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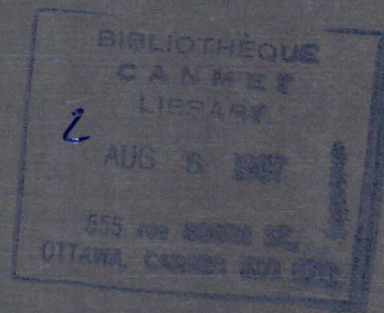
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SPOC

Simulated Processing of Ore and Coal



Chapter 3 Material Balance



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The **SPOC** Manual

Chapter 3 Material Balance

Material Balance Computation for Process Evaluation and Modelling

D. Laguitton and D. Hodouin

Editor: D. Laguitton

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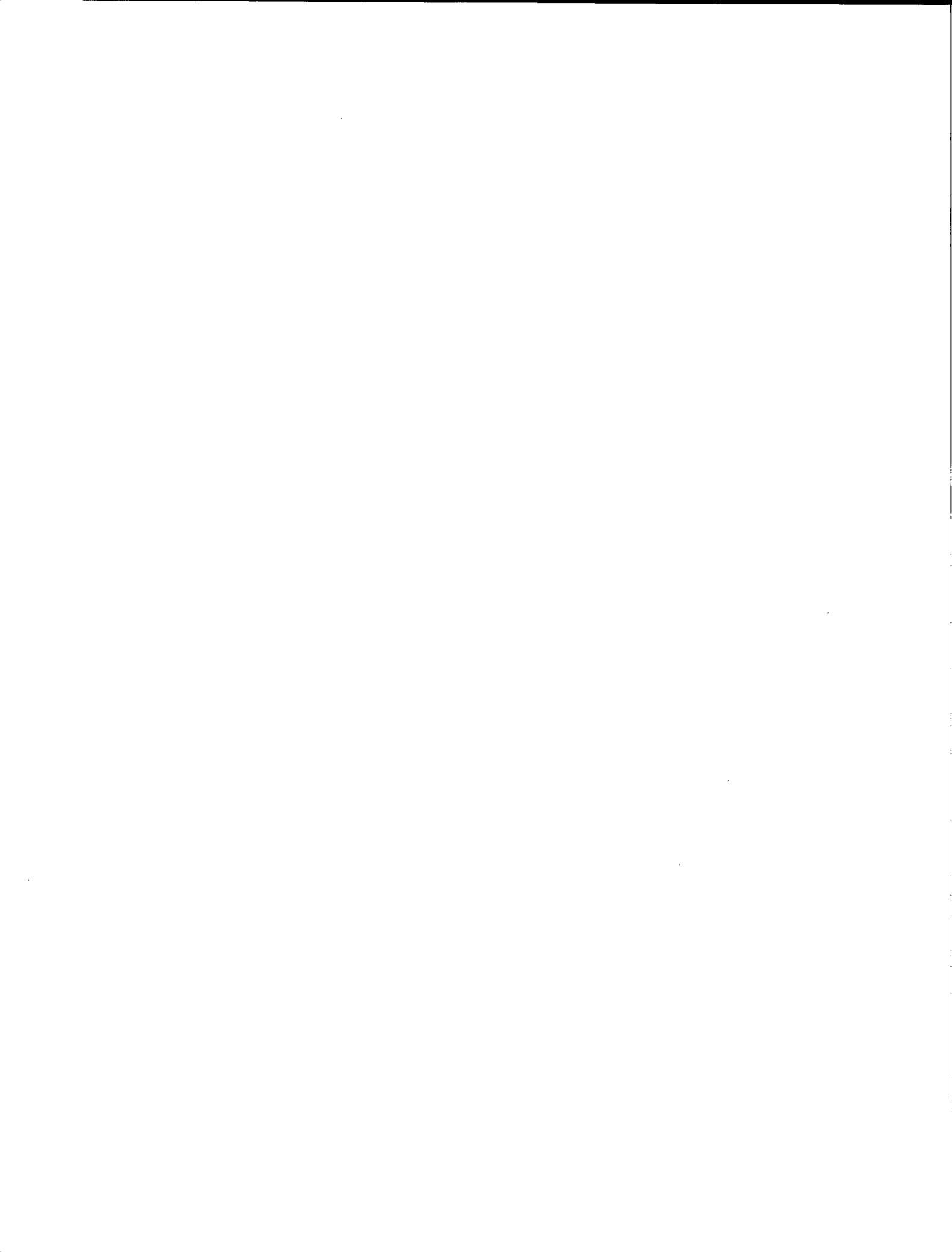
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THE SPOC MANUAL

The **SPOC*** manual consists of eighteen chapters, published separately. Their numbers and short titles are as follows:

- | | |
|------------------------------------|--------------------------------------|
| 1. Summary | 5. Unit Models: Part A |
| 2. Sampling Methodology | 5.1 Unit Models: Part B |
| 2.1 SAMBA Computer Program | 5.2 Unit Models: Part C |
| 2.2 Grinding Circuit Sampling | 6. Flowsheet Simulators |
| 3. Material Balance | 7. Model Calibration |
| 3.1 BILMAT Computer Program | 7.1 STAMP Computer Program |
| 3.2 MATBAL Computer Program | 7.2 FINDBS Computer Program |
| 4. Modelling and Simulation | 7.3 RTD and MIXERS Computer Programs |
| 4.1 Industrial Ball Mill Modelling | 8. Miscellaneous Computer Programs |

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FOREWORD

High energy costs and depleting ore reserves combine to make process evaluation and optimization a challenging goal in the 80's. The spectacular growth of computer technology in the same period has resulted in widely available computing power that can be distributed to the most remote mineral processing operations. The SPOC project, initiated at CANMET in 1980, has undertaken to provide Canadian industry with a coherent methodology for process evaluation and optimization assisted by computers. The SPOC Manual constitutes the written base of this methodology and covers most aspects of steady-state process evaluation and simulation. It is expected to facilitate industrial initiatives in data collection and model upgrading.

Creating a manual covering multidisciplinary topics and involving contributions from groups in universities, industry and government is a complex endeavour. The reader will undoubtedly notice some heterogeneities resulting from the necessary compromise between ideals and realistic objectives or, more simply, from oversight. Critiques to improve future editions are welcomed.

D. Laguitton
SPOC Project Leader
Canada Centre for Mineral and Energy Technology

AVANT-PROPOS

La croissance des coûts de l'énergie et l'appauvrissement des gisements ont fait de l'évaluation et de l'optimisation des procédés un défi des années 80 au moment même où s'effectuait la dissémination de l'informatique jusqu'aux concentrateurs les plus isolés. Le projet SPOC, a été lancé en 1980 au CANMET, en vue de développer pour l'industrie canadienne, une méthodologie d'application de l'informatique à l'évaluation et à l'optimisation des procédés minéralurgiques. Le Manuel SPOC constitue la documentation écrite de cette méthodologie et en couvre les différents éléments. Les retombées devraient en être une vague nouvelle d'échantillonnages et d'amélioration de modèles.

La rédaction d'un ouvrage couvrant différentes disciplines et rassemblant des contributions de groupes aussi divers que les universités, l'industrie et le gouvernement est une tâche complexe. Le lecteur notera sans aucun doute des ambiguïtés ou contradictions qui ont pu résulter de la diversité des sources, de la traduction ou tout simplement d'erreurs. La critique constructive est encouragée afin de parvenir au format et au contenu de la meilleure qualité possible.

D. Laguitton
Chef du projet SPOC,
Centre canadien de la technologie des minéraux et de l'énergie



ABSTRACT

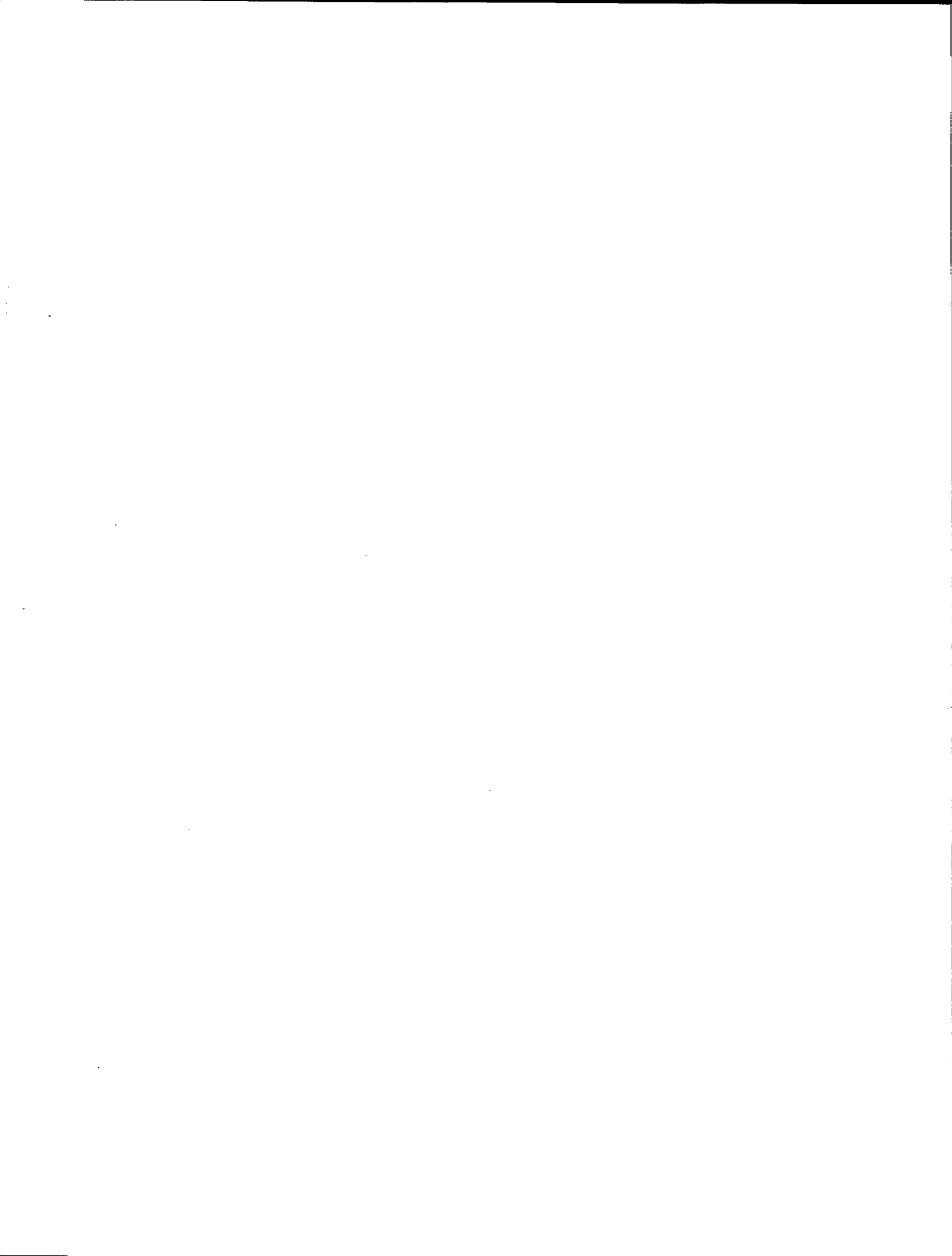
This chapter of the SPOC manual has been developed for the participants to a workshop on material balance computation. The various methods of material balance calculation are reviewed in order of increasing complexity. The two packages BILMAT and MATBAL are largely documented in Chapters 3.1 and 3.2 to which the reader is referred. The justification of methods requiring computers is presented on the basis of a search for best estimators, i.e., estimators that take into consideration as much experimental information as possible in order to provide "well informed" estimates. Each family of methods is illustrated by numerical exercises.

