimated mineral composition, composition of the sulfide fraction and distribution of nickel of typical ore sample from Montcalm.**

		Mineral composition wt.%	Sulfide fraction %	Distribution of Ni %
Pyrrhotite	po	23.34	66.2	5.2
Pyrite	py	6.30	17.9	0.3
Pentlandite	pn	3.66	10.4	92.9
Chalcopyrite	ccp	1.94	5.5	0.0
Calcite	cal	0.91		0.0
Phlogopite	phl	2.4		0.2
Plagioclase	pl	23.9		0.0
Chlorite	chl	13.3		0.7
Amphibole	amp	21.5		0.7
Magnetite	mag	1.8		0.0
Sum		99.0	100.0	100.0
Fe			52.2	
Ni			3.9	
Cu			1.9	
Co			0.2	
As			0.0	
S			41.1	
Sum			99.4	

Table 7.   Mineral calculation matrixes of Montcalm. Calculation is divided in three rounds each continuing from the residue of the previous round. SVD is applied except round 3 is solved using weighted non-negative least squares method.

		po	py	pn	ccp
	S(Leco)	0.3953	0.5353	0.3346	0.3494
A(1) =	FeO(BrMeOH)	0.7600	0.0413	0.3917	0.3913
	Ni(BrMeOH)	0.0031	0.0000	0.3502	0.0000
	Cu(BrMeOH)	0.0001	0.0000	0.0000	0.3463

	S(Leco)	14.5
b(1)=	FeO(BrMeOH)	20.19
	Ni(BrMeOH)	1.355
	Cu(BrMeOH)	0.675

	po	23.3359
x(1)=	py	6.2982
	pn	3.6619
	ccp	1.9422

		cal	phl
A(2)=	CO2(Leco)	0.4397	0.0000
	K2O	0.0000	0.0816

b(2)=	CO2(Leco)	0.4
	K2O	0.197

x(2)=	cal	0.9098
	phl	2.4146

		pl	chl	amp
	Na2O	0.0245	0.0000	0.0044
	MgO	0.0016	0.2666	0.1761
A(3)=	Al2O3	0.3244	0.2121	0.0398
	SiO2	0.4745	0.2731	0.5175
	CaO	0.1580	0.0000	0.1187

	Na2O	0.9075
	MgO	7.6025
b(3)=	Al2O3	10.218
	SiO2	31.0982
	CaO	5.3389

	pl	23.8784
	chl	13.308
x(3)=	amp	21.5244

	Na2O	0.2278
	MgO	0.226
R(3)=	Al2O3	-1.2074
	SiO2	4.9946
	CaO	-0.9888

		mag
A(4)=	FeOtot	0.9309

b(4)=	FeOtot	1.6352

x(4)=	mag	1.7566