

NBS TECHNICAL NOTE 796

A UNITED STATES
DEPARTMENT OF
COMMERCE
PUBLICATION



FRAME: An On-Line Correction Procedure for Quantitative Electron Probe Microanalysis

QC

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U5753

no. 796

1973

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APR 29 1974

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Issued October 1973

National Bureau of Standards Technical Note 796

Nat. Bur. Stand. (U.S.), Tech. Note 796, 51 pages (Oct. 1973)

CODEN: NBTNAE

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FRAME: An On-Line Correction Procedure for
Quantitative Electron Probe Microanalysis

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A procedure using a mini-computer for on-line correction of x-ray data from electron microprobe analysis has been developed. This program, called FRAME, allows mass fractions to be computed on-line by the ZAF approach. Besides x-ray intensities, the only input data required are the atomic numbers of the elements present, the analytical line being used, and the operating voltage. Other required parameters such as atomic weights and x-ray mass attenuation coefficients are stored or calculated by the program. FRAME is in FORTRAN IV, and requires about 4K of computer core. Results from FRAME are very close to those of COR2, which is considered to be the most accurate program available for quantitative electron probe microanalysis.

Key Words: Electron probe; mini-computer; on-line correction; quantitative microanalysis.

Introduction

The relative characteristic x-ray intensities measured with electron probe microanalyzers can be converted into concentrations of emitting elements, either by theoretical procedures, or by empirical calibration.

The theoretical procedures use generalized, though empirically adjusted expressions for the intensities of x-ray emission and absorption. They do not require, in principle, the use of any standards except elements, but they involve extensive calculation [1-3]¹. The empirical procedures, though simpler in calculation [4-5], usually require composite standards which may not be available in practice.

Electron probe microanalysis frequently consists of a sequence of measurements at various points on the specimen surface. In such case, it is useful to obtain the corrected results of the analysis at any point before deciding on the next step. Therefore, an on-line calculation procedure is desirable. However, such calculations were, in the past, available only for empirical data evaluation procedures [6].

This communication describes a correction procedure incorporated into a program (FRAME) which renders possible the on-line calculation with a minicomputer of concentrations (mass fractions) by the traditional ZAF approach [7].

Evolution of the Program FRAME

The program MULTI8, developed by us and recently published [8], evaluates the experimental x-ray intensity lines by the theoretical method. MULTI8 can be used in a batch mode on a time-shared computer; it is similar to several programs now in use for such purposes in other laboratories. This program has the following limitations:

¹ Figures in brackets indicate the literature references at the end of this paper.

a. It omits the correction for fluorescence due to the continuum, and the integration over the range of energies of the electrons decelerating within the specimen in the calculation of generated primary x-ray intensities.

b. It requires, as part of the input, the mass absorption coefficients for all lines and elements and decisions concerning the applicability of fluorescence corrections for each line and element present.

c. The ZAF procedure requires the input of relative x-ray intensities corrected for detector dead-time and background. As MULTI8 does not contain provisions for these corrections, they must be performed externally before input.

A program such as MULTI8 would therefore be inadequate for on-line use, and its accuracy may be less than optimal under certain operating conditions. As it is more basic to obtain an accurate procedure than to apply it on-line, for the next step we developed a program, COR [3], which avoids, insofar as possible, all simplifications, and incorporates all correction steps known to us, including the correction for continuum fluorescence. Our aim was to obtain a benchmark, against which other programs could be compared. In effect, diverse correction formulae can be tested by substituting them into COR2, and the effects of simplifications, omissions, and empirical fits to partial correction steps can thus be evaluated.

The correction for the continuum fluorescence requires a fairly large computation. It was found expedient in the development of COR2 to calculate internally all x-ray mass absorption coefficients required for this correction; it was also necessary to divide the wavelength of the continuum range into regions confined by the absorption edges of the elements present in the specimen. Once these provisions were included, it was relatively simple to also determine internally which

corrections for fluorescence due to characteristic lines would apply in each case. We added an auxiliary sub-program to correct for background and dead-time, and we thus arrived at a rigorous procedure which requires a minimum of input — except for a table of constants which are kept in memory — and which is quite easy to handle in practical applications. Therefore, COR2, which was conceived primarily as a research tool, has proven to be very satisfactory for routine data calculation in the batch mode and is used extensively for this purpose at the National Bureau of Standards and in several other laboratories.

In the program FRAME, which is described here, we have incorporated into a simple model based upon MULTI8 the decision making and the internal calculation of parameters which were first developed for COR2. In this manner, we have obtained a program simple enough to be used on-line in a small computer. We have also included procedures to correct the observed x-ray intensities for dead-time and background, so that the scaler readings can be directly used as input. As to the simplifications contained in MULTI8, and also used in FRAME, we have concluded that they will not observably degrade the accuracy of the procedure if certain working conditions are adopted. The working rules concerning the choice of lines and operating voltage are discussed at the end of the paper. A comparison of important features of the three programs mentioned above is found in Table I.

Fundamentals of FRAME (See Figure 1)

We have adapted from MULTI8, essentially without change, the multiplicative correction factors for absorption of primary radiation, of the atomic number effects, and of fluorescence due to characteristic lines (ZAF procedure). The program thus operates on the assumption that the relationship between concentration and background dead-time

corrected intensity ratios can be expressed by

$$C = k \cdot k_Z \cdot k_A \cdot k_F \quad (1)$$

where C is the mass fraction of the element being measured
k is the x-ray intensity ratio obtained from the
microprobe corrected for background and dead-time
k_Z is the atomic number correction
k_A is the absorption correction, and
k_F is the characteristic fluorescence correction.

As in MULTI8, there is no provision for fluorescence due to the continuum. The significant changes with respect to MULTI8 are as follows.

1. As an input, FRAME can use x-ray photon counts collected in fixed time periods on scalars. A background correction is performed by subtracting background counts, which must also be accumulated in scalars, from the line intensities. The operator must determine from where the background readings are to be collected. In the present version, the background readings are made on the standards, and FRAME distinguishes background from standard line readings by the intensities of the observed radiation.

2. A correction for counter coincidence (dead-time), according to the equation of Ruark and Brammer [9] is applied to the observed count rates for specimens and standards. A method for determining the dead-time constant, τ , has been described elsewhere [10]. The relative x-ray intensity ratio, k, is calculated for each measurement by dividing the corrected intensity from the specimen by that of the standard.

3. The energies of the K, L, and M absorption edges are calculated by means of fits based on Moseley's law. The energies of the $K\alpha$, $L\alpha$, and $M\alpha$ lines are also calculated in the same way. One of these three lines is almost always chosen as the analytical line. If conditions unfavorable for analysis result from choice of line by the operator, a warning is printed by the program. The operator can then use another line or readjust the operating voltage, depending on the nature of the difficulty.

4. The correction for absorption in the specimen of primary x-rays is carried out with use of the absorption correction factor f_p proposed by Heinrich and Yakowitz [11]:

$$1/f_p = 1 + 3 \times 10^{-6} (E_o^{1.65} - E_q^{1.65}) \chi + 4.5 \times 10^{-13} \cdot (E_o^{1.65} - E_q^{1.65})^2 \chi^2 \quad (2)$$

where $\chi = \mu \csc \psi$; and ψ is the x-ray emergence angle;

E_o and E_q are the operating voltage and critical excitation potential for the line of interest, in kilovolts.

The absorption correction is

$$k_A = f_p / f_p^* \quad (3)$$

(Here, and in what follows, terms with an asterisk refer to the unknown, and terms without superscript refer to the standard.)

The mass attenuation coefficients are calculated as proposed by Heinrich [12]. To reduce requirements for storage, least squares fits were made to the logarithm of the coefficient C and the exponent n in the general relation:

²The exponent "-13" in the third term of equation (2) is correct. This value was erroneously given as "-14" in reference [11].

$$\mu = C\lambda^n$$

in which λ is the wavelength in angstroms.

A comparison of the calculated values with those given by Heinrich [12] showed agreement within a few percent. To avoid uncertainties which arise where lines close to edges are used, a warning is printed if the analytical line falls within 100 eV of an absorption edge of another element present in the specimen.

5. The correction for atomic number used in FRAME is:

$$k_Z = \left(\frac{R^*}{R}\right) \left(\frac{\bar{S}}{\bar{S}^*}\right) \quad (4)$$

The electron backscattering correction factors, R and R^* , are those obtained by Duncumb and Reed from Monte Carlo calculations [13], fitted by us with respect to the over-voltage, U , and the mean atomic number, \bar{Z} , as follows:

$$R = R_1 - R_2 \ln (R_3 \bar{Z} + 25) \quad (5)$$

$$\text{where } R_1 = 8.73 \times 10^{-3} U^3 - 0.1669 U^2 + 0.9662 U - 0.4523$$

$$R_2 = 2.703 \times 10^{-3} U^3 - 5.182 \times 10^{-2} U^2 + 0.302 U - 0.1836$$

$$R_3 = (0.887 U^3 - 3.44 U^2 + 9.33 U - 6.43) / U^3$$

$$U = E_o / E_q, \quad \bar{Z} = \sum C_i Z_i, \quad C_i = \text{weight fraction of element } i.$$

If $U > 10$, then U is set equal to 10.