RÉSUMÉ

Ce chapitre du manuel SPOC a été écrit pour les participants à un atelier sur les bilans matière. Les diverses méthodes d'équilibrage de bilans matière y sont passées en revue par ordre de complexité croissante. Les deux programmes BILMAT et MATBAL sont amplement décrits dans les chapitres 3.1 et 3.2, auxquels le lecteur est renvoyé. Les méthodes requérant un ordinateur sont justifiées par la recherche d'estimateurs améliorés qui tiennent compte du maximum d'information possible. Des exercices numériques illustrent chaque groupe de méthodes.

ACKNOWLEDGEMENTS

The SPOC project has benefited from such a wide range of contributions throughout the industry, the university, and the government sectors that a nominal acknowledgement would be bound to make unfair omissions. The main groups that contributed are: the various contractors who completed project elements; the Industrial Steering Committee members who met seven times to provide advice to the project leader; the various users of project documents and software who provided feedback on their experience; the CANMET Mineral Sciences Laboratories staff members who handled the considerable in-house task of software development, maintenance, and documentation; the EMR Computer Science Centre staff who were instrumental in some software development; and the CANMET Publications Section. Inasmuch as in a snow storm, every flake is responsible, their contributions are acknowledged.



CONTENTS

THE SPOC MANUAL	i
FOREWORD	iii
AVANT-PROPOS	iii
ABSTRACT	v
RÉSUMÉ	v
ACKNOWLEDGEMENTS	v
1. INTRODUCTION	1
2. SYMBOLS AND DEFINITIONS	2
2.1 Symbols	2
2.2 Definitions	2
3. TRADITIONAL METHODS	3
3.1 Two-Product Formula	3
3.2 Three-Product Formula	3
3.3 n-Product Formula	3
3.4 Limitation of the n-Product Formula	4
4. METHODS OF IMPROVING ESTIMATES	7
4.1 Regressions	7
4.1.1 Orthogonal regression	7
4.1.2 Other regressions	7
4.1.3 Regression forced through the origin when $\bar{X}, \bar{Y} \neq 0,0$	8
4.2 Node Imbalance Minimization	8
4.3 Generalized Least-Squares Method	9
4.3.1 Lagrange multipliers	9
4.3.2 Weighting factors	10
5. ERROR STRUCTURE IN THE EXPERIMENTAL DATA	11
5.1 Components of the Total Error for an Assay Value	11
5.1.1 Fundamental error	11
5.1.2 Errors due to time and space variations in the lot	11
5.1.3 Errors due to sampler design and operation	11
5.1.4 Errors due to sample preparation and analysis	11
6. SENSITIVITY ANALYSIS	12
6.1 Introduction	12
6.2 The Analytical Method of Sensitivity Analysis	12
6.2.1 Principle	12
6.2.2 Factors influencing the variance estimates	12
6.2.3 Practical use of the estimate variances	13
6.2.3.1 Confidence Interval	13
6.2.3.2 Residuals analysis	13
6.3 The Monte-Carlo Analysis	13
7. MATERIAL BALANCE PROGRAMS	14
7.1 MATBAL: Iterative Lagrange Solution to the GLS Problem	14
7.2 BILMAT: Hierarchical Solution to the GLS Problem	14
7.2.1 Flowsheet variables	14
7.2.2 Flowsheet analysis	15
7.2.3 Mass conservation equations	15
7.2.4 Independent variables	15
7.2.5 Data redundancy	15
7.2.6 BILMAT algorithm	16
7.3 Node Imbalance + GLS Combination Methods	16
7.4 Miscellaneous	16

8. EXERCISES ON MATERIAL BALANCE COMPUTATION	17
8.1 Exercise 1: n-Product Formula	17
8.2 Exercise 2: Regression	17
8.3 Exercise 3	18
8.4 Exercise 4	18
8.5 Exercise 5	19
8.6 Exercise 6	19
8.7 Exercise 7: Sensitivity Analysis by the Monte-Carlo Method	19
8.8 Exercise 8: Sensitivity Analysis by the Analytical Method	19
9. ANSWERS TO EXERCISES	20
9.1 Exercise 1	20
9.2 Exercise 2	20
9.3 Exercise 3	20
9.4 Exercise 4	20
9.5 Exercise 5	20
9.6 Exercise 6	20
9.7 Exercise 7	21
9.8 Exercise 8	21
10. REFERENCES	22

TABLES

1. Symbols for material balance	2
2A. Hydrocyclone experimental data (non-cumulative)	5
2B. Hydrocyclone experimental data (cumulative passing)	6

FIGURES

1. One product	1
2. Two products	3
3. Three products	3
4. Four products	4
5. Hydrocyclone. $CIR = \frac{U}{0}$	5
6. Raw data 4" hydrocyclone (non-cumulative)	7
7. Regressions on hydrocyclone non-cumulative data	8
8. Regressions on hydrocyclone cumulative data	8
9. Principle of the two-error estimators	12
10. Various levels of the mass balance variables	14
11. Sample flowsheet for data redundancy test	15

1. INTRODUCTION

The material balance problem in a process flowsheet arises from the necessity for the process engineer to make periodical inventories of the distribution of material through the various process streams. It may be dictated by bookkeeping requirements (e.g., concentrate inventories, daily, monthly, yearly), by trouble shooting or process control requirements (e.g., circulating load calculations), by environmental requirements (e.g., tailing flow rates and assays) or by other requirements. This problem is also designated as a "mass balance", "metallurgical inventory", or "process audit".

Ideally, if one could measure the true value of every assay and flow rate in a flowsheet, the material balance problem would not exist. However, the reality of process instrumentation is such that 1) only a few process variables can be measured and these are mostly assays, 2) the experimental estimates of these variables are subject to errors from various sources and do not satisfy mass balance principles.

The mass balance principle is that in our usual environment, the total mass of material undergoing a physical or chemical transformation remains constant. This stops being true in relativistic mechanics where mass can be converted into energy and vice versa.

In all mineral processes therefore, the mass of consumed elements is equal to that of produced elements. For instance, the mass of copper or chalcopyrite entering a mill or a cyclone or a flotation cell per unit of time is equal to that leaving the same unit per unit of time provided the steady-state conditions have been reached.

In a single unit such as shown in Figure 1, if W_i represents the solid flow rate of stream i , and X_j an assay for

species i in the solid of stream j , the following mass conservation equations are valid:

$$W_1^* = W_2^*$$

$$W_1^* X_1^i = W_2^* X_2^i$$

where the asterisk denotes the *true* value of the variables.

In practice, one can only measure an experimental estimate of W_j and X_j and it is essential to keep in mind that its use as a *true* value is a decision that bears consequences on the confidence level associated with any figure resulting from such an assumption. Intuitively it is obvious that the better the experimental precision, the more valid the assumption that it is the best estimate. In practice, we will see that better estimates than W_j and X_j can be calculated. They will be called \hat{W}_j and \hat{X}_j , the *maximum likelihood estimates*.

The material balance calculations can be divided into two families; one where the experimental estimates are taken as best estimates, and the other where improved estimates are calculated. The following discussion will cover successively the traditional methods corresponding to the former case, and more recent methods corresponding to the latter.



Fig. 1 - One product

2. SYMBOLS AND DEFINITIONS

2.1 SYMBOLS

Symbols are to equations what words are to a text. In all likelihood two different authors will select different symbols to represent the same theory unless a general convention has been established through years of practice in a field of science.

Such conventions do not exist in material balance computation. The two packages MATBAL and BILMAT have evolved from two distinct groups and suffer from this heterogeneity. For instance, the symbol for flow rates in MATBAL is W (for Weight) while it is D in BILMAT (for Débit, French for Flow Rate). The MATBAL symbols were kept as in the original papers by Wiegel et al. in order to maintain a coherence within a family of programs and papers. Only through years of practice will these differences vanish.

The following Table 1 is an attempt to define uniform series of symbols for a material balance context and will be adhered to in this chapter.

Table 1 – Symbols for material balance

W	general symbol for flow rate
W_p	pulp flow rate
W_s	solid flow rate
W_w	water flow rate
F	feed flow rate of a cyclone
O	overflow flow rate of a cyclone
U	underflow flow rate of a cyclone
f_i, o_i, u_i	assay i of the feed, overflow or underflow
I _j	imbalance for element i across node j
$R_j^w, R_j^p, R_j^s, R_j^k$ R_j^k (or $\Delta X_j \Delta X$)	residual in stream j for water, pulp, solids, assay i or assay k in class i.
E_j^w, E_j^p, E_j^s , E_j, E_j^k	error in stream j for water, pulp, etc.
X_j^k	assay for element k in class i in branch j
X_j^{*k}	true value (any symbol with asterisk)
\hat{X}_j^k	estimate (any symbol with hat)
σ	standard deviation, e.g., σ_j^p
s	estimate of σ , e.g., s_j^p
\hat{V}	variance (or σ^2)
V	estimate of V (or S^2)
S_p, S_L	solid weight fraction in pulp, in liquid
L_p, L_s	liquid weight fraction in pulp, in solid
100 S_p	per cent solid

Note: To avoid confusion with exponents, assay X_j^k is represented by X_i for $i = 1$, X_i' for $i = 2$, X_i'' for $i = 3$, etc. In practical examples, one can use Cu_i, Zn_i, Pb_i, Fe_i , etc.

2.2 DEFINITIONS

Chapters 3.1 and 3.2 contain their own glossary of terms. For the purpose of this chapter the following are useful.

Random or stochastic variable

A variable which can take different values from one measurement to another, depending on the random error.

Deterministic, or error-free, variable

A variable which can be determined without random error (e.g., number of increments in a composite sample).

True-value of a random variable

That value which cannot be exactly determined.

Estimate

The value of a variable obtained via an estimator.

Estimator

A method to try to access the true value of a variable.

Maximum likelihood estimates

In the case of a material balance problem based on normal error models, these are the **minimum variance estimates** of the process variables. They are the best available estimates.

Error

The difference between the true value and the estimate.

Residual

The difference between the estimate and the experimental value.

Probability distribution

A graph or table giving the probability that an estimate of a variable takes a given value.

Normal probability distribution (or gaussian, or bell shaped)

A distribution characterized by two parameters; its mean and its standard deviation. It is usually followed by random errors resulting from several contributing errors.

Biased estimator

An estimator is said to be biased when the mean value of the estimate it produces does not approach the true value as the number of estimations increases.

3. TRADITIONAL METHODS

These are known under the name of n-product formulae. They consist in calculating n-product variables around a process unit where experimental variables can be measured. Usually the unknown variables are the flow rates and the known variables are the assays. The symbol, *n*, refers to the number of streams in which the flow rate must be calculated. In the following, *W* represents the flow rate of interest and could apply to pulp, solid, or liquid depending on which is analyzed.

The simplest case is the one-product-formula valid for a simple unit, such as represented in Figure 1. The valid equations are:

$$W_1 = W_2$$

$$W_1 X_1^i = W_2 X_2^i$$

The variables are: W_1, W_2, X_1^i, X_2^i

In order to calculate, for example W_2 , it is only necessary to know W_1 . No assay X_1^i is required. Similarly, X_2^i is known if only X_1^i is known. In both cases, the value of the calculated variable has the same error as that of the measured variable.