Figure 2 shows the values given by Duncumb and Reed and the curves predicted by equation (5). The value of R^*

$$\text{is obtained from } R^* = \sum_{k_i=1}^n C_i R_i. \quad (6)$$

Equation (5) is simpler than the fifth-order fit in voltage and atomic number [14] used in MULTI8 and COR2, and gives very similar results.

The term \bar{S} in equation (4) is an average stopping power according to a form of Bethe's equation [15]. The stopping power calculation is identical to that in MULTI8, except that the mean electron energy is obtained, as previously proposed by Thomas [16], as follows:

$$\bar{E} = \frac{E_0 - E_q}{2}.$$

This approximation is sufficiently accurate except at extreme overvoltage values which should be avoided for accurate analysis [17]. The atomic weights, required for the stopping power calculation, are stored in the program.

As in MULTI8, the characteristic fluorescence correction is performed with the aid of Reed's equation [18]. MULTI8 and COR2 require the input of fluorescent yields; in COR2 these are stored in the permanent data file. In FRAME we use the fluorescence yields for K and L shells which are calculated by means of empirical fits. No fluorescence correction is carried out if fluorescence is excited by M lines. We assume that the absorption jump ratio is 0.88 for the K edge and 0.75 for the L_3 edge.

FRAME contains the following provisions which decide whether fluorescence correction is required. Call E_ρ the energy of the exciting line (K- or L-line), E_K and E_L the critical excitation potentials for the excited K- and L-lines.

If $0 < (E_\rho - E_K) \leq 5$ then the K-line is indirectly excited.

If $0 < (E_\rho - E_L) \leq 3.5$, then the L-line is indirectly excited.

If fluorescent excitation occurs, the program prints a message to this effect. Then, it calculates the new mass attenuation coefficients required for Reed's equation [18] and performs the necessary calculation for the fluorescence effect.

This automatic selection of corrections for fluorescent excitation where necessary eliminates the need for the operator to decide if such corrections must be applied.

As mentioned previously [2], the iteration procedure used in MULTI8 and COR2 — and again in FRAME — converges very rapidly. For this reason, we stop the iteration after three cycles in order to reduce the time of computation. The resulting errors are smaller than the errors of measurement.

Structure of the Program Versions FRAME2 to FRAME4

The version of FRAME which is reproduced in Appendix I is called FRAME3. The list of symbols used in FRAME3 is in Appendix II. This version is adapted for on-line processing of microprobe data on an EMR-6135³ computer. FRAME2 is a computationally identical version for batch-processing on UNIVAC 1108; FRAME4 is adapted to a telephone-transmittal time-share system. The statement lists of FRAME2 and FRAME4 are available on request. The sectors for input and output will require modifications for use on other computers. These adaptations should be easy to carry out. Areas of the program specific to our set-up are identified by comment statements in the appended program list.

FRAME3 can be considered a model for on-line application. The first part (2.8K) of this version (with subroutines CFCAL and MAC) performs all preliminary calculations which are

³ Identification of any specific instrument does not imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

independent of the composition of the specimen and standard, and do not, therefore, require x-ray intensity data. The second part (with subroutine QUDAT, 1.5K) uses the measured x-ray intensities from standards and unknowns to compute intensity ratios, which then are converted to concentrations, by means of the correction factors obtained in CFCAL. For a particular case — which may extend over several points to be analyzed — the first part is calculated only once, while the second part is executed every time a measurement is being performed. The interaction of the main program with the subprograms can be observed on the flow-diagram in Figure 1.

Limitations of FRAME

As mentioned above, the limitations of available memory have required several simplifications, (e.g., omission of the correction for fluorescence due to the continuum). These simplifications will not cause significant increase in the error of the analyses, as long as the following operational conditions are maintained:

1. The overvoltage (E_o/E_q) should not be smaller than 1.5.
2. The primary x-ray absorption factor, f_p , should not be smaller than 0.75.
3. If an x-ray line falls within 100 eV on the high energy side of an absorption edge, or 30 eV on the low energy side of any element present, then this line should not be used because of the uncertainties concerning mass absorption coefficients near the edge.

The restrictions imposed by these conditions are not serious or specific to FRAME, since working outside these specifications is unwise, even when a more detailed computer program is used. FRAME3 produces a printed warning when an analysis is computed with disregard of the above rules. In

the case of f_p , the warning criterion is applied to the absorption factor of the standard. For further control of conditions, the values of f_p for the specimen are printed in the output of FRAME.

In our version of FRAME, it is assumed that the x-ray emergence angle is equal to 52.5° . The value of this angle can be changed by a single statement, and even a change to different angles for each spectrometer could be introduced without much effort.

The number of elements in the specimen is presently limited, in our versions, to six. Since the program is primarily intended for on-line use, it is assumed that the six intensities are read simultaneously. However, the number of x-ray lines which can be read simultaneously in an instrument cannot exceed that of the spectrometers, unless part of the information is provided by an energy-dispersive spectrometer. Since this limitation may be significant in the analysis of minerals, we consider introducing, in a later version, modifications which would permit the reading of x-ray intensities in two steps in time, so that spectrometers could be realigned to read more than one line per analysis.

We have also assumed that all measurements for a case were performed at the same operating voltage. If a two-step read-out is considered, this criterion will be modified so that, if desired, some lines can be read at a lower voltage than the rest. This experimental procedure is, however, awkward and is not used frequently. Therefore, the limitation to a single operating voltage is not stringent.

All versions of FRAME permit the determination of one element by difference. The use of stoichiometric relations to determine the concentration of one element is provided in FRAME4 and can easily be incorporated into other versions.

At present, it is not possible to replace the mass absorption coefficients calculated in the program by others provided externally. Such a feature will be incorporated later, as well as an option to assume that the concentration of certain elements is known. We also plan to indicate at a later date, modifications to obtain atomic fractions rather than weight fractions. The number of iterations, which presently is limited to three, could be changed by a simple statement. However, as indicated by the data presented in Tables II and III, computation beyond three iterations does not provide any significant improvement in accuracy.

Comparison of Results from FRAME with COR2

We have compared the FRAME and COR correction programs, using the measurements on 228 binary alloys listed by Poole [19], as well as 16 analyses on the binary alloys Au-Ag and Au-Cu designated NBS Standard Reference Materials 481 and 482, respectively, [20]. The analyses listed by Poole were performed in several laboratories from all over the world, and they cover a variety of systems and experimental conditions.

A comparison of the results by FRAME with those by COR2 for the Poole-Thomas compilation is shown in Figure 3. We note that the disagreement between COR2 and FRAME is small compared with the errors shown in the diagram for COR2. We also note that larger discrepancies occur when the operating conditions are outside the suggested boundaries.

Analyses performed at NBS are presented in Tables II, III, and IV. The results show that FRAME is suitable for a wide variety of analytical studies. They also show, in the case of the two minerals analyzed, that the method of determining the background level was not critically important.

An example of the input and output for the analysis of the Diopside 65 - Jadeite 35 is shown in Table V. All required input statements follow a colon or question mark. The program

version is FRAME4. Table V shows that the concentrations obtained by calculating oxygen by difference or by stoichiometric relations are the same. The FRAME4 routine was run on a time-shared computer with background dead-time corrected relative intensity ratios (k-values) already calculated. The k-values shown are referred to pure element standards as calculated from the mineral standards listed in Table IV.

Example Showing On-line Analysis using FRAME

An iron-silicon alloy containing 3.22% Si and 96.8% Fe, designated SRM-483, was analyzed. This material is certified to be homogeneous with respect to iron and silicon [21]. Five individual analyses were carried out. The results, as printed on a teletype display during analysis, are shown in Table VI. The average silicon content is 3.34%, and the average iron content is 96.2%.

Conclusions

The FRAME program is entirely suitable for on-line or off-line use to reduce data from the electron probe micro-analyzer to concentrations of the elements present. FRAME employs the analytical ZAF approach, but only occupies about 4K of computer memory. The FRAME program should be readily adaptable to most mini-computers for use in many microprobe laboratories.

Comparisons have shown that FRAME give virtually the same results as the much larger batch-type program, COR, provided that fluorescence effects due to the continuum can be safely neglected. All of the data discussed in this paper, including those reported by Poole, fall into this category. The input requirements for FRAME are virtually the same,

however (see Table I). Results on two minerals, Johnstown Hypersthene and Diopside 65 - Jadeite 35, give results close to the chemical analysis (Tables III, IV and V). Furthermore, the results were only slightly affected by the method of determining background level or by using FRAME to calculate the results obtaining the oxygen content by difference or by stoichiometric relations.

Table Ia. Comparison of NBS programs for quantitative microprobe analysis

	<u>COR2</u>	<u>MULTI8</u>	<u>FRAME3</u>
<u>MECHANICAL ASPECTS</u>			
Memory	50 K	4 K	4 K
Intended mode of operation	Batch	Time-share	Time-share or dedicated
Input	Cards and magnetic tape	Teletype	Teletype or on-line
Output	Line printer	Teletype	Teletype

Table Ib. Comparison of NBS programs for quantitative microprobe analysis

	<u>COR2</u>	<u>MULTI8</u>	<u>FRAME3</u>
<u>INPUT</u>			
No. of elements	Yes	Yes	Yes
Atomic numbers	Yes	Yes	Yes
Atomic weights	Data file	Yes	Stored
Operating voltage	Yes	Yes	Yes
Line	Yes	No	Yes
Crit. excit. potential	Data file	Yes	Calculated
Fluorescence yield	Data file	Yes	Calculated
μ/ρ	Calculated	Yes	Calculated
Fluorescence-decisions	Made internally	Yes	Made internally
Valences	Yes (if used)	Yes (if used)	Yes (if used)
X-ray intensities	Raw or k-values	k-values only	Raw or k-values

Table Ic. Comparison of NBS programs for quantitative microprobe analysis

	<u>COR2</u>	<u>MULTI8</u>	<u>FRAME3</u>
	<u>CONTENTS and OPTIONS</u>		
Background	Yes	No	Yes
Dead-time	Yes	No	Yes
Atomic-number correction	Philibert-Tixier	Thomas modified	Thomas modified
Absorption correction	Philibert-Duncumb Heinrich sigma	Philibert-Duncumb Heinrich sigma	Heinrich-Yakowitz
Characteristic fluorescence	Hénoc	Reed	Reed
Continuum fluorescence	Hénoc	No	No
k-values	Yes	Yes	Yes
Element by difference	Yes	Yes	Yes
Element by stoichiometry	Yes	Yes	Yes
Element with fixed conc.	Yes	No	Not yet

Table II. Analysis of alloys: SRM's 481 and 482 (Au-Ag and Cu-Au)

Element and line	Operating voltage (kv)	$k_{\text{meas.}}$	$C_{\text{FRAME Itr.*I}}$	$C_{\text{FRAME Itr.*II}}$	$C_{\text{FRAME Itr.*III final}}$	C_{COR2}	C Certified	$f(x)$
Au-M α	10	0.201	0.227	0.227	0.227	0.234	0.2243	0.84
Au-M α	10	0.362	0.399	0.399	0.399	0.409	0.4003	0.84
Au-M α	10	0.559	0.598	0.598	0.598	0.608	0.6005	0.84
Au-M α	10	0.771	0.799	0.799	0.799	0.806	0.8005	0.85
Ag-L α	10	0.764	0.778	0.776	0.776	0.764	0.7758	0.89
Ag-L α	10	0.584	0.600	0.598	0.598	0.582	0.5993	0.86
Ag-L α	10	0.386	0.399	0.398	0.398	0.382	0.3992	0.83
Ag-L α	10	0.191	0.198	0.197	0.197	0.187	0.1996	0.80
Cu-K α	25	0.240	0.206	0.205	0.205	0.203	0.1983	0.90
Cu-K α	25	0.453	0.413	0.411	0.411	0.402	0.3964	0.91
Cu-K α	25	0.651	0.619	0.618	0.617	0.604	0.5992	0.93
Cu-K α	25	0.834	0.818	0.817	0.817	0.805	0.7985	0.95
Au-L α	25	0.745	0.788	0.795	0.795	0.794	0.8015	0.92
Au-L α	25	0.529	0.592	0.598	0.598	0.598	0.6036	0.91
Au-L α	25	0.331	0.391	0.395	0.395	0.396	0.4010	0.90
Au-L α	25	0.154	0.192	0.193	0.193	0.194	0.2012	0.89

*Itr. is the abbreviation for iteration.

Table III. Analysis of a silicate: Diopside 65 - Jadeite 35
 (done with oxygen by stoichiometry using FRAME4)

10 kV = E_o

	<u>Na</u>	<u>Mg</u>	<u>Al</u>	<u>Si</u>	<u>Ca</u>
k_m^*	0.0281	0.0593	0.0363	0.236	0.111
C^\dagger - Iteration I	0.0392	0.0730	0.0439	0.270	0.121
C - Iteration II	0.0384	0.0718	0.0433	0.268	0.121
C - Iteration III	0.0384	0.0718	0.0433	0.268	0.121
C - Chemical Analysis	0.0398	0.073	0.0467	0.266	0.120
f_p	0.69	0.77	0.81	0.86	0.97

* k_m - measured intensity ratio corrected for background and
 dead-time effects.

$^\dagger C$ - mass fraction

Table IV. Analysis of the silicates: Johnstown Hypersthene and Diopside 65 - Jadeite 35, using FRAME4

Chemical analysis of minerals used

<u>Mineral</u>	<u>Na</u>	<u>Mg</u>	<u>Al</u>	<u>Si</u>	<u>Ca</u>	<u>Ti</u>	<u>Fe</u>
NaCl	0.393						
SiO ₂				0.467			
Fayalite	--	--	--	0.137	--	--	0.525
Diopside·2%TiO ₂	--	0.110	--	0.254	0.181	0.012	--
Garnet 110752	--	0.043	0.120	0.188	0.129 ₅	0.002	0.088
Diopside 65 - Jadeite 35	0.0398	0.073	0.0467	0.266	0.120	--	--
Johnstown Hypersthene	--	0.164	0.002	0.251	0.0099	0.0013	0.121

Standards used:

NaCl for Na in diopside 65 - Jadeite 35

SiO₂ for Si in both

Fayalite for Fe in Johnstown Hypersthene

Diopside·2%TiO₂ for Mg, Ca, Ti, in Johnstown Hypersthene

Garnet 110752 for Mg, Al, Ca, in Diopside 65 - Jadeite 35

Al₂O₃ used as BKG specimen for Fe, Si, Mg, Ca, Ti

MgO used as BKG specimen for Na, Al, Ca

} Scheme I

Off-peak (above and below used for all elements

Scheme II

Table IV. Continued

Analysis of Johnstown Hypersthene**

 $E_o = 15 \text{ kV}$

	<u>Mg</u>	<u>Si</u>	<u>Ca</u>	<u>Ti</u>	<u>Fe</u>
k_m^* - S1	0.114	0.202	0.011	0.0008	0.104
k_m^* - S2	0.117	0.202	0.010	0.0007	0.104
C^\dagger					
CALC - S1	0.165	0.260	0.0110	0.0009	0.121
CALC - S2	0.169	0.260	0.0110	0.0008	0.121
C - Chemical Analysis	0.164	0.251	0.0099	0.0013	0.121

Analysis of Diopside 65 - Jadeite 35

 $E_o = 10 \text{ kV}$

	<u>Na</u>	<u>Mg</u>	<u>Al</u>	<u>Si</u>	<u>Ca</u>
k_m^* - S1	0.0281	0.0593	0.0363	0.236	0.111
k_m^* - S2	0.0275	0.0595	0.0363	0.236	0.111
C^\dagger					
CALC - S1	0.0384	0.0716	0.0433	0.268	0.121
CALC - S2	0.0375	0.0718	0.0433	0.268	0.121
C - Chemical Analysis	0.0398	0.073	0.0467	0.266	0.120

**Analysis carried out calculating oxygen by stoichiometry was exactly the same as that carried out calculating oxygen by difference.

S1 - Background taken on Al_2O_3 or MgO

S2 - Background taken off-peak

k_m^* - Measured intensity ratio corrected for background and dead-time effects.

C^\dagger - Mass fraction

Table V. Input-output for FRAME4 —
analysis of Diopside 65 - Jadeite 35

(a) Analysis with oxygen calculated by difference.

```

NUMBER OF ELEMENTS AND EO: 6 10
ATOMIC NUMBER AND LINE FOR EACH: 11 1 12 1 13 1 14 1 20 1 8 0
WARNING - THE LINE OF 11 IS CLOSE TO AN EDGE OF 11
ELEMENT 11 IS EXCITED BY ELEMENT 12
ELEMENT 11 IS EXCITED BY ELEMENT 13
ELEMENT 11 IS EXCITED BY ELEMENT 14
ELEMENT 11 IS EXCITED BY ELEMENT 20
ELEMENT 12 IS EXCITED BY ELEMENT 13
ELEMENT 12 IS EXCITED BY ELEMENT 14
ELEMENT 12 IS EXCITED BY ELEMENT 20
ELEMENT 13 IS EXCITED BY ELEMENT 14
ELEMENT 13 IS EXCITED BY ELEMENT 20
ELEMENT 14 IS EXCITED BY ELEMENT 20
VALENCES:0 0 0 0 0 0
STANDARDS DATA? NO
K-VALUES: .0281 .0593 .0363 .2364 .111
AT. NO. K-VALUE CONCENTRATION F(X)

11. .0281 .0384 .6867
12. .0593 .0718 .7690
13. .0363 .0433 .8121
14. .2364 .2685 .8555
20. .1110 .1207 .9670
8. 0.0000 .4573 1.0000
TOTAL C = 1.0000