3.1 TWO-PRODUCT FORMULA

A two-product problem corresponds to the illustration in Figure 2. The valid equations are:

$$W_1 = W_2 + W_3$$

$$W_1 X_1^i = W_2 X_2^i + W_3 X_3^i$$

These two equations allow us to calculate a maximum of two unknowns. W_2 and W_3 are the usual variables to be calculated. By simple elimination one can easily show that:

$$W_3 = \frac{X_1 - X_2}{X_3 - X_2} W_1 \text{ and } W_2 = \frac{X_1 - X_3}{X_2 - X_3} W_1$$

It is important to note that all conservation equations remain valid to a scale factor, i.e., if the total mass of material in the circuit is, for example, doubled, the above formulae apply and it is therefore customary to express all flow rates as a ratio to a feed of 1 or 100 flow units.



Fig. 2 - Two products

Note that for a two-product unit, all streams can be determined if one assay is known in each stream.

3.2 THREE-PRODUCT FORMULA

In a three-product situation, as in Figure 3, the valid equations are:

$$W_1 = W_2 + W_3 + W_4$$

$$W_1 X_1 = W_2 X_2 + W_3 X_3 + W_4 X_4$$

If the three-product streams' flow rates are to be calculated, one must measure a second assay X_1^i in all streams in order to obtain a third equation:

$$W_1 X_1^i = W_2 X_2^i + W_3 X_3^i + W_4 X_4^i$$

The same elimination technique used above gives the following results:

$$\frac{W_4}{W_1} = \frac{(X_1^i - X_2^i)(X_3 - X_2) + (X_1 - X_2)(X_2^i - X_3^i)}{(X_4^i - X_2^i)(X_3 - X_2) + (X_4 - X_2)(X_2^i - X_3^i)}$$

and others by permutation: e.g.,

$$\frac{W_3}{W_1} = \frac{(X_1^i - X_2^i)(X_4 - X_2) + (X_1 - X_2)(X_2^i - X_4^i)}{(X_3^i - X_2^i)(X_4 - X_2) + (X_3 - X_2)(X_2^i - X_4^i)}$$

Note that for a three-product unit, all streams can be determined if two assays are known in each stream.

3.3 n-PRODUCT FORMULA

As a generalization of the above cases, an n-product problem can be solved if n-1 assays are known on each stream. As n grows bigger, the substitution method of solving the equation becomes cumbersome and the determinant method can advantageously be used.

This method simplifies the solution of a system of linear equations to a calculation of determinants.

Consider the system:

$$a_1 x_1 + b_1 x_2 + c_1 x_3 = d_1$$

$$a_2 x_1 + b_2 x_2 + c_2 x_3 = d_2$$

$$a_3 x_1 + b_3 x_2 + c_3 x_3 = d_3$$

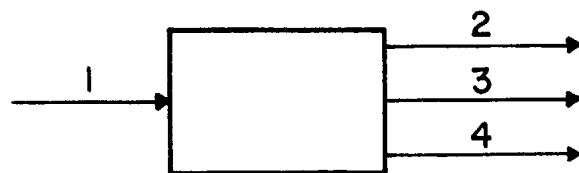


Fig. 3 - Three products

It can also be written in matrix notation:

$$M X = D$$

with

$$M = \begin{pmatrix} a_1 & b_1 & c_1 \\ a_2 & b_2 & c_2 \\ a_3 & b_3 & c_3 \end{pmatrix}$$

$$X = \begin{pmatrix} x_1 \\ x_2 \\ x_3 \end{pmatrix}$$

$$D = \begin{pmatrix} d_1 \\ d_2 \\ d_3 \end{pmatrix}$$

By definition, the determinant of matrix M is:

$$\det M = \begin{vmatrix} a_1 & b_1 & c_1 \\ a_2 & b_2 & c_2 \\ a_3 & b_3 & c_3 \end{vmatrix} = a_1 (b_2 c_3 - b_3 c_2) - b_1 (a_2 c_3 - a_3 c_2) + c_1 (a_2 b_3 - a_3 b_2)$$

Any determinant can be calculated along any row or column by carefully alternating the +1 and -1 factors in front of the various terms. The solution of the system of equations $M X = D$ is given by:

$$x_i = \frac{M_i^*}{M}$$

where M_i^* designates the determinant of matrix M after column i has been replaced by the values in D.

This can be illustrated by an example from Taggard's handbook of mineral dressing, (pages 12 - 123), for a four-product circuit.

The following assays are known:

	Pb %	Zn %	Cu %
Feed	7.1	5.7	2.26
Pb conc	60	1	1
Zn conc	1	40	1
Cu conc	2	2	10
Tail	1	2	0.1

The valid equations, according to Figure 4, are:

$$W_1 = W_2 + W_3 + W_4 + W_5$$

$$7.1 W_1 = 60 W_2 + W_3 + 2 W_4 + W_5$$

$$5.7 W_1 = W_2 + 40 W_3 + 2 W_4 + 2 W_5$$

$$2.26 W_1 = W_2 + W_3 + 10 W_4 + 0.1 W_5$$



Fig. 4 - Four products

using the determinant method:

$$W_2 = \frac{\begin{vmatrix} 1 & 1 & 1 & 1 \\ 7.1 & 1 & 2 & 1 \\ 5.7 & 40 & 2 & 2 \\ 2.26 & 1 & 10 & 0.1 \end{vmatrix}}{\begin{vmatrix} 1 & 1 & 1 & 1 \\ 60 & 1 & 2 & 1 \\ 1 & 40 & 2 & 2 \\ 1 & 1 & 10 & 0.1 \end{vmatrix}} = \frac{2216.07}{22160.7} = 0.1$$

$$W_3 = \frac{\begin{vmatrix} 1 & 1 & 1 & 1 \\ 60 & 7.1 & 2 & 1 \\ 1 & 5.7 & 2 & 2 \\ 1 & 2.26 & 10 & 0.1 \end{vmatrix}}{22160.7} = \frac{2216.02}{22160.7} = 0.1$$

and similarly: $W_4 = 0.2$, $W_5 = 0.60$

3.4 LIMITATION OF THE n-PRODUCT FORMULA

The limitation of the n-product formula comes from the fact that the calculated variables are influenced by the precision of the observed variables in a non-quantified way. Consequently, when more than n-1 assay types are known in each stream each subset of an n-1 assay will give a different answer which can discredit the results completely to the plant engineer's eyes. Let's, for instance, consider a two-product circuit where the following data are known:

	Cu %	Zn %	Fe %
1. Feed	0.163	3.93	11.57
2. Conc	0.657	52.07	14.67
3. Tail	0.140	0.49	13.09

Although three assay types have been measured in each stream, only one is required to calculate the concentrate and tailing flow rates; W_2 and W_3 by the two-product formula.

For a feed of 100 t/h, the results corresponding to each mode of calculation are:

$$\begin{aligned} \text{with Cu data: } W_2 &= 4.5 & W_3 &= 95.5 \\ \text{with Zn data: } W_2 &= 6.7 & W_3 &= 93.3 \\ \text{with Fe data: } W_2 &= -96.2 & W_3 &= 196.2 \end{aligned}$$

These three sets of results are quite different due to the large variations in assay values and precisions.

A conventional error calculation performed on the two-product formula gives the following result:

$$\frac{\Delta W_2}{W_2} \leq \frac{\Delta W_1}{W_1} + \frac{\Delta X_1 + \Delta X_3}{X_1 - X_3} + \frac{\Delta X_2 + \Delta X_3}{X_2 - X_3}$$

The relative error on a calculated flow rate depends, therefore, not only on the magnitude of the errors on all assays but also on the differences, $X_1 - X_3$ and $X_2 - X_3$, which are proportional to the quality of the separation being performed.

For an ideal separation, one has:

$$X_3 = 0 \text{ and } \frac{\Delta W_2}{W_2} \leq \frac{\Delta W_1}{W_1} + \frac{\Delta X_1}{X_1} + \frac{\Delta X_2}{X_2}$$

If all assays are known with a 6.0% accuracy in the example above, the maximum relative error on W_2 is found to be 19.2% with Cu assays, 13.1% with Zn, 156% with Fe.

A more realistic estimate of the sensitivity of the solution to assay errors can be obtained by a technique called Monte-Carlo Simulation which requires a computer. In this method, a perturbation similar to the suspected random error present in all assays is imposed on the measured value in order to simulate a set of synthetic assay values. These are used for an n-product solution and the process is repeated enough times to accumulate a number of possible solutions. The mean and standard deviation of these possible solutions can be calculated.

This method, applied to the problem above, gave the following results:

$$\frac{\Delta W_2}{W_2} \approx \begin{matrix} 72\% \text{ with Cu assays,} \\ 8\% \text{ with Zn,} \\ 1000\% \text{ with Fe} \end{matrix}$$

The problem of selecting the best subset of assays in order to calculate the unknown variables becomes especially acute when the assays consist of size distributions with up to a dozen intervals or more. Process evaluation around a hydrocyclone is typical of this situation as shown in Figure 5.

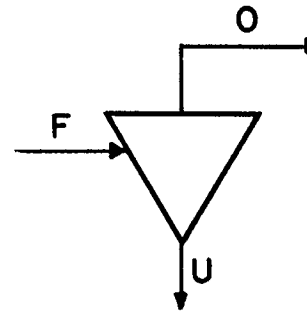


Fig. 5 - Hydrocyclone. $CIR = \frac{U}{O}$

In most cases, the variable of interest is the circulating load ratio U/O , i.e., the ratio of solid flow rates in the underflow and the overflow respectively. Using the two-product formula, with f_i , u_i , and o_i , the non-cumulative per cent retained on size interval i , one has:

$$CIR_i = \frac{U}{O} = \frac{f_i - o_i}{u_i - f_i}$$

each interval i gives a different value of CIR , as shown in Table 2.

Having so many possible estimates of CIR , one can start thinking of a method to determine a best estimate. Several methods of obtaining such an estimate are subsequently described.

Table 2A - Hydrocyclone experimental data (non-cumulative)

F	U	O	F-U	O-F	(F-U)(F-U)	(O-F)(O-F)	(F-U)(O-F)	CIR
3.97	5.95	0.00	-1.98	-3.97	3.92	15.76	7.86	2.01
15.25	21.45	0.00	-6.20	-15.25	38.44	232.56	94.55	2.46
14.80	20.70	0.00	-5.90	-14.80	34.81	219.04	87.32	2.51
9.71	13.05	0.00	-3.34	-9.71	11.16	94.28	32.43	2.91
8.27	11.05	0.80	-2.78	-7.47	7.73	55.80	20.77	2.69
4.70	6.03	0.00	-1.33	-4.70	1.77	22.09	6.25	3.53
10.06	9.44	0.00	0.62	-10.06	0.38	101.20	-6.24	-16.23
4.01	4.53	1.82	-0.52	-2.19	0.27	4.80	1.14	4.21
2.25	1.56	6.61	0.69	4.36	0.48	19.01	3.01	6.32
1.92	1.23	16.83	0.69	14.91	0.48	222.31	10.29	21.61
2.26	1.04	22.32	1.22	20.06	1.49	402.40	24.47	16.44
2.56	0.50	20.70	2.06	18.14	4.24	329.06	37.37	8.81
2.67	0.99	16.04	1.68	13.37	2.82	178.76	22.46	7.96
3.07	0.65	9.85	2.42	6.78	5.86	45.97	16.41	2.80
3.38	0.31	3.21	3.07	-0.17	9.42	0.03	-0.52	-0.06
3.63	0.56	1.39	3.07	-2.24	9.42	5.02	-6.88	-0.73
2.29	0.23	0.42	2.06	-1.87	4.24	3.50	-3.85	-0.91
5.20	0.73	0.01	4.47	-5.19	19.98	26.94	-23.20	-1.16
SUMS								
100.00	100.00	100.00	0.00	0.00	156.92	1978.52	323.64	65.17
MEANS								
5.56	5.56	5.56	0.00	0.00	8.72	109.92	17.98	3.62

Table 2B – Hydrocyclone experimental data (cumulative passing)