```

(b) Analysis with oxygen calculated stoichiometrically.

```

NUMBER OF ELEMENTS AND EO: 6 10
ATOMIC NUMBER AND LINE FOR EACH: 11 1 12 1 13 1 14 1 20 1 8 0
WARNING - THE LINE OF 11 IS CLOSE TO AN EDGE OF 11
ELEMENT 11 IS EXCITED BY ELEMENT 12
ELEMENT 11 IS EXCITED BY ELEMENT 13
ELEMENT 11 IS EXCITED BY ELEMENT 14
ELEMENT 11 IS EXCITED BY ELEMENT 20
ELEMENT 12 IS EXCITED BY ELEMENT 13
ELEMENT 12 IS EXCITED BY ELEMENT 14
ELEMENT 12 IS EXCITED BY ELEMENT 20
ELEMENT 13 IS EXCITED BY ELEMENT 14
ELEMENT 13 IS EXCITED BY ELEMENT 20
ELEMENT 14 IS EXCITED BY ELEMENT 20
VALENCES:1 2 3 4 2 2
STANDARDS DATA? NO
K-VALUES: .0281 .0593 .0363 .2364 .111
AT. NO. K-VALUE CONCENTRATION F(X)

11. .0281 .0384 .6871
12. .0593 .0718 .7693
13. .0363 .0433 .8123
14. .2364 .2685 .8557
20. .1110 .1207 .9671
8. 0.0000 .4531 1.0000
TOTAL C = .9957

```

Table VI. Results of analysis of SRM 483
 containing 0.0322 Si - 0.968 Fe.
 Analysis performed "on-line" at NBS

FRAME3 1118HRS 187 DAY, 1973

ELECTRON PROBE ANALYSIS 15.0 KV

ELEMENT 14 IS EXCITED BY ELEMENT 26

Z-LINE	Z-ABSORBER	MASS ABS. COEFF.
14	14	330.0
14	26	2495.8
26	14	113.8
26	26	70.9

AT. NO.	K-VALUE	CONC	F(CHI)
14	.0237	.0337	.5544
26	.9532	.9595	.9834
TOTAL C = .9932			
14	.0233	.0332	.5543
26	.9583	.9640	.9834
TOTAL C = .9972			
14	.0238	.0339	.5544
26	.9530	.9593	.9834
TOTAL C = .9932			
14	.0234	.0333	.5543
26	.9578	.9635	.9834
TOTAL C = .9968			
14	.0236	.0337	.5544
26	.9552	.9612	.9834
TOTAL C = .9949			

FRAME

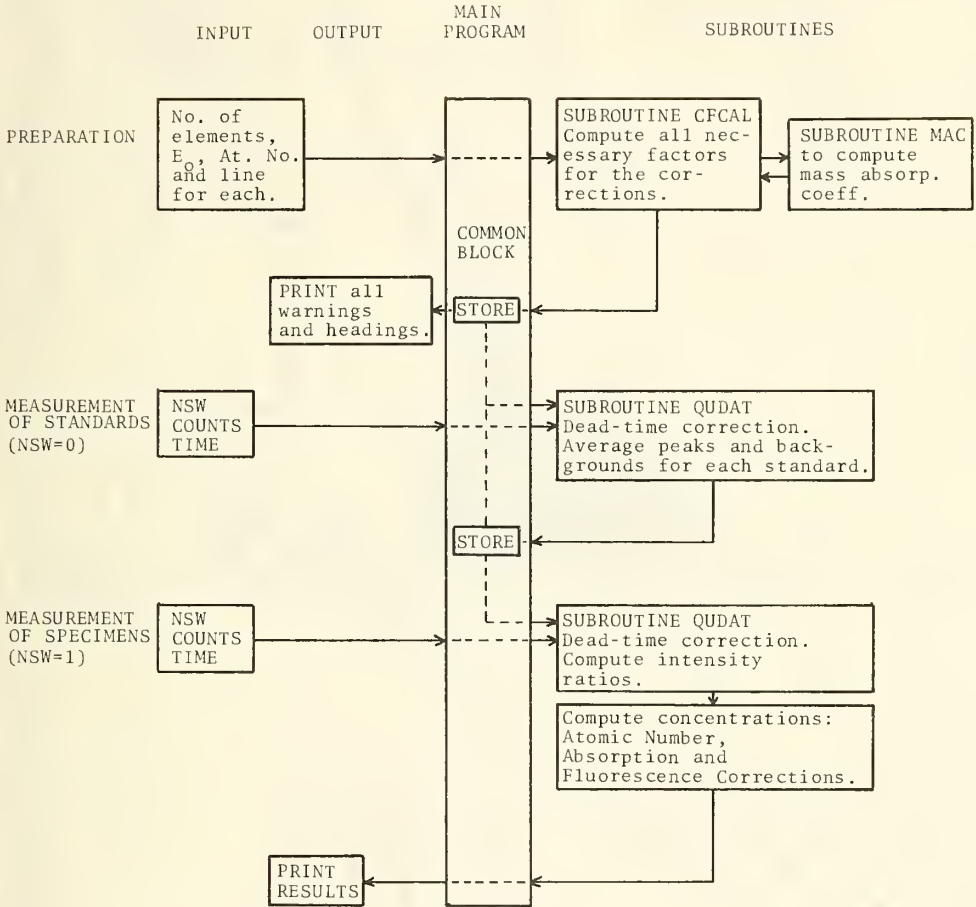


Figure 1. Flow sheet showing operations carried out by FRAME program.

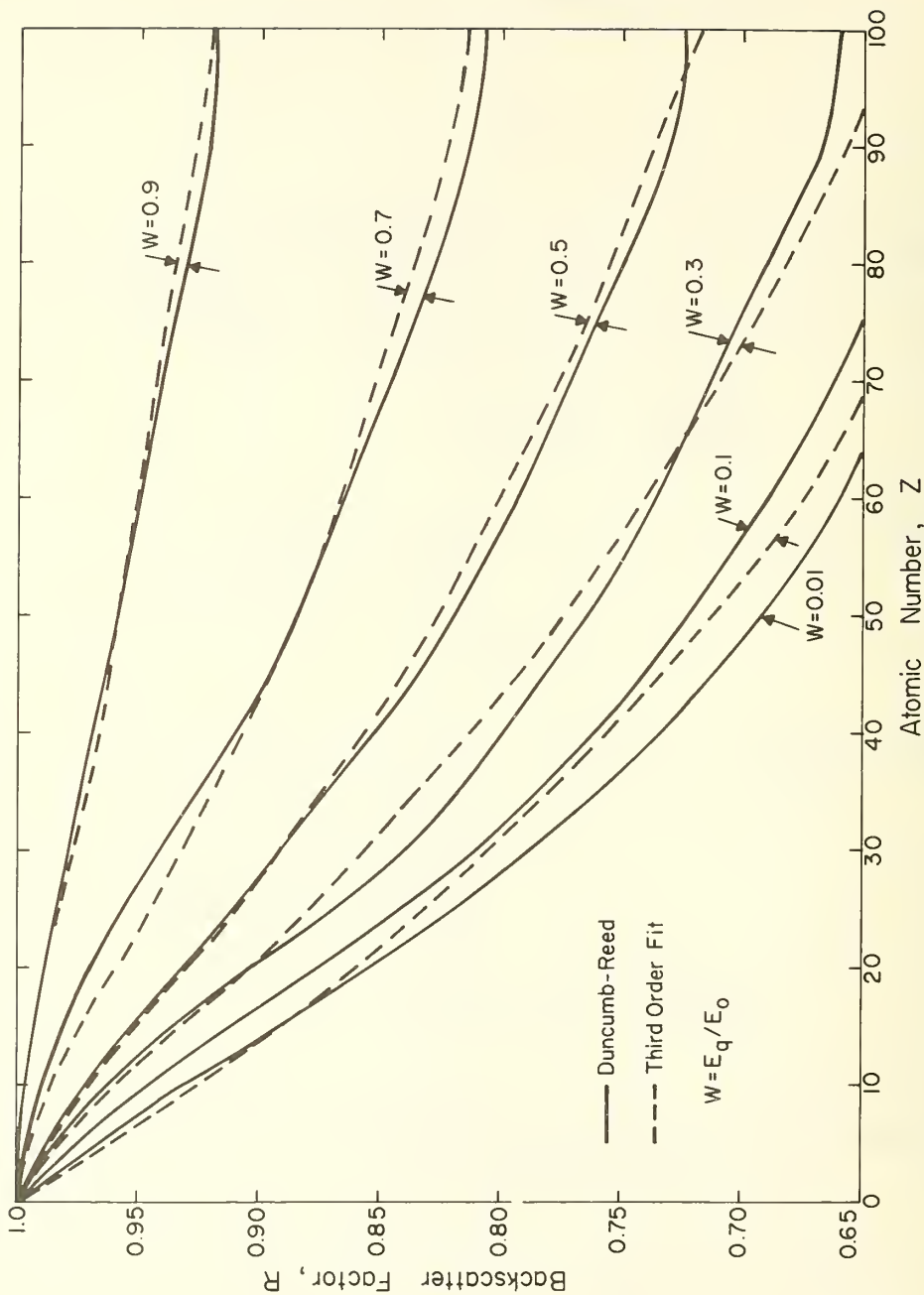


Figure 2. Analytical fit to backscatter factor R . Solid lines given by Duncumb and Reed [5]; dotted lines fitted by equation 5. For $W < 0.1$ the FRAME program uses R corresponding to $W=0.1$.

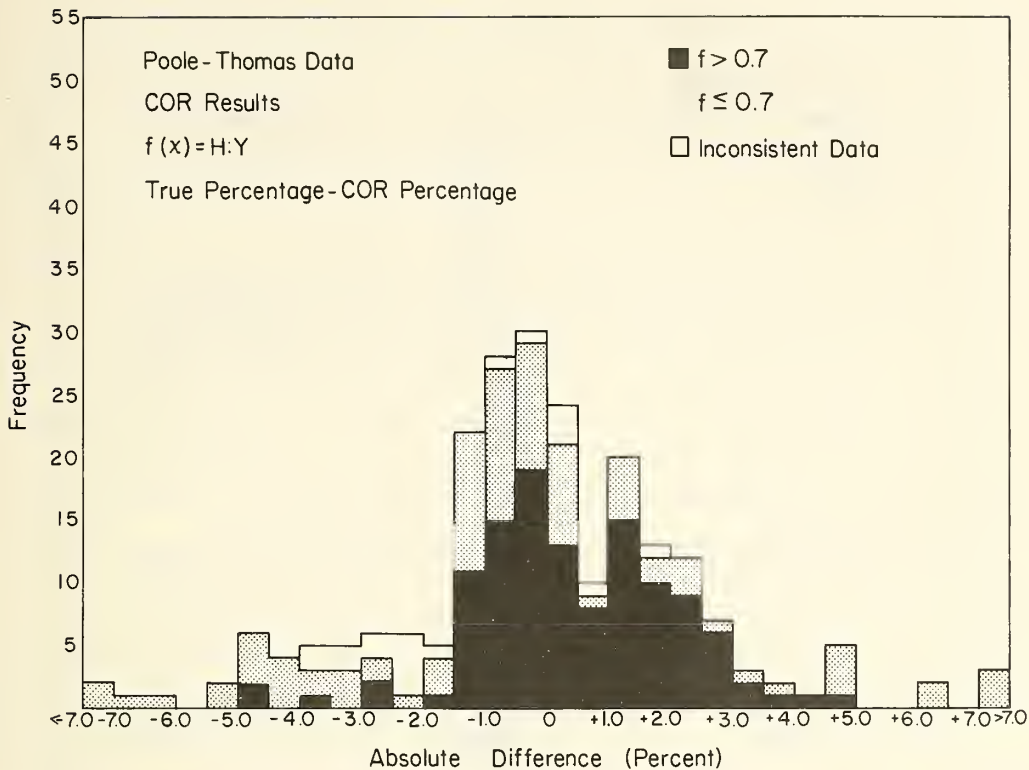


Figure 3a. Results of analysis on alloy data quoted by Poole [19] as computed by COR using the Heinrich-Yakowitz $f(\chi)$ expression [11].

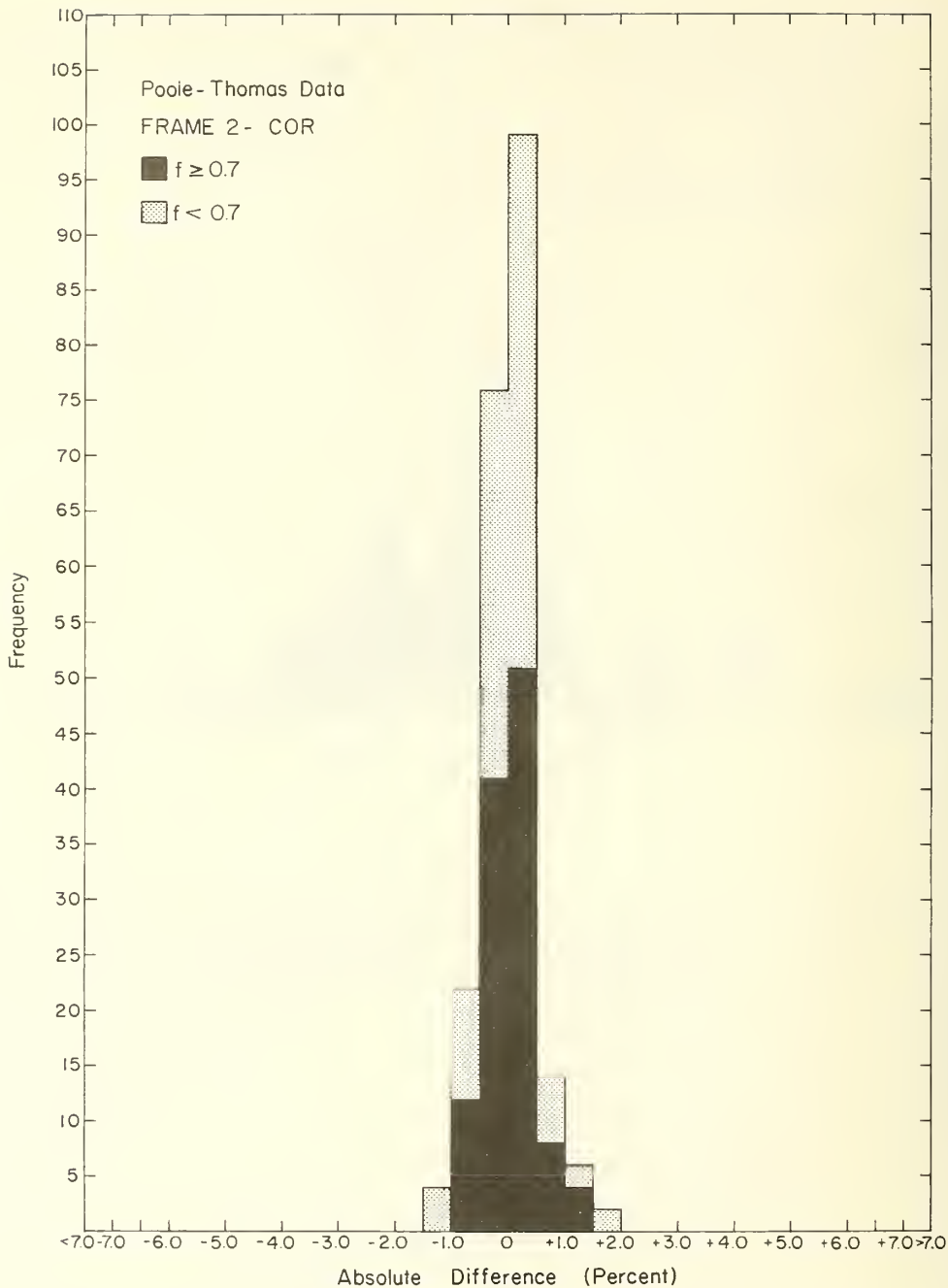


Figure 3b. Comparison of FRAME2 and COR for the alloy data shown by Figure 3a.

References

- [1] Heinrich, K. F. J., Editor, "Quantitative Electron Probe Microanalysis," NBS Special Publication 298, U.S. Government Printing Office, Washington, D. C. (1968).
- [2] Heinrich, K. F. J., *Anal. Chem.* 44, 350 (1972).
- [3] Hénoc, J., Heinrich, K. F. J., and Myklebust, R. L., "A Rigorous Correction Procedure for Quantitative Electron Probe Microanalysis (COR 2)," U.S. Natl. Bur. Stds. Technical Note 769, U.S. Government Printing Office, Washington, D. C. (1973).
- [4] Ziebold, T. O. and Ogilvie, R. E., *Anal. Chem.* 36, 322 (1964).
- [5] Bence, A. E. and Albee, A. L., *J. Geology* 76, 382 (1968).
- [6] Chodos, A. A. and Albee, A. L., Proc. Sixth Natl. Conf. on Electron Probe Analysis, Pittsburgh, Pa., p. 15A (1971).
- [7] Philibert, J. and Tixier, R. in "Quantitative Electron Probe Microanalysis," Heinrich, K. F. J., Editor, NBS Special Publication 298, p. 13, U.S. Government Printing Office, Washington, D. C. (1968).
- [8] Heinrich, K. F. J., Myklebust, R. L., Yakowitz, H., and Raspberry, S. D., "A Simple Correction Procedure for Quantitative Electron Probe Microanalysis," U.S. Natl. Bur. Stds. Technical Note 719, U.S. Government Printing Office, Washington, D. C. (1972).
- [9] Ruark, A. and Brammer, F. E., *Phys. Rev.* 52, 322 (1937).
- [10] Heinrich, K. F. J., Vieth, D. L., and Yakowitz, H., in Advances in X-Ray Analysis, Vol. 9, Mallett, G. R., Fay, M. J., and Mueller, W. M., Editors, p. 208, Plenum Press, New York (1966).
- [11] Heinrich, K. F. J., Yakowitz, H., and Vieth, D. L., Proc. Seventh Natl. Conf. on Electron Probe Microanalysis, San Francisco, CA, paper no. 3, 6 pp. (1972).
- [12] Heinrich, K. F. J., in The Electron Microprobe, McKinley, T. D., Heinrich, K. F. J., and Wittry, D. B., Editors, p. 296, John Wiley & Sons, Inc., New York (1966).