F	U	O	F-U	O-F	(F-U)(F-U)	(O-F)(O-F)	(F-U)(O-F)	CIR
96.03	94.05	100.00	1.98	3.97	3.92	15.76	7.86	2.01
80.78	72.60	100.00	8.18	19.22	66.91	369.41	157.22	2.35
65.98	51.90	100.00	14.08	34.02	198.25	1157.36	479.00	2.42
56.27	38.85	100.00	17.42	43.73	303.46	1912.31	761.78	2.51
48.00	27.80	99.20	20.20	51.20	408.04	2621.44	1034.24	2.53
43.30	21.77	99.20	21.53	55.90	463.54	3124.81	1203.53	2.60
33.24	12.33	99.20	20.91	65.96	437.23	4350.72	1379.22	3.15
29.23	7.80	97.38	21.43	68.15	459.24	4644.42	1460.45	3.18
26.98	6.24	90.77	20.74	63.79	430.15	4069.16	1323.00	3.08
25.06	5.01	73.94	20.05	48.88	402.00	2389.25	980.04	2.44
22.80	3.97	51.62	18.83	28.82	354.57	830.59	542.68	1.53
20.24	3.47	30.92	16.77	10.68	281.23	114.06	179.10	0.64
17.57	2.48	14.88	15.09	-2.69	227.71	7.24	-40.59	-0.18
14.50	1.83	5.03	12.67	-9.47	160.53	89.68	-119.98	-0.75
11.12	1.52	1.82	9.60	-9.30	92.16	86.49	-89.28	-0.97
7.49	0.96	0.43	6.53	-7.06	42.64	49.84	-46.10	-1.08
5.20	0.73	0.01	4.47	-5.19	19.98	26.94	-23.20	-1.16
SUMS								
603.79	353.31	1064.40	250.48	460.61	4351.56	25859.50	9188.98	24.29
MEANS								
35.52	20.78	62.61	14.73	27.09	255.97	1521.15	540.53	1.43

4. METHODS OF IMPROVING ESTIMATES

Confronted with a situation where a method provides several estimates for some process variables, two different avenues are open to produce unique best estimates. One consists in taking the means as the most efficient estimates of the variables; the other in using a different method of estimation which would give a unique estimate of each variable and could be justified as giving the best estimate.

4.1 REGRESSIONS

As discussed above, a typical case with several estimates available is the calculation of the circulating load ratio of a hydrocyclone when the size distribution of its feed and product streams is known. The two-product formula gives:

$$CIR_i = \frac{U}{O} = \frac{f_i - o_i}{u_i - f_i}$$

The mean of all values obtained for the n size intervals is often used as a best estimate:

$$CIR = \frac{\sum CIR_i}{n}$$

or
$$CIR = \frac{\sum (f_i - o_i)}{\sum (u_i - f_i)}$$

It must be noted that the size distributions used in conjunction with these formulae can be cumulative or non-cumulative. The non-cumulative is preferred because of a less complex error correlation among the various sizes. Table 2 gives the results obtained with both formulae.

Another method of extracting a best estimate of CIR is to plot the values of $f_i - o_i$ against those of $u_i - f_i$. The slope of the best fit curve to those values can be taken as a best estimate of CIR. This can be done graphically as shown in Figure 6.

If non-cumulative values are used, the graph necessarily goes through the origin since the mean values of $f_i - o_i$ and $u_i - f_i$ are zero, and a best fit curve always goes through the mean coordinates of its points.

The calculation of the best fit curve can also be done by regression, using a computer program such as STAMP (1).

The regression consists in calculating the best estimates \hat{y}_i of variables $y_i = f_i - o_i$, and/or $\hat{x}_i = u_i - f_i$, as those which minimize a figure of merit or minimization criterion which can be one of the following:

1. Regression of y on x : $\sum (\hat{y} - y)^2$
2. Regression of x on y : $\sum (\hat{x} - x)^2$
3. Regression of x and y : $\frac{\sum (a + bx - y)^2}{(p - b)^2} (p^2 + 1)$
4. Regression of orthogonal: $\sum h_i^2 = \frac{\sum (a + bx - y)^2}{b^2 + 1}$

h_i = distance from point i to regression curve

p = split coefficient

b = slope of regression line

The regression coefficients for a linear regression $Y = a + bX$ are obtained by the following formulae:

4.1.1 Orthogonal Regression

$$\text{Slope} = b = \frac{\sqrt{e^2 + g^2} - e}{g}$$

where

$$e = (\sum Y_i)^2 - (\sum X_i)^2 - n(\sum Y_i^2 - \sum X_i^2)$$

$$g = 2(n\sum X_i Y_i - \sum X_i \sum Y_i)$$

$$\text{intercept} = a = \frac{\sum Y_i - b\sum X_i}{n}$$

4.1.2 Other Regressions (Y on X, X on Y, on X and Y)

$$\text{Slope} = b = \frac{\sum Y_i - na}{\sum X_i} \quad \text{if } \sum X_i \neq 0$$

$$b = \frac{(\sum Y_i)^2 - n\sum Y_i^2 + np\sum X_i Y_i}{np\sum X_i^2 - n\sum X_i Y_i} \quad \text{if } \sum X_i = 0$$

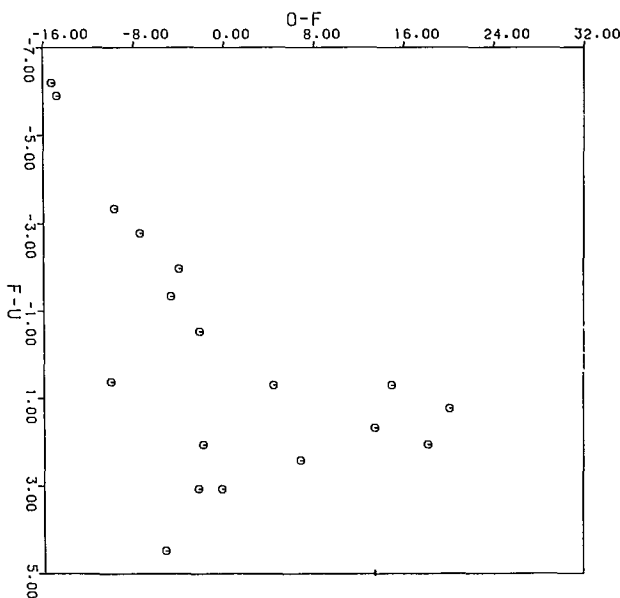


Fig. 6 - Raw data 4" hydrocyclone (non-cumulative)

where

intercept = a

$$a = \frac{\sum X_i \sum Y_i^2 - \sum Y_i \sum X_i Y_i + p(\sum Y_i \sum X_i^2 - \sum X_i \sum X_i Y_i)}{\sum X_i \sum Y_i - n \sum X_i Y_i + p(n \sum X_i^2 - \sum X_i \sum X_i)}$$

split coefficient = p = slope of residuals

p = 0 for regressions of X on Y

p = ∞ for regressions of Y on X

$$|p| = \frac{E_2}{E_1} \text{ for regressions on Y and X}$$

E_i = relative error on variable i

$$p = -\frac{1}{b} \text{ for orthogonal regression}$$

4.1.3 Regression Forced Through the Origin When X, Y ≠ 0,0

$$a = 0 \quad b = \bar{Y}/\bar{X}$$

The results obtained for several types of linear regression applied to non-cumulative and cumulative size distributions of Table 2 are given in Figures 7 and 8. The poor correlation coefficient obtained in particular with non-cumulative data confirms that some points are definite outliers and might as well be remeasured or deleted.

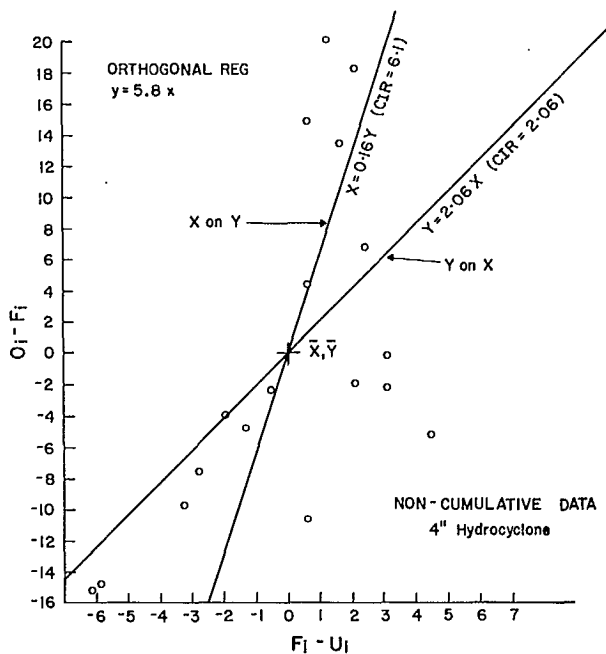


Fig. 7 – Regressions on hydrocyclone non-cumulative data. Correlation coefficient: 0.58

The method can be refined further by weighting each variable in the regression inversely to its assumed variance so that it contributes to the regression curve only in proportion to its precision.

4.2 NODE IMBALANCE MINIMIZATION

The mass conservation equations satisfied by the flow rates and assays in a two-product circuit can be written:

$$W_1 - W_2 - W_3 = I^s$$

$$W_1 X_1 - W_2 X_2 - W_3 X_3 = I^x$$

where I^s and I^x are the node imbalances, or departure of the mass conservation equation from balance, or residuals. Several equations with I^x residuals can be written if several assay types are known.

The node imbalance minimization method consists in saying that rather than choosing the minimum subset of the assays that allows the use of a n-product formula, one uses as best estimates of W_j the values that minimize the sum of the squares of all imbalances:

$$F = \sum (I^s)^2 + \sum (I^x)^2 = \text{minimum}$$

Intuitively one can indeed accept that since there are no W_j's that balance exactly all equations, those which come closest to an overall balance qualify as best estimates. The calculation of these W_j's requires the derivatives of F with respect to all unknown W_j's. The solution values are such that all derivatives are zero at the same time.

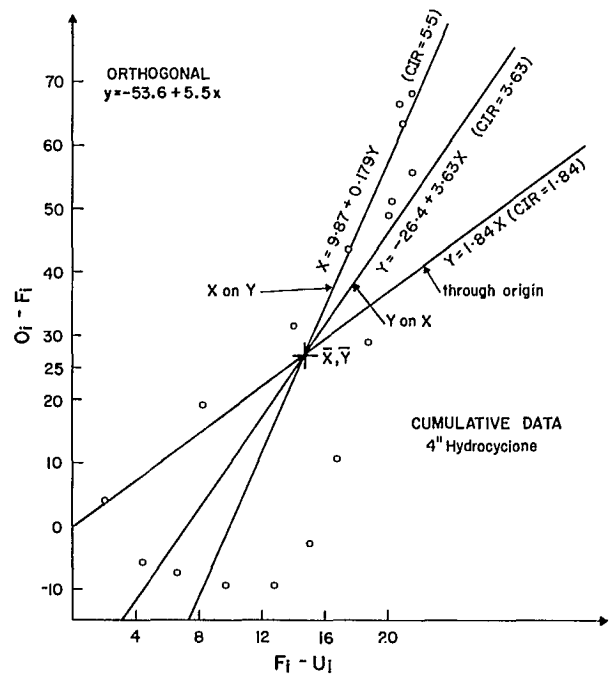


Fig. 8 – Regressions on hydrocyclone cumulative data. Correlation coefficient: 0.81

Therefore:

$$\frac{\delta[\sum(I^s)^2 + \sum(I^x)^2]}{\delta W^2} = 0$$

$$\frac{\delta[\sum(I^s)^2 + \sum(I^x)^2]}{\delta W_3} = 0$$

$$F = \sum(I^s)^2 + \sum(I^x)^2 = -W_1^2 + W_2^2 + W_3^2 - 2W_1W_2 - 2W_1W_3 + 2W_2W_3 + W_1^2 \sum X_1^2 + W_2^2 \sum X_2^2 + W_3^2 \sum X_3^2 - 2W_1W_2 \sum X_1X_2 - 2W_1W_3 \sum X_1X_3 + 2W_2W_3 \sum X_2X_3$$

$$\frac{\delta F}{\delta W_2} = 2(W_2 - W_1 + W_3 + W_2 \sum X_2^2 - W_1 \sum X_1X_2 + W_3 \sum X_3X_2) = 0$$

By symmetry:

$$\frac{\delta F}{\delta W_3} = 2(W_3 - W_1 + W_2 + W_3 \sum X_3^2 - W_1 \sum X_1X_3 + W_2 \sum X_3X_2) = 0$$

By standard elimination technique:

$$\frac{W_3}{W_1} = \frac{(1 + \sum X_1X_3)(1 + \sum X_2^2) - (1 + \sum X_1X_2)(1 + \sum X_2X_3)}{(1 + \sum X_3^2)(1 + \sum X_2^2) - (1 + \sum X_2X_3)^2}$$

and $\frac{W_2}{W_1}$ is obtained by symmetry.

If one has only one assay type, the \sum sign can be removed and the equation simplifies to:

$$\frac{W_3}{W_1} = \frac{X_1 - X_2}{X_3 - X_2}$$

which is the two-product formula corresponding to the special case $(I^s)^2 + (I^x)^2 = 0$: the equations have an exact solution.

4.3 GENERALIZED LEAST-SQUARES METHOD

All methods of material balance computation reviewed above assume that the experimental values of the assays are their best estimate. In the following, a method is discussed in which the experimental values are also adjusted by an amount which is such that the sum of squares of all adjustments is minimum, whence the name "least squares" method.

If best estimates \hat{X} and \hat{W} are associated to each assay X and flow rate W , the maximum-likelihood estimators for a normal population of X and W are such that:

$$\sum(X - \hat{X})^2 + \sum(W - \hat{W})^2 \text{ minimum}$$

This estimator is also called an *estimator of minimum variance*.

In a process where X and W have been measured, one may want to calculate the best estimates \hat{X} and \hat{W} . These can be obtained by solving all valid mass balance equations in the flowsheet.

These equations are of the type:

$$\hat{W}_1 - \hat{W}_2 - \hat{W}_3 = 0$$

$$\hat{W}_1\hat{X}_1 - \hat{W}_2\hat{X}_2 - \hat{W}_3\hat{X}_3 = 0$$

The second type of equation can be written:

$$(W_1 - R_1^s)(X_1 - R_1^x) - (W_2 - R_2^s)(X_2 - R_2^x) - (W_3 - R_3^s)(X_3 - R_3^x) = 0$$

using the relation

$$X = X^* + E^x$$

i.e., experimental value = true value + experimental error which is approximated by $X = \hat{X} + R^x$. (Experimental value = estimate + residual.)

The number of unknown R^s and R^x in a general mass balance problem is usually much larger than the number of mass conservation equations. This implies that an infinite number of solutions exist that satisfy the mass balance equations. The objective of a mass balance method is, therefore, to produce the maximum-likelihood solution.

As stated above, if the assay and flow rate populations can be considered as normally distributed as well as randomly sampled (i.e., unbiased and uncorrelated), the maximum-likelihood estimates are such that:

- the estimates variance is minimum;
- the mass balance equations are satisfied.

The reader of Chapters 3.1 and 3.2 (2,3) will notice that the notion of data redundancy is sometimes very subtle and hard to grasp at first. In particular the BILMAT presentation is based on the concept that the only true variables to be estimated are the flow rates and that the number of equations is therefore usually much larger than the number of unknowns. The solution to this problem also happens to produce better estimates of the assays. The MATBAL presentation, from the onset, classifies assays and flowrates as problem variables for which better estimates are sought. In that case the number of equations is smaller than the number of variables. Nevertheless, both programs solve essentially the same problem. (See also Section 7.2.5.)

4.3.1 Lagrange Multipliers

The problem configuration described above is called a *constrained minimization problem* which has the general form:

$$\begin{array}{ll} \text{Min } \{F(x)\} & \text{minimum criterion} \\ G_i(x) = 0 & \text{constraints} \end{array}$$

It can be shown that the values of x that are solutions to this problem are also solutions to:

$$\text{Stat } \{L(x_i, \lambda_i)\} \quad \text{Stat} = \text{Stationary point}$$

where

$$L(x_i, \lambda_i) = F(x) + \sum_i \lambda_i G_i(x)$$

λ_i is called a Lagrange coefficient or multiplier;
 L is called a Lagrangian.

The material balance problem can therefore be solved by calculating the stationary point of the corresponding Lagrangian.

$$\Sigma(X - \hat{X})^2 + \Sigma(W - \hat{W})^2 + \sum_i \lambda_i G_i(\hat{X}, \hat{W})$$

where $G_i(\hat{X}, \hat{W})$ are the mass conservation constraints and λ_i the Lagrange unknown coefficients or multipliers.

As discussed later on, various methods can be used to compute this stationary point.

4.3.2 Weighting Factors

One further refinement can be introduced in the method by weighting each variable adjustment in proportion to its precision.

The analogy with a weighted mean for several scores in school can illustrate this point. For scores of 60, 60, and 90 in three subjects equally weighted, the mean score is 70. If the scores are weighted 4, 2, and 1 respectively, the weighted mean is:

$$\frac{60 \times 4 + 60 \times 2 + 90 \times 1}{(4 + 2 + 1)} = 64.3$$

More weight has been given to a low score; therefore, the mean has been lowered.

In the minimization of the Lagrange function, it is appropriate to weight each adjustment of a variable by the inverse of its variance. The larger the variance (i.e., the less precise the assay), the lower the weighting factor $1/\sigma_x^2$.

The stationary-point coordinates will not be as affected by an imprecise assay as by a precise one. This adds to the efficiency of the estimator.

The most efficient Lagrangian is, therefore:

$$\sum \frac{(X - \hat{X})^2}{\sigma_x^2} + \sum \frac{(W - \hat{W})^2}{\sigma_w^2} + \sum_i \lambda_i G_i(\hat{X}, \hat{W})$$

The necessity for the user to provide a realistic estimate of the variances of all measurements is an intrinsic part of the mass balance problem. This is referred to as providing an *error model for the experimental values*. This error model is characterized by **assumptions**: normal distribution; unbiased, uncorrelated variables; and by **data**: variable variances; or standard deviations.

5. ERROR STRUCTURE IN THE EXPERIMENTAL DATA

The problem of error structure in the experimental data is the subject of a separate chapter in the "SPOC Manual", Chapter 2 (4). The main facts about error models for material balance computation are summarized in this section.

5.1 COMPONENTS OF THE TOTAL ERROR FOR AN ASSAY VALUE*

The **total error** is a combination of:

1. the sampling fundamental error;
2. the errors due to time and space variations in the lot;
3. the errors due to sampler design and operation;
4. the errors due to sample preparation and analysis.

Factors 1 and 4 cannot be eliminated; 2 and 3 can be minimized.

5.1.1 Fundamental Error (FE)

P.Gy (5) shows the variance of FE to be:

$$\sigma_{FE}^2 = C \ell f g d^3 \left[\frac{1}{M_S} - \frac{1}{M_L} \right]$$

where

- M_S = sample mass (g)
- M_L = lot mass (g)
- C = composition factor (g/cm³)
- ℓ = liberation factor (0 or 1)
- f = particle shape factor (0.5)
- g = size distribution factor
- d = maximum particle size (cm).

C , the most variable coefficient is given by:

$$C = \frac{1 - X_L}{X_L} [(1 - X_L) \rho_c + X_L \rho_g]$$

where

- X_L = critical concentration
- ρ_c = specific gravity of critical component
- ρ_g = specific gravity of non-critical component (g/cm³).

One easily sees that for a trace element, $X_L \ll 1$.

Therefore, C is large and, consequently, σ_{FE}^2 is large unless M_S itself is very large (large sample).

Inversely, for a concentrate, $X_L \approx 1$, and σ_{FE} can remain small when smaller samples are collected:

- ℓ – for slurry sampling should be given a value of 1;
- f – for most concentrated solids should be given a value of 0.5;
- g – for material containing several sizes of particles, should be 0.25 for single size material, $g = 1$.

5.1.2 Errors Due to Time and Space Variations in the Lot

These errors can follow various patterns, such as long-range and short-range variations. The periodicity of these variations, however, can introduce another source of error.

The only way to reduce these errors is to collect composite samples. Systematic sampling (at regular intervals) is preferable to random sampling unless a cyclic variation exists in the lot.

5.1.3 Errors Due to Sampler Design and Operation

These errors occur when all particles do not have the same probability to enter the sampler.

The basic rules to minimize them are:

- The cutter edges must be parallel.
- The cutter must move perpendicular to the axis of the stream, at a constant speed (≤ 0.6 m/s).
- The cutter width must be at least 10 mm and greater than three-times the largest particle.

5.1.4 Errors Due to Sample Preparation and Analysis

The sources of such errors are numerous; e.g., contamination, loss, alteration, sub-sampling, analysis method. They normally represent a minor fraction of the total error.

In the solution of material balance problems, the standard deviation of the experimental data is an essential part of the input data. It is used to distribute the data adjustment in proportion to the data accuracy and also to calculate the resulting error on the variable estimates. In a first approximation, all errors are assumed to be normal, unbiased, and uncorrelated.

6. SENSITIVITY ANALYSIS

6.1 INTRODUCTION

No material balance problem is complete without a value being calculated for the precision of the flow rate estimates and, ideally, for the adjusted assays. In other words, the confidence interval of the material balance results must be estimated. The benefits of a sensitivity analysis are, therefore:

- assessment of the error on the estimates;
- reflexion on the experimental results and the error model used to perform the computation.

A few basic principles of statistics must be reviewed before the sensitivity analysis can be understood:

- For a normally distributed variable, the mean value and the standard deviation of a series of measurements (sample) are the maximum-likelihood estimates of the distribution parameters. They are estimates of minimum variance.
- In a material balance problem, the experimental variables, the estimates, the residuals, and the errors are assumed to be normally distributed. The estimates being of minimum variance, the variance of the calculated variables is smaller than, or equal to, that of the experimental variables. Similarly, the variance of the residuals is smaller than that of the errors.

The sensitivity analysis consists in studying the relationship between the error model of the experimental data and that of the calculated estimates. It can be done by two different approaches; the analytical method, and the Monte Carlo method illustrated in Figure 9.

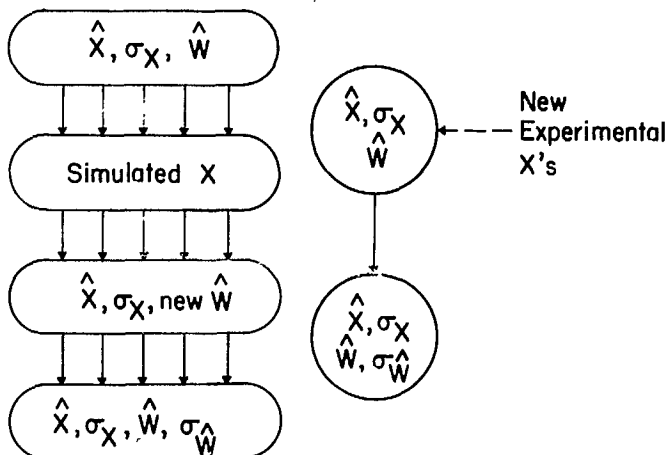


Fig. 9 - Principle of the two-error estimators

6.2 THE ANALYTICAL METHOD OF SENSITIVITY ANALYSIS

6.2.1 Principle

The principle of the method is illustrated by the calculation of the variance of a linear combination of two random variables, X_1 and X_2 .

$$\text{If } Y = aX_1 + bX_2, \text{ then } \sigma_Y^2 = a^2\sigma_{X_1}^2 + b^2\sigma_{X_2}^2 - 2ab\sigma_{X_1X_2}.$$

If X_1 and X_2 are independent, the covariance term, $\sigma_{X_1X_2}$, is zero.