- [13] Duncumb, P. and Reed, S. J. B., in "Quantitative Electron Probe Microanalysis," Heinrich, K. F. J., Editor, NBS Special Publication 298, p. 133, U.S. Government Printing Office, Washington, D. C. (1968).
- [14] Heinrich, K. F. J., in "Present State of the Classical Theory of Quantitative Electron Probe Microanalysis," NBS Technical Note 521, p. 5, U.S. Government Printing Office, Washington, D. C. (1970).
- [15] Heinrich, K. F. J., Anal. Chem. 44, 350 (1972).
- [16] Thomas, P. M., AERE Report 4593, U. K. Atomic Energy Authority (1964). Also see reference [12].
- [17] Heinrich, K. F. J. and Yakowitz, H., Mikrochim. Acta 1970, p. 123.
- [18] Reed, S. J. B., Brit. J. Appl. Phys. 16, 913 (1965).
- [19] Poole, D. M., in "Quantitative Electron Probe Microanalysis," Heinrich, K. F. J., Editor, NBS Special Publication 298, p. 93, U.S. Government Printing Office, Washington, D. C. (1968).
- [20] Heinrich, K. F. J., Myklebust, R. L., and Rasberry, S. D., "Preparation and Evaluation of SRM's 481 and 482 Gold-Silver and Gold-Copper Alloys for Microanalysis," NBS Special Publication 260-28, U.S. Government Printing Office, Washington, D. C. (1971).
- [21] Yakowitz, H., Fiori, C. E., and Michaelis, R. E., "Homogeneity Characterization of Fe-3Si Alloy," NBS Special Publication 260-22, U. S. Government Printing Office, Washington, D. C. (1971).

APPENDIX I

Listing of FRAME3 in FORTRAN IV

C

FRAME3

C ON-LINE PROGRAM FOR QUANTITATIVE ELECTRON PROBE MICROANALYSIS.
C FORTRAN IV MAY 22, 1973
C THE INPUT AND OUTPUT WILL NEED TO BE MODIFIED TO FIT THE COMPUTER
C BEING USED.
C NOTE - ALL STATEMENTS PRECEDED BY AN * RESULT FROM SYSTEM ROUTINES
C ON THE ANALYTICAL CHEMISTRY DIVISIONS COMPUTER FOR DATA ACQUISITION
C AND CONTROL.

SUBROUTINE CFCAL(TAB)

C THIS ROUTINE IS LOADED FIRST TOGETHER WITH MAC. READ THE INITIAL
C INPUT PARAMETERS AND COMPUTE ALL NECESSARY CONSTANTS.

```
INTEGER MESS(40),PST
* INTEGER TAB,CRLF
REAL JI
* LOGICAL SETSW
DIMENSION Z(6),EC(9,6),EL(3,6),LI(6),EQ(6),E(6),AC(6,6),FAC(6,6),H
1(6),FC(6),R(6,6),S(6,6),U(6),KEY(6,6),C1(9),C2(9),C3(9)
2 ,P(6),R1(6),OM(6),A(6),X(6),AN(92),Y(6)
* DIMENSION ISPC(4),IFDB(3)
DIMENSION MHD(5),IZ(6)
DIMENSION XL(6),XXK(6),BK(6),PK(6),PB(6)
DIMENSION IBUF(50),SC(7)
COMMON/ENER/EL,EC
COMMON/STORE/N,N1,LI,S,R,AC,H,KEY,FAC,EQ,EO,P,OM,R1,X,Y,A,FC,Z,SC,
1SETSW,IBUF,PST,NFSW,NFLAG,XL,XXK,BK,PK,PB,COSEC
* COMMON/ESTOR/IDUM
EQUIVALENCE(MESS(1),CRLF)
DATA AN/1.008,4.003,6.94,9.013,10.82,12.011,14.007,16.,19.,20.18,
122.99,24.31,26.98,28.09,30.97,32.06,35.45,39.95,39.1,40.08,44.96,
247.9,50.94,52.,54.94,55.85,58.93,58.71,63.54,65.37,69.72,72.59,
374.92,78.96,79.91,83.8,85.47,87.62,88.91,91.22,92.91,95.94,99.,
4101H1,102.9,106.4,107.9,112.4,114.8,118.7,121.8,127.6,126.9,131.3,
5132H9,137.3,138.9,140.1,140.9,144.2,145.,150.4,152.,157.2,158.9,
6162.5,164.9,167.3,168.9,173.,175.,178.5,180.95,183.85,186.2,190.2,
7192.2,195.1,196.97,200.6,204.4,207.2,209.,210.,210.,222.,223.,226.
8,227.,232.,231.,238./
DATA C1/-0.0397931,-0.0339160,-0.0865397,-0.2283427,1.251788,
1.8349031,.4422173,.2514096,.2729506/
DATA C2/2.423000,2.825262,3.323153,4.311724,-7.837999,
1-4.149247,-.9792409,.9319132,.6889060/
DATA C3/5.509104,9.035256,10.25054,12.00253,-11.58026,
1-3.338016,3.153478,8.035612,7.424300/
```

C THE FOLLOWING CONSTANTS ARE NEEDED BY OUR COMPUTER TO PRINT OUT THE
C RESULTS ON A TELETYPE.

```
* DATA ISPC/2*$41503,2*$41101/
* DATA IADTTY/$176145/
* DATA IFDB(2)/0/
* DATA IPRI/10/
* DATA CRLF/$41101/
```

C THE FOLLOWING STATEMENTS APPLY TO OUR COMPUTER AND ARE EQUIVALENT TO
C READING THE INPUT DATA.
C THIS SECTION READS AND DECODES A BUFFER CONTAINING THE INPUT
C PARAMETERS - EO(KILOVOLTS), LI(LINE- 1=K, 2=L, 3=M, 0= NOT ANALYZED),
C N(NUMBER OF ELEMENTS), IZ(ATOMIC NUMBER).

```

*      CALL RESTOR(TAB)
*      I = KBCDBN(IEXTR(5,16,IBUF(8)))
      EO=I/10.
*      LI(1) = IEXTR(13,16,IRUF(12))
*      LI(2) = IEXTR(10,12,IRUF(12))
*      LI(3) = IEXTR(7,9,IBUF(12))
*      LI(4) = IEXTR(4,6,IBUF(12))
*      LI(5) = IEXTR(1,3,IBUF(12))
*      LI(6) = IEXTR(13,16,IRUF(11))
*      N = IEXTR(1,4,IBUF(11))
*      IZ(1)= KBCDBN(IEXTR(9,16,IBUF(14)))
*      IZ(2)= KBCDBN(IEXTR(1,8,IRUF(14)))
*      IZ(3)= KBCDBN(IEXTR(9,16,IBUF(13)))
*      IZ(4)= KBCDBN(IEXTR(1,8,IRUF(13)))
*      IZ(5)= KBCDBN(IEXTR(9,16,IBUF(16)))
*      IZ(6)= KBCDBN(IEXTR(1,8,IRUF(16)))
      DO 10 I=1,6
10  Z(I)=FLOAT(IZ(I))
      COSEC=1.2605
      N1=N
      IF(LI(N).EQ.0) N1=N-1

C      OPEN THE BUFFERS FOR STORING TELETYPE OUTPUT.

*      CALL ALU(LUTY,IADTTY)
*      CALL OPEN(LUTY,IFDB)
*      IF(IFDB(2).LE.0) CALL ERROR(721,TAR)
*      NW=4

C      RIOS IS THE SYSTEM I/O ROUTINE USED HERE FOR SPOOLING TELETYPE OUTPUT.
C      EVERYTHING IS PRINTED ON THE TELETYPE WHEN THIS FILE IS CLOSED
C      (STATEMENT 777). SCHENT IS THE SCHEDULED PROGRAM ENTRY FROM RIOS.
C      IS REPRESENTS STATUS RETURNED FROM RIOS.

*      CALL RIOS2(0,0,IPRI,$1,LUTY,IADTTY,IS,NW,ISPC)
*      1 SCHENT
*      IF(IS.LE.0) GO TO 8

C      GET THE TIME OF YEAR.

*      CALL GDB(MHD)
      MHD(4)=MHD(4)+MHD(5)
      NYEAR=1973

C      PRINT TITLE AND DATE.

      ENCODE(72,2004,MESS(2),NC) MHD(4),MHD(3),NYEAR
2004  FORMAT('  FRAME3  ',I4,'HRS ',I3,' DAY, ',I4)
*      NW=18
*      CALL RIOS2(0,0,IPRI,$9,LUTY,IADTTY,IS,NW,MESS)
*      9 SCHENT
*      IF(IS.LE.0) GO TO 8
      MESS(2)=CRLF

C      PRINT KILOVOLTAGE.

      ENCODE(72,2001,MESS(3),NC) EO
2001  FORMAT('ELECTRON PROBE ANALYSIS',1X,F4.1,' KV ')
      MESS(19)=CRLF
*      NW=19
*      CALL RIOS2(0,0,IPRI,$2,LUTY,IADTTY,IS,NW,MESS)