The variance of the dependent variable, Y , is therefore a function of the functions of the independent variables.

In a material balance problem, the mass conservation equations are non-linear and the variance relationships are more complex. They must be linearized around a consistent set of values of the variables in order to derive the suitable relationships.

Ideally, the linearization should be done around the true values of the variables. In practice, it is done around a set of estimates generated by the material balance program.

If \hat{W} and \hat{X} represent these estimates, one can define the following variations:

$$\delta W = W - \hat{W}$$

$$\delta X = X - \hat{X}$$

$$\delta \hat{W} = \hat{W} - \hat{W}$$

$$\delta \hat{X} = \hat{X} - \hat{X}$$

When \bar{W} and \bar{X} are considered as true values, the first two variations approximate the errors on flow rates and class fractions. Therefore, the variances of δW and δX are the variances of the measured variables W and X .

Similarly, the variances of $\delta \hat{W}$ and $\delta \hat{X}$ are the variances of the variable estimates.

The BILMAT User's Guide shows how the values of σ_W^2 and σ_X^2 can be calculated by variance algebra as a function of $\sigma_{\hat{W}}^2$ and $\sigma_{\hat{X}}^2$. The resulting variances are conservative; i.e., larger than their true values, but very often accurate.

6.2.2 Factors Influencing the Variance Estimates

The following factors contribute to the validity of the variance estimates obtained by the analytical method:

- The measurement errors follow a normal distribution.
- They are uncorrelated.

- The assumed error variances are reasonable.
- The estimate used as a true value is accurate.

The following cause-to-effect relationships must be underlined:

- The better the data, the smaller the estimate variances.
- The more redundant the data, the smaller the estimate variances.
- The more efficient the process with respect to the problem variables (e.g., good concentration ratio), the smaller the estimate variances.
- The fewer recycle streams that exist, the smaller the estimate variances are.

6.2.3 Practical Use of the Estimate Variances

6.2.3.1 Confidence interval

If $\sigma_{\hat{X}}^2 = 10^{-4}$ and $\hat{X} = 5\% = 0.05$,
then $\sigma_{\hat{X}} = 10^{-2} = 0.01$.

This means that there is a 68% chance that X^* is between $\hat{X} - \sigma_{\hat{X}}$ and $\hat{X} + \sigma_{\hat{X}}$: i.e., between 0.04 and 0.06.

The chances of X^* being within $\hat{X} \pm 2\sigma_{\hat{X}}$ (i.e., 0.03 and 0.07) are 95%.

6.2.3.2 Residuals analysis

The residuals (e.g., $\hat{W} - W$) are random variables with their own distribution. Their variances are given by:

$$\sigma_W^2 - \sigma_{\hat{W}}^2 \text{ and } \sigma_X^2 - \sigma_{\hat{X}}^2$$

Using the example above where $\sigma_{\hat{X}} = 0.01$ and $\sigma_X = 0.02$, the 95% confidence interval of the residual $\hat{X} - X$ is:

$$\begin{aligned} \pm 2 \sqrt{\sigma_X^2 - \sigma_{\hat{X}}^2} &= \pm 2 \sqrt{4 \cdot 10^{-4} - 10^{-4}} \\ &= \pm 3.56 \cdot 10^{-2} \end{aligned}$$

If the calculated residual $\hat{X} - X$ is outside this interval ($-3.56 \cdot 10^{-2} + 3.56 \cdot 10^{-2}$), the possibilities are:

1. The experimental data X have been accidentally altered.
2. The sampling procedure has introduced a significant bias, or the sampling variance is underestimated.
3. The assay procedure (sample preparation and analysis) has introduced a significant bias.

Case 1 is more likely if only one X_j is out of range.

Case 2 is more likely if several X_j 's are out of range in the same stream j for different species i .

Case 3 is more likely if several X_j 's are out of the range for the same species i in several streams j .

Repeated positive (or negative) values in the residuals obtained for several material balances of the same circuit can also help to detect bias in the experimental data. Usually, residuals with values outside a $\pm 3 \sigma_X$ range are considered as outliers and should lead to either data rejection or error model modification. Repeated abnormally-small residuals should also suggest a reassessment of the assumed error variance. The overall contribution of some data to the material balance solution can also be estimated and suggest new sampling strategies.

6.3 THE MONTE-CARLO ANALYSIS

A digital computer can generate random numbers belonging to a given probability distribution. For instance, given a mean and a standard deviation, a program can generate as many samples of a normal population as required.

In a material balance problem, each measurement - X and W - is characterized by:

- its standard deviation (from the error model);
- its best estimate \hat{X} and \hat{W} (from the result).

It is, therefore, possible to generate pseudo-experimental values belonging to the same population. In other words, we can simulate assay values that are equivalent to those that could be obtained in repeated sampling. This can be done only if the assumed error model is valid.

After one mass balance calculation has been completed, all the experimental values can be renewed by simulation and solved for a new series of calculated variables. The accumulation of several such series allows the computation of their means and standard deviations. These quantify the sensitivity of the calculated flow rates to the natural variations in the observed variables.

The **larger** the number of *repeats*, the **more reliable** the *errors estimate*.

The potential of this method to check the sensitivity of the results to the error model must be emphasized. If the error model used for the Monte-Carlo simulation is different from that used in the mass balance solution, the magnitude of the variations in the estimates and their standard deviation are an indicator of the importance of using the proper error model in a particular problem.

7. MATERIAL BALANCE PROGRAMS

An exhaustive survey of all published material balance programs is beyond the scope of this text and the reader is referred to such a survey by Reid, Smith, Voller and Cross (6).

Material balance programs can be classified into four broad families depending on the method of solution of the material balance equations:

1. programs using an iterative Lagrange solution, e.g., MATBAL;
2. programs using a hierarchical solution, e.g., BILMAT;
3. programs using a two-step node imbalance + GLS solution;
4. miscellaneous.

The following discussion covers types 1 and 2 more extensively.

7.1 MATBAL: ITERATIVE LAGRANGE SOLUTION TO THE GLS PROBLEM

In the MATBAL approach, the stationary point of the Lagrangian is obtained by a classical calculus method: the cancellation of all derivatives with respect to the variables.

$$\frac{\delta L}{\delta x} = 0$$

$$\frac{\delta L}{\delta w} = 0$$

$$\frac{\delta L}{\delta \lambda_i} = 0$$

The above system of equations has necessarily as many unknowns as equations.

All equations can be derived by hand in order to bring the system to the form:

$$M X = C$$

where X is a vector of variables \hat{X} , \hat{W} , λ_i , and M and C are matrices of coefficients.

These equations have been coded in FORTRAN and can be solved automatically by the program. The user has to input a flowsheet description that is suitable, using MATBAL nomenclature.

As one can imagine, the systems to be solved are large and the applicability is strictly restricted to the type of mass constraints that have been considered in the derivatives calculation. In this family, several other programs have been published especially by Cutting (7).

For full narrative, mathematical, and programming details on the MATBAL program, the reader is referred to Chapter 3.2 of the "SPOC Manual" (3) which is a user's guide.

7.2 BILMAT: HIERARCHICAL SOLUTION TO THE GLS PROBLEM

7.2.1 Flowsheet Variables

BILMAT has been designed to simultaneously balance data describing various subdivisions of a pulp stream. The first level of division consists of two phases; usually one solid and one liquid. Each phase can itself be described by classes (e.g., particles in a size interval). Each class can be divided into sub-classes (e.g., particles in a specific gravity range within a size interval). Each class or subclass can be described by characteristics (e.g., chemical assay, mineralogical assay, etc.).

This is summarized in Figure 10. The upper two levels of the stream description (e.g., pulp, solid and/or liquid) are called *macroscopic*. The lower two levels are called *microscopic*. Specific variables can describe each level and they are referred to as macroscopic and microscopic variables.

Level one is described by flow rate measurements. Level two is described by flow rate and phase concentration in the pulp (e.g., liquid fraction in the pulp, or solid fraction in the pulp). Level three is described by mass fractions of each class in one of the level-two phases (e.g., mass fractions of a particle size distribution). Level four can either be described by mass fractions of sub-classes within a level-three class, or assays of level-three classes.

The scale of four levels can be translated by considering one phase as level one, classes of that phase as level two, sub-classes as level three, and characteristics as level four. In comparison, MATBAL handles only data

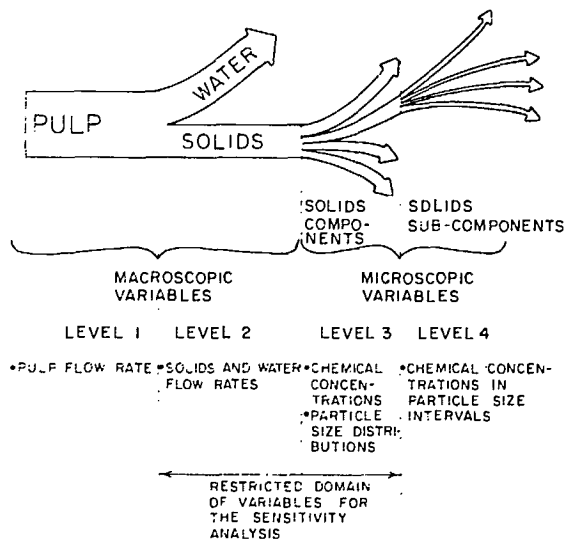


Fig. 10 - Various levels of the mass balance variables

relative to two adjacent levels; e.g., (pulp + solid and water), or (solid + assay), or (size interval + assay) etc. The BILMAT User's Guide gives examples of flowsheet variables.

7.2.2 Flowsheet Analysis

Flowsheet analysis in terms of networks is a prerequisite to using the BILMAT program. A **network** is a graphical representation of the mass conservation across process units.

Each level of subdivision of the process streams must be considered for network definition. A total of eight different networks can be described: one at level one, one at level two, four at level three, and two at level four.

Details on network definition are given in the BILMAT User's Guide. For computation purposes, networks are expressed as matrices according to the definitions of the User's Guide.

7.2.3 Mass Conservation Equations

The mass conservation equations corresponding to the existing networks are described in Sections 2.4.2 to 2.4.5 of the BILMAT User's Guide and in Chapter 3.1 of the "SPOC Manual" (2).

7.2.4 Independent Variables

Independent flowsheet variables are those that allow the others to be calculated by the material balance equations. For instance, in a solid network consisting of q nodes and p branches, there are q equations of conservation, p flow rates and, consequently, $p - q$ independent flow rates. There may be several sets of independent flow rates, but no set of $p - q$ values is a set of independent flow rates.

This concept of independence can be applied to variables of several levels in the global material balance context.

For instance, if we consider solid flow rates W_j^s ($j = 1, \dots, p$) and mass fractions X_i^j in classes $i = 1, \dots, n$, the valid material balance equations are:

q – solid conservation equations

nq – class conservation equations

The number of independent variables is:

$$(p + np) - (q + nq) - p = n(p - q) - q$$

If the set of classes is complete (i.e., the total mass fraction is one), the number of independent class mass fractions must also be reduced by one per stream. Therefore, the actual number of independent variables in the above problem is:

$$(p + np - 1) - (q + nq) = (n + 1)(p - q) - 1$$

7.2.5 Data Redundancy

This concept is a consequence of the variable independence described above. If a set of independent variables has been measured, all variables can be calculated; the problem is said to be exactly determined, or to correspond to a minimal experimental design. If fewer variables have been measured, no complete solution can be calculated and the problem is called *underdetermined*. If extra variables have been measured, they constitute *redundant* data.

A problem can be redundant with respect to some variables, and underdefined – or exactly defined – with respect to others. The checking of redundancy is an important and sometimes delicate prerequisite to using BILMAT. This is illustrated by the following example.

Consider a two-stage flotation circuit (as illustrated in Figure 11). The following four data sets can be analyzed for redundancy:

- A: $W_1^s, W_2^s, X_2^1, X_3^1, X_4^1, X_1^2, X_3^2$
- B: $W_1^s, W_2^s, X_2^1, X_3^1, X_4^1, X_5^1, X_1^2, X_3^2, X_4^2$
- C: $W_1^s, W_2^s, X_2^1, X_3^1, X_4^1, X_5^1, X_1^2, X_2^2, X_3^2$
- D: $W_1^s, W_2^s, W_6^s, X_2^1, X_3^1, X_4^1, X_5^1, X_6^1, X_1^2, X_2^2, X_3^2, X_4^2, X_5^2$

The data describe one phase (solid), and two classes X^1 and X^2 . The number of independent variables is: $(6 + 12) - (3 + 6) = 9$ (assuming that $X_1^1 + X_2^1 \neq 1$).

Case A is underdefined.

Case B is exactly defined.

Case C is overdefined for all variables except X_j^2 ($j = 4, 5, 6$) which are not calculable.

Case D is overdefined.

The program can produce meaningless results when underdefined data are used. If only one flow rate is measured (or fixed), no adjustment of its value can be done, the measured value is the best estimate and the number of independent variables should be reduced by one ($p - q - 1$) or $n(p - q) - q - 1$.

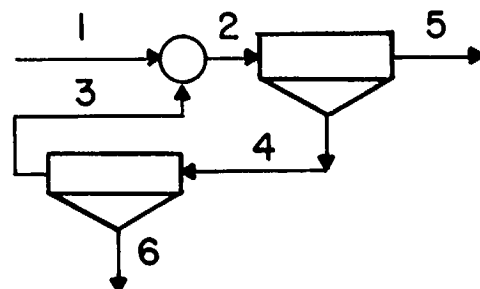


Fig. 11 – Sample flowsheet for data redundancy test

7.2.6 BILMAT Algorithm

In conformity with the GLS method, the following criterion is to be minimized:

$$\begin{aligned}
 J &= \sum_j (W_j^p - \hat{W}_j^p)^2 / (\sigma_j^p)^2 & \text{a) pulp flow rates} \\
 &+ \sum_j (W_j^w - \hat{W}_j^w)^2 / (\sigma_j^w)^2 & \text{b) water flow rates} \\
 &+ \sum_j (W_j^s - \hat{W}_j^s)^2 / (\sigma_j^s)^2 & \text{c) solid flow rates} \\
 &+ \sum_j (S_j - \hat{S}_j)^2 / (\sigma_j^{SL})^2 & \text{d) solid/liquid ratio} \\
 &+ \sum_j \sum_i (X_j^i - \hat{X}_j^i)^2 / (\sigma_j^i)^2 & \text{e) class fractions} \\
 &+ \sum_j \sum_i \sum_k (X_j^{ik} - \hat{X}_j^{ik})^2 / (\sigma_j^{ik})^2 & \text{f) characteristic} \\
 & & \text{fractions.}
 \end{aligned}$$

This weighted sum of squares is minimized (subject to the valid mass conservation equations), by a hierarchical method fully detailed in Section 4 of the BILMAT Manual. In a first step, an adjustment is made for the relative solid flow rates (W_j/W_1); then, the class mass fractions are adjusted; finally, the remaining variables are adjusted (a,b,d). These steps are repeated until all variables converge to steady-state values.

7.3 NODE IMBALANCE + GLS COMBINATION METHODS

When experimental data have been collected around several units in a complex flowsheet, the node imbalance minimization method described in Section 4.2 can be generalized.

The valid mass balance equations are of two types:

- solid mass balance, $AW = I^s$
- characteristic mass balance, $AWX^i = I^{xi}$;

where

- A is the incidence matrix describing the flowsheet
- W is the solid flow-rate column vector
- X^i is the diagonal matrix of assay X^i .

It can be shown that the estimate of W (that minimizes the sum of squares of the node-imbalances) is given by:

$$W = M^+ I$$

where

- M is the stack of matrices A and AX^i
- M^+ is $(M^T M)^{-1} M^T$ (also called the pseudo-inverse of M)
- I is the stack of right hand side vectors I^s and I^{xi}
- X^i is used as the best estimate of X^i .

SAMBA is a conversational program to calculate such estimates and has been developed by Smith, Burroughs, and Laguiton. Its documentation constitutes Chapter 2.1 of the "SPOC Manual" (8).

If a refined solution is needed, a second stage of calculation consists in using the estimates of the mass flow rates obtained above as constants. The experimental assays can then be adjusted according to a least-squares method using the mass conservation equations as constraints.

A variation of the above method consists in using the sum of squares of the node imbalances as criteria to be minimized and the mass conservation equations as constraints in a Lagrange-multipliers method. The computation of the inverse of the derivative coefficients matrix requires that all constraints be linearly independent.

This is used in the COEF subroutine of the MATBAL program. This requirement for independent equations does not apply to the pseudo-inverse algorithm described above. Both methods provide useful first approximations for the iterative general least-squares methods.

7.4 MISCELLANEOUS

The White technique (9) does not require the calculation of a Lagrangian. First, a minimum set of independent variables is selected from which all other variables can be calculated. This set is optimized, variable by variable, in a direct, unidimensional search program so that the minimization criterion, $\sum (X - \hat{X})^2 / \sigma_{\hat{X}}^2 + \sum (W - \hat{W})^2 / \sigma_{\hat{W}}^2$, can be computed at each step of the search.

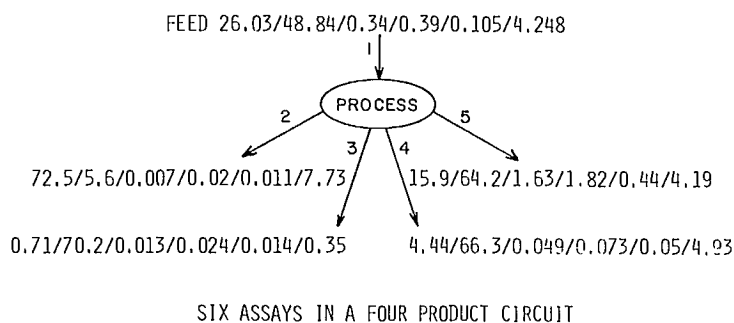
The variables entering this criterion are obtained by means of the valid mass conservation relations that exist between them and the search variables.

A review of data-adjustment procedures was published by A. Mular in *Computer Methods for the 80's in the Mineral Industry* (10).

8. EXERCISES ON MATERIAL BALANCE COMPUTATION

8.1 EXERCISE 1: n-PRODUCT FORMULA

1. Derive the two-product formula.
2. Consider the four-product circuit illustrated hereafter, in which each stream has been assayed for six species.
 - a) How many solutions can be obtained by the n-product formula?
 - b) Calculate several of them using the NPRD* program.
 - c) If you were responsible for calculating inventories on this circuit, what would you do?



*NPRD is one of the miscellaneous utility programs documented in Chapter 8 of the SPOC Manual (11).

8.2 EXERCISE 2: REGRESSION

Consider the attached data collected around a hydrocyclone. F is the feed, U the underflow; O the overflow. These data are the size distribution of the solids.

1. Using the two-product formula, calculate the ratio U/O for several size intervals. What conclusions do you draw?
2. The circulating load is given by:

$$\frac{X_O - X_F}{X_F - X_U} = CIR$$

Calculate the mean circulating load.

Plot $X_O - X_F$ vs $X_F - X_U$. What is the best value of CIR that you can get from your plot?

3. Use the STAMP* program to determine CIR by linear regression.

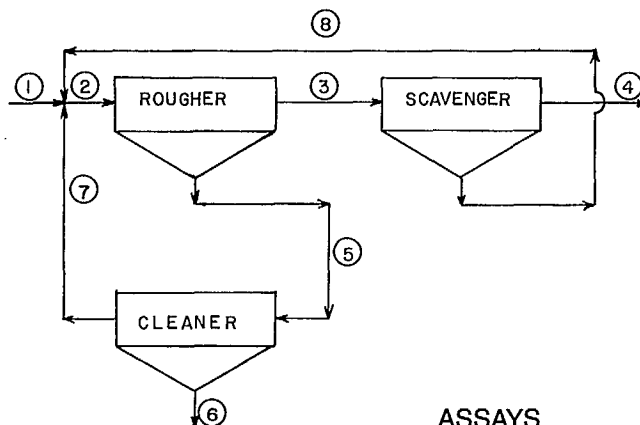
X_F	X_U	X_O	$X_F - X_U$	$X_O - X_F$
3.97	5.95	0.00	-1.98	-3.97
15.25	21.45	0.00	-6.20	-15.25
14.80	20.70	0.00	-5.90	-14.80
9.71	13.05	0.00	-3.34	-9.71
8.27	11.05	0.80	-2.78	-7.47
4.70	6.03	0.00	-1.33	-4.70
10.06	9.44	0.00	0.62	-10.06
4.01	4.53	1.82	-0.52	-2.19
2.25	1.56	6.61	0.69	4.36
1.92	1.23	16.83	0.69	14.91
2.26	1.04	22.32	1.22	20.06
2.56	0.50	20.70	2.06	18.14
2.67	0.99	16.04	1.68	13.37
3.07	0.65	9.85	2.42	6.78
3.38	0.31	3.21	3.07	-0.17
3.63	0.56	1.39	3.07	-2.24
2.29	0.23	0.42	2.06	-1.87
5.20	0.73	0.01	4.47	-5.19
100.00	100.00	100.00	0.00	0.00

*See SPOC Manual, Chapter 8 (11).

8.3 EXERCISE 3

Consider the attached flowsheet. All eight streams have been assayed for Cu, Zn, Fe with results as given on the flowsheet.

1. For a feed of 100 t/h of solids, calculate flow rates W_4 and W_6 by the two-product formula.
2. Calculate the precision, $\frac{\Delta W_6}{W_6}$, when all assays are known with a 6% relative precision and $\frac{\Delta W_1}{W_1} = 0.1$
3. Calculate W_4 and W_6 , using the node imbalance formula.
4. Use the SAMBA program to calculate the general node imbalance minimization.*



	ASSAYS		
	1	2	3
1 STREAM 1	0.163	3.93	11.57
2 STREAM 2	0.447	11.63	12.79
3 STREAM 3	0.466	8.11	14.35
4 STREAM 4	0.140	0.49	13.09
5 STREAM 5	0.960	34.64	14.37
6 STREAM 6	0.657	52.07	14.67
7 STREAM 7	1.020	42.73	14.66
8 STREAM 8	0.440	11.86	13.75

8.4 EXERCISE 4

Consider the attached flowsheet. Ten numbered sampling points have been assayed for *four* species, except 2, 3, 7 and 8 which have been assayed for *three* species.