```

```

* 2 SCHEM
* IF(IS.LE.0) GO TO 8

C COMPUTE ALL ABSORPTION EDGES AND X-RAY LINES.

DO 20 I=1,N
ZL=ALOG(Z(I))
DO 30 M=1,9
30 EC(M,I)=EXP(C1(M)*ZL*ZL+C2(M)*ZL-C3(M))
EL(1,I)=EXP(-.0188608*ZL*ZL+2.218585*ZL-5.170825)
EL(2,I)=EXP(-.1239411*ZL*ZL+3.295325*ZL-9.758363)
EL(3,I)=EXP(-.4755497*ZL*ZL+6.846618*ZL-20.08326)
NZ=Z(I)
A(I)=AN(NZ)
L=LI(I)
IF(L.EQ.0) GO TO 20
LS=L*L
EQ(I)=EC(LS,I)
E(I)=EL(L,I)
U(I)=E0/EQ(I)
Y(I)=U(I)
IF(U(I).GT.10.) U(I)=10.

C PRINT WARNING IF THE OVERVOLTAGE IS LESS THAN 1.5.

IF(U(I).GE.1.5) GO TO 20
ENCODE(72,2006,MESS(2),NC) IZ(I)
2006 FORMAT('WARNING - THE OVERVOLTAGE OF ELEMENT ',I2,' IS BELOW 1.5')
* NW=27
* CALL RIOS2(0,0,IPRI,$800,LUTY,IADTTY,IS,NW,MESS)
* 800 SCHEM
* IF(IS.LE.0) GO TO 8
20 CONTINUE
DO 50 I=1,N1
L=LI(I)
DO 40 J=1,N

C GET MASS ABSORPTION COEFFICIENTS.

CALL MAC(I,L,J,Z,ABCO)
DO 600 M=1,9
IF(EC(M,J).LE.EL(L,I)-.1) GO TO 600
IF(EC(M,J).GE.EL(L,I)+.03) GO TO 600

C PRINT WARNING IF X-RAY LINE FALLS TOO NEAR AN ABSORPTION EDGE.

ENCODE(72,2005,MESS(2),NC) IZ(I),IZ(J)
2005 FORMAT('WARNING - THE LINE OF ',I2,' IS CLOSE TO AN EDGE OF ',I2)
* NW=26
* CALL RIOS2(0,0,IPRI,$900,LUTY,IADTTY,IS,NW,MESS)
* 900 SCHEM
* IF(IS.LE.0) GO TO 8
GO TO 40
600 CONTINUE
40 AC(J,I)=ABCO
50 CONTINUE

C COMPUTE BACKSCATTER COEFFICIENTS AND STOPPING POWERS.

DO 200 I=1,N1
A1=8.73E-3*U(I)**3-.1669*U(I)**2+.9662*U(I)+.4523
B=2.703E-3*U(I)**3-5.182E-2*U(I)**2+.302*U(I)-.1836

```

```

Q=.887-3.44/U(I)+9.33/U(I)**2-6.43/U(I)**3
DO 100 J=1,N
JI=9.76*Z(J)+58.5*Z(J)**(-.19)
R(J,I)=A1-B*ALOG(Q*Z(J)+25.)
100 S(J,I)=2.*Z(J)/(A(J)*(E0+EQ(I)))*ALOG(583.*(E0+EQ(I))/JI)

```

```

C COMPUTE ABSORPTION CORRECTION.
C PRINT WARNING IF CORRECTION IS TOO LARGE.

```

```

H(I)=3.0E-6*(F0**1.65-EQ(I)**1.65)
FC(I)=1.+H(I)*AC(I,I)*COSEC+H(I)**2*(AC(I,I)*COSEC)**2*.05
RFC=1./FC(I)
IF(RFC.GT.0.75) GO TO 3
ENCODE(72,2002,MESS(2),NC) IZ(I),RFC
2002 FORMAT('WARNING - THE F(X) OF ELEMENT ',I2,' IS',F5.3)
* NW=21
* CALL RIOS2(0,0,IPRI,$4,LUTY,IADTTY,IS,NW,MESS)
* 4 SCHENT
* IF(IS.LE.0) GO TO 8
3 CONTINUE

```

```

C DETERMINE IF THERE IS A CHARACTERISTIC FLUORESCENCE CORRECTION.

```

```

L=LI(I)
GO TO (280,290,295),L
280 CO=5.
KK=0
GO TO 340
290 CO=3.5
KK=1
GO TO 340
295 CO=3.5
KK=2
340 DO 300 J=1,N
IF(J.EQ.I) GO TO 300
K=1
309 IF(EL(K,J)-EQ(I)) 330,330,310
310 IF(EL(K,J)-EQ(I)-CO) 320,320,329
320 KEY(I,J)=K+2*KK

```

```

C PRINT THE FLUORESCENCE CORRECTIONS FOUND.

```

```

ENCODE (72,2003,MESS(2),NC) IZ(I),IZ(J)
2003 FORMAT(' ELEMENT ',I2,' IS EXCITED BY ELEMENT ',I2)
* NW=19
* CALL RIOS2(0,0,IPRI,$5,LUTY,IADTTY,IS,NW,MESS)
* 5 SCHENT
* IF(IS.LE.0) GO TO 8
KS=K*K
X(J)=E0/EC(KS,J)
DO 325 M=1,N
IF(EL(K,J)-E(M)) 325,335,325
325 CONTINUE
DO 11 II=1,N

```

```

C GET ANY ADDITIONAL MASS ABSORPTION COEFFICIENTS REQUIRED.

```

```

CALL MAC(J,K,II,Z,ABCO)
FAC(II,J)=ABCO
11 CONTINUE
GO TO 330
335 DO 336 II=1,N

```

```

336 FAC(II,J)=AC(II,J)
    GO TO 330
329 IF(K.EQ.2) GO TO 330
    K=2
    GO TO 309
330 CONTINUE
300 CONTINUE

```

C SELECT PARAMETERS FOR CHARACTERISTIC FLUORESCENCE CORRECTION.

```

DO 199 J=1,N
  IF(KEY(I,J).EQ.0) GO TO 199
  P(J)=1.
  R1(J)=.88
  K=KEY(I,J)
  GO TO (410,420,410,420,410,420),K
410 OM(J)=EXP(2.373*ALOG(Z(J))-8.902)
  IF(K.EQ.1) GO TO 199
  IF(K.EQ.5) GO TO 198
  P(J)=.24
  R1(J)=.75
  GO TO 199
420 OM(J)=EXP(2.946*ALOG(Z(J))-13.94)
  IF(K.EQ.6) GO TO 198
  IF(K.EQ.2) P(J)=4.2
  IF(K.EQ.4) R1(J)=.75
  GO TO 199
198 P(J)=.02
  R1(J)=.5
199 CONTINUE
200 CONTINUE

```

C PRINT TABLE OF MASS ABSORPTION COEFFICIENTS.

```

      ENCODE(72,708,MESS(2),NC)
708 FORMAT('Z-LINE',5X,'Z-ABSORBER',5X,'MASS ABS. COEFF.')
```

*
* NW=22
* CALL RIOS2(0,0,IPRI,\$12,LUTY,IADTTY,IS,NW,MESS)

* 12 SCHENT
* IF(IS.LE.0) GO TO 8
* NW=19
* DO 500 I=1,N1
* DO 400 J=1,N
* ENCODE(72,710,MESS(2),NC) IZ(I),IZ(J),AC(J,I)

710 FORMAT(2X,I2,11X,I2,12X,F7.1)
* CALL RIOS2(0,0,IPRI,\$13,LUTY,IADTTY,IS,NW,MESS)

* 13 SCHENT
* IF(IS.LE.0) GO TO 8
* 400 CONTINUE
* 500 CONTINUE
* NW=1
* CALL RIOS2(0,0,IPRI,\$7,LUTY,IADTTY,IS,NW,CRLF)

* 7 SCHENT
* IF(IS.LE.0) GO TO 8

C PRINT HEADINGS.

```

      ENCODE(72,707,MESS(2),NC)
707 FORMAT('AT. NO.',5X,'K-VALUE',5X,'CONC',8X,'F(CHI)')
```

*
* NW=22
* CALL RIOS2(0,0,IPRI,\$6,LUTY,IADTTY,IS,NW,MESS)

* 6 SCHENT

C CLOSE PRINT FILE. THE FOLLOWING STATEMENTS APPLY TO OUR COMPUTER.

```
* 777 CALL CLOSE(LUTY,IFDB)
*      CALL ERROR (333,TAB)
      NFSW=0
```

C SAVE ALL PARAMETERS NEEDED IN QUDAT.

```
* 666 CALL SAVE(TAB)
      RETURN
8     CONTINUE
      END
```

SUBROUTINE MAC(J,L,I,Z,ABCO)

C LOAD WITH CFCAL. THIS SUBROUTINE COMPUTES A MASS ABSORPTION
C COEFFICIENT FOR ALL NECESSARY LINES AND ABSORBERS.

```
      DIMENSION EC(9,6),EL(3,6),Z(6), CN(4), D1(4),D2(4),D3(4),R(10)
      COMMON/ENER/EL,EC
      DATA D1/- .2322294,-.2544711,.2562163,1.359165/
      DATA D2/4.070053,4.769245,1.15119,-9.492116/
      DATA D3/-6.220746,-10.37878,-5.684848,18.64081/
      DATA R/1.,1.,1.17,1.63,1.,1.16,1.4,1.621,1.783,1./
      DATA CN(3),CN(4)/2.6,2.22/
      ZL=ALOG(Z(I))
      CN(1)=EXP(-.0045522*ZL*ZL-.0068535*ZL+1.070181)
      CN(2)=2.73
      IF(Z(I).LT.42.) GO TO 30
      CN(2)=EXP(-.1131595*ZL*ZL+.8368829*ZL-.5459687)
30     CONTINUE
      DO 50 M=1,10
      IF(M.EQ.10) GO TO 40
      IF(EL(L,J).LT.EC(M,I)) GO TO 50
40     MI=M-M/3-M/4-M/7
      C=EXP(D1(MI)*ZL+D2(MI)*ZL+D3(MI))/R(M)
      ABCO=C*(12.398/EL(L,J)**CN(MI))
      RETURN
50     CONTINUE
      END
```