Assays are as follows:

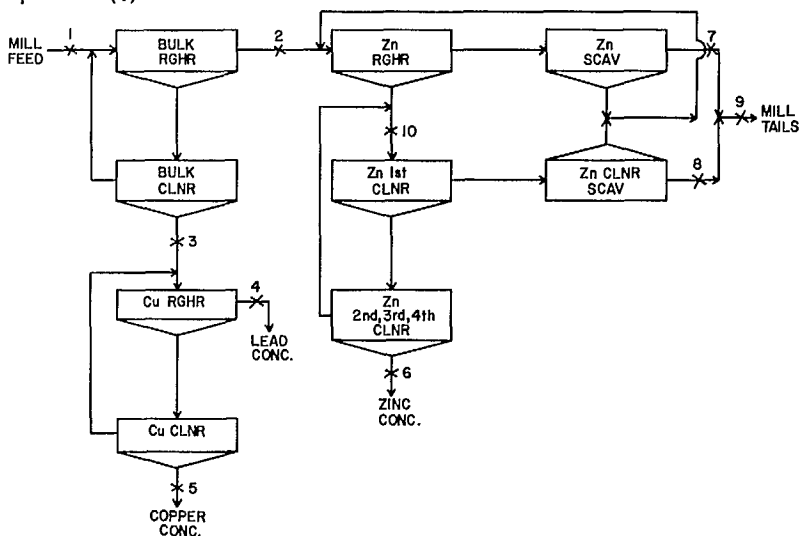
1. Mill Heads	.87	1.27	3.69	1.61
2. Blk Scav Tls	.27	.52	3.54	
3. Cu-Pb Conc	10.45	13.06	4.05	
4. Pb Conc	1.50	16.70	3.62	11.22
5. Cu Conc	25.29	4.22	4.63	11.51
6. Zn Conc	.87	1.37	48.4	2.39
7. Zn Scav Tls	.22	.49	.80	
8. Zn Cl Scav Tls	.37	.99	1.63	
9. Final Tails	.21	.51	0.89	0.67
10. Str. Cntrl	.90	.95	30.0	

1. For a feed of 100 t/h, calculate the three concentrates and the final tailings flow rates.
2. Prepare the MATBAL input file for this problem. The Table of assay precisions is given below.*
3. What can you say about sampling point No. 10?

Table of assay precisions

VG	G	VG	F
F	F	G	F
F	F	F	F
VG	F	F	F
F	F	VG	F
F	F	G	F
F	F	F	F
G	G	G	F
F	F	F	F

*See "SPOC Manual", Chapter 2.1 (1).



8.5 EXERCISE 5

1. Solve Sample Runs 1 and 2 provided in the MATBAL User's Guide "SPOC Manual", Chapter 3.2 (3).

8.6 EXERCISE 6

1. Solve Sample Runs 1 and 2 provided in the BILMAT User's Guide ("SPOC Manual", Chapter 3.1).

8.7 EXERCISE 7: SENSITIVITY ANALYSIS BY THE MONTE-CARLO METHOD

1. Rerun the NPRD program with data from Exercise 3 and activate the Monte-Carlo simulation option.
 - a) Impose a 5% relative standard deviation on all assays.
 - b) Impose a 10% relative standard deviation.
 - c) Impose a 2% relative standard deviation.

Conclusions?

2. Solve Exercise 3 with MATBAL, Monte-Carlo option "on" (ICON = 1, NMC = 10) and successively mask the various assays (IO = 3, 1, 2, 3; IO = 2, 1, 2; IO = 2, 1, 3; etc.).

Conclusions?

8.8 EXERCISE 8: SENSITIVITY ANALYSIS BY THE ANALYTICAL METHOD

Execute the BILMAT program for sample Run 1 (Chapter 3.1).

1. Using the results of the sensitivity analysis, explain the variation of the precision of flow rates from .06% to 19%.
2. Calculate the 95% confidence interval on the concentrate flow rate and its Nb assay. Compare to the measured values and comment.
3. Can the flow rates be determined with an acceptable precision without using the Nb assays?
4. Determine the accuracy of the estimated values of the cleaner-4 concentrate assays, if experimental values were poorly known for that stream.
5. If you had to reduce the cost of a sampling campaign, which of Mg or P assays would you delete?
6. Suppose a value of 3.09 is entered instead of 3.69 for the Fe assay in the rougher tailing. How does this error affect all the results? Can it be detected with the sensitivity analysis?

9. ANSWERS TO EXERCISES

9.1 EXERCISE 1

1. See Section 3.1.
- 2a. 20 solutions (there are 20 combinations of six assays, 3 by 3).
- 2b. See CIM Bull. Vol. 75, N° = 840, p 166-170.
- 2c. Use MATBAL or BILMAT.

9.2 EXERCISE 2

1. See Table 2A.
2. 3.62 (see Table 2A and Fig. 6).
3. See the STAMP program user's guide, Chapter 7.1, SPOC Manual.

9.3 EXERCISE 3

1. Using assay type 1, $W_4 = 4.5$ $W_6 = 95.5$
 Using assay type 2, $W_4 = 6.7$ $W_6 = 93.3$
 Using assay type 3, $W_4 = -96.2$ $W_6 = 196.2$

2. Using assay type 1, $\frac{\Delta W_6}{W_6} = 0.72$

Using assay type 2, $\frac{\Delta W_6}{W_6} = 0.08$

Using assay type 3, $\frac{\Delta W_6}{W_6} = 10.0$

3. Using the formula given in Section 4.2:

$$W_6 = \frac{(1+374.47)(1+171.6)-(1+153.4)(1+217.6)}{(1+2926.9)(1+171.6)-(1+217.6)^2} \times 100$$

$$= 6.81; W_4 = 93.19$$

4. Solution given by SAMBA:

$$W_1 \text{ to } W_8 = (100.0, 236.6, 207.5, 93.6, 29.0, 6.4, 22.66, 113.9)$$

9.4 EXERCISE 4

1. By the four-product formula (the NPRD program)
 Using assays 1, 2, 3: $W_1 = 100$, $W_4 = 3.88$,
 $W_5 = 2.29$, $W_6 = 5.49$, $W_9 = 88.34$
 Using assays 2, 3, 4: $W_1 = 100$, $W_4 = 3.37$
 $W_5 = 4.55$ $W_6 = 5.34$ $W_9 = 86.74$

2. The MATBAL4 input file for this problem is:

```

DATA FOR EXERCISE 4 CHAPTER 3, SPOC MANUAL
9 4 1 3 0 9 1 1 10
0 0 0 0 1 0 0 6 0 0 0 99 0.1 0.1
  1 MILL HEADS
  2 BLK SCAV TLS
  3 CU-PB CONC
  4 PB CONC
  5 CU CONC
  6 ZN CONC
  7 ZN SCAV TLS
  8 ZN CL-SCAV TLS
  9 FINAL TAILS
  1 .87 1.27 3.69 1.61
  2 .27 .52 3.54 0
  3 10.45 13.06 4.05 0
  4 1.5 16.7 3.62 11.22
  5 25.29 4.22 4.63 11.51
  6 .87 1.37 48.4 2.39
  7 .22 .49 .80 0
  8 .37 .99 1.63 0
  9 .21 .51 .89 .67
  1 ASSAY 1
  2 ASSAY 2
  3 ASSAY 3
  4 ASSAY 4
  VG G VG F
  F F G F
  F F F F
  F F F F
  VG F F F
  F F VG F
  F F G F
  F F F F
  G G G F
  1 5 1 -4 -5 -6 -9
  1 3 1 -2 -3
  2 3 3 -4 -5
  3 3 2 -6 -9
  1 100 .01
  1 3 1 2 3
  2 3 1 2 3
  3 3 1 2 3
  4 1 2 3 4
    
```

3. Sampling point No. 10 is not useful in calculating a material balance. It was probably sampled for control purposes.

9.5 EXERCISE 5

See "SPOC Manual", Chapter 3.2 (3).

9.6 EXERCISE 6

See "SPOC Manual", Chapter 3.1 (2).

9.7 EXERCISE 7

- Using the NPRD program and data for Exercise 3, the following results were obtained for 20 repeats of the Monte-Carlo option, using assay types 1, 2, 3, successively.

Assay type 1	5% pre- cision	10% pre- cision	2% pre- cision
W_4	95.55 ± 1.8	± 4.23	± 0.78
W_6	5.29 ± 1.8	± 4.23	± 0.78
Assay type 2			
W_4	93.33 ± 0.43	± 1.05	± 0.19
W_6	6.67 ± 0.43	± 1.05	± 0.19
Assay type 3			
W_4	196.2 ± 103.4	± 555.8	± 21.5
W_6	-96.2 ± 103.4	± 555.8	± 21.5

The estimate of the precision of the calculated flow rates is more reliable when the experimental data are more reliable.

- By masking successively the various assays, it is found that assay type 2 is the only data that seem acceptable in this data set. The solution obtained when this assay alone is used is less sensitive to experimental errors:

$$\begin{aligned}
 W_1 &= 100.0 \\
 W_2 &= 339.8 \pm 84 \\
 W_3 &= 297.3 \pm 78 \\
 W_4 &= 93.26 \pm 0.7 \\
 W_5 &= 42.5 \pm 15 \\
 W_6 &= 6.74 \pm 0.7 \\
 W_7 &= 35.78 \pm 15.6 \\
 W_8 &= 204.0 \pm 78.
 \end{aligned}$$

See also the treatment of this problem by the SAMBA program in Chapter 2 of the "SPOC Manual", example 5.3 (4).

9.8 EXERCISE 8

- The value of the relative standard deviations of a relative ore flow rate depends mostly on the value of the flow rate. A low flow rate generally has a high relative standard deviation.

Absolute standard deviations are affected by the error model defined by the user and by the general structure of the plant network.

- Cleaner-5 concentrate: 95% confidence interval.

	Measured	Estimated
Relative ore flow rate	—	$.025 \pm .004$
Solid flow rate	$.295 \pm .176$	$.337 \pm .066$
Nb assay	36.27 ± 14.51	33.48 ± 6.17

The Nb assay reliability is improved by a large factor greater than two. Its standard deviation is still around 10% but could be lowered if Nb were assayed with a better accuracy (see Section 5.2.5 of Chapter 3.1 [2]).

- Using the results of the mass balance calculation, redo the sensitivity analysis without using the Nb assay. Results and discussion are in Section 5.2.5, Chapter 3.1.
- Use the sensitivity analysis editor to change the error model of the cleaner-4 concentrate assay and to mask the solid flow rate on that stream. Then run the sensitivity analysis program.

The results are:

Relative standard deviation (%)	
Nb	35.86
Mg	72.11
Si	41.33
P	21.25
Ca	13.81
Fe	14.49
S	8.54

An error model of 100% was used on that stream.

After computation, the same values have an acceptable accuracy. But this has destroyed the reliability of the assay of the cleaner-5 output streams. Therefore, cleaner-4 concentrate should be assayed with accuracy. By performing the same experiment with cleaner-1 concentrate, one would see that this stream is well known after computation and that it has no significant effect on other streams.

- Use the sensitivity analysis editor to mask Mg assay and then P assay. Observation of the relative ore flow rate accuracies leads to the conclusion that deleting P assays does not significantly affect the reliability of the results (see Section 5.2.5 of Chapter 3.1 [2]). Therefore, P could not be assayed in a future sampling campaign.
- Use the mass balance editor to modify the Fe assay in the rougher tailing. Run BILMAT and then the sensitivity analysis program. The mass balance results are affected by that modification and, at first glance, nothing wrong can be detected. However, the sensitivity analysis results show that the outlier flag is equal to 3 for that assay in that stream.

10. REFERENCES

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