SUBROUTINE QUDAT(NSW)

C THIS SUBROUTINE TAKES DATA FROM THE SCALERS, COMPUTES AN INTENSITY
C RATIO FOR EACH ELEMENT, AND THEN COMPUTES AND PRINTS THE
C CONCENTRATIONS OF EACH ELEMENT.
C THE OUTPUT IS PRINTED IN THE SAME MANNER AS IN CFCAL.

```
      REAL L,K
      INTEGER MESS(40),PST
*     INTEGER TAB,CRLF
*     LOGICAL SETSW
      DIMENSION Z(6),LI(6),EQ(6),AC(6,6),FAC(6,6),H(6),FC(6),R(6,6)
      1),S(6,6),KEY(6,6),P(6),R1(6),OM(6),A(6),X(6),Y(6),XK(6),C(6),FCS(6)
      2),W(6),XKF(6),BK(6),PK(6),PB(6),K(6),L(6),SF(6),ISPC(4),IFDB(3),SC
```

```

3(7),IBUF(50)
COMMON/STORE/N,N1,L1,S,R,AC,H,KEY,FAC,E0,EQ,P,OM,R1,X,Y,A,FC,Z,SC,
1SETSW,IBUF,PST,NFSW,NFLAG,L,K,BK,PK,PB,COSEC
*
COMMON/ESTOR/IDUM
EQUIVALENCE(MESS(1),CRLF)
*
DATA ISPC/2*$41503,2*$41101/
*
DATA IADTTY/$176145/
*
DATA IFDB(2)/0/
*
DATA IPRI/10/
*
DATA CRLF/$41101/
DATA SF/6*1./
DEADT(E)=E/(1.-E*3.E-6)

C READ IN COMPOUND STANDARD FACTORS AT THIS POINT, OTHERWISE THEY ARE
C ASSUMED EQUAL TO 1. (SF(6))

NI=N1+1
IF(NFSW.EQ.1) GO TO 1

C COLLECT AND STORE PEAK AND BACKGROUND DATA FOR EACH ELEMENT.

3 NFSW=1
NFLAG=0
DO 2 I=1,N1
L(I)=-1.
BK(I)=0.
PK(I)=0.
2 K(I)=-1.
1 IF(NFLAG.EQ.1) GO TO 800

C FOR STANDARDS ONLY - IF THE TOTAL COUNTS IN A SCALER IS LESS THAN
C 10000, THE MEASUREMENT IS A BACKGROUND, IF IT IS GREATER THAN 10000
C IT IS A PEAK.
C ANY SCALER CONTAINING A ZERO IS NOT COUNTED.

DO 10 I=1,N1
IF(SC(I).LT.1.) GO TO 10
IF(SC(I).GT.10000.) GO TO 5
K(I)=K(I)+1.
BK(I)=(BK(I)*K(I)+DEADT(SC(I)/SC(NI)*10.))/(K(I)+1.)
GO TO 10
5 L(I)=L(I)+1.
PK(I)=(PK(I)*L(I)+DEADT(SC(I)/SC(NI)*10.))/(L(I)+1.)
10 CONTINUE
IF(NSW.EQ.1) GO TO 40
RETURN
40 NFLAG=1

C AFTER ALL STANDARDS HAVE BEEN READ, SUBTRACT THE AVERAGE BACKGROUND
C FROM THE AVERAGE PEAK FOR EACH ELEMENT.

DO 20 I=1,N1
20 PB(I)=PK(I)-BK(I)
RETURN
800 IF(NSW.EQ.1) GO TO 15
GO TO 3

C COMPUTE THE INTENSITY RATIO FOR EACH ELEMENT.

15 DO 30 I=1,N1
30 XK(I)=(DEADT(SC(I)/SC(NI)*10.)-BK(I))/PB(I)*SF(I)

```

C IF INTENSITY RATIOS ARE TO BE ENTERED DIRECTLY, THEY SHOULD BE READ
 C HERE. TO OBTAIN INTENSITY RATIOS FROM KNOWN CONCENTRATIONS, READ
 C CONCENTRATIONS HERE.

```

    ES=0.
    DO 500 I=1,N1
      IF(XK(I).LE.0.) XK(I)=0.
500  ES=ES+XK(I)
      IF(LI(N).NE.0) GO TO 501
      C(N)=1.-ES
      ES=1.
501  DO 502 I=1,N1
502  C(I)=XK(I)/ES
  
```

C BEGIN ITERATION FOR COMPUTING CONCENTRATIONS.

C TO OBTAIN INTENSITY RATIOS FROM KNOWN CONCENTRATIONS, CHANGE
 C THE NEXT STATEMENT TO NM=3

```

    DO 550 NM=1,3
    DO 510 I=1,N1
      SS=0.
      RS=0.
      AS=0.
      DO 520 J=1,N
        SS=SS+C(J)*S(J,I)
        RS=RS+C(J)*R(J,I)
520  AS=AS+C(J)*AC(J,I)
      FCS(I)=1.+H(I)*AS*COSEC+H(I)**2*(AS*COSEC)**2*.05
      W(I)=(R(I,I)*SS)/(S(I,I)*RS)
  
```

C COMPUTE CHARACTERISTIC FLUORESCENCE CORRECTIONS.

```

    XKF(I)=0.
    DO 530 J=1,N
      IF(KEY(I,J).EQ.0) GO TO 530
      AT=0.
      DO 528 II=1,N
528  AT=AT+C(II)*FAC(II,J)
      XU=COSEC*AT/AS
      V=(3.33E5/(E0**1.65-EQ(I)**1.65))/AT
      XUV=ALOG(1.+XU)/XU+ALOG(1.+V)/V
      XKF(I)=XKF(I)+C(J)*P(J)*OM(J)/2.*R1(J)*((X(J)-1.)/(Y(I)-1.))**1.67
      1*A(I)/A(J)*FAC(I,J)/AT*XUV
530  CONTINUE
      IF(XK(I)) 511,511,510
510  EK=C(I)/W(I)*(1.+XKF(I))*FC(I)/FCS(I)
  
```

C TO OBTAIN INTENSITY RATIOS FROM KNOWN CONCENTRATIONS, INSERT THE
 C TWO STATEMENTS -

```

    C(I)=EK
    GO TO 512
  
```

C COMPUTE CONCENTRATIONS BY ITERATION METHOD ACCORDING TO CRISS.

```

    C(I)=(XK(I)*C(I)*(1.-EK))/(XK(I)*(C(I)-EK)+EK*(1.-C(I)))
    GO TO 512
511  C(I)=0.
512  CONTINUE
    ES=0.
    DO 540 I=1,N1
540  ES=ES+C(I)
      IF(LI(N).NE.0) GO TO 541
  
```

```

      C(N)=1.-ES
      ES=1.
541  IF(NM.EQ.3) GO TO 550
      DO 545 I=1,N
545  C(I)=C(I)/ES
550  CONTINUE

C   PRINT RESULTS.

*   CALL ALU(LUTY,IADTTY)
*   CALL OPEN(LUTY,IFDB)
*   IF(IFDB(2).LE.0) CALL ERROR(721,TAB)
*   NW=4
*   CALL RIOS2(0,0,IPRI,$100,LUTY,IADTTY,IS,NW,ISPC)
* 100  SCHENT
*   IF(IS.LE.0) GO TO 650
*   NW=22
      DO 600 I=1,N
      NZ=Z(I)
      IF(N1.EQ.N) GO TO 125
      IF(I.EQ.N) GO TO 130
125  RFC=1./FCS(I)
      ENCODE(72,706,MESS(2),NC) NZ,XK(I),C(I),RFC
706  FORMAT(2X,I2,8X,F6.4,5X,F6.4,7X,F6.4)
      GO TO 140
130  ENCODE(72,707,MESS(2),NC) NZ,C(I)
707  FORMAT(2X,I2,10X,'N.A. ',4X,F6.4,1X)
*   NW=16
* 140  CALL RIOS2(0,0,IPRI,$110,LUTY,IADTTY,IS,NW,MESS)
* 110  SCHENT
*   IF(IS.LE.0) GO TO 650
600  CONTINUE
      MESS(2)=CRLF
      ENCODE(72,708,MESS(3),NC) ES
708  FORMAT(13X,'TOTAL C = ',F6.4,1X)
*   NW=17
*   CALL RIOS2(0,0,IPRI,$120,LUTY,IADTTY,IS,NW,MESS)
* 120  SCHENT

C   CLOSE THE PRINT FILE AND PRINT OUT THE RESULTS.

* 650  CALL CLOSE(LUTY,IFDB)
*   IF (IFDB(2).LF.0) CALL ERROR(721,TAB)
      RETURN
      END

```

APPENDIX II

List of Symbols Used in FRAME Program

Variables in FRAME
Subroutine CFCAL

<u>Program Variable Name</u>	<u>Common Symbols</u>	<u>Remarks</u>
A(I)	A_j	Atomic weight of element I
<u>A1</u>		Intermediate step in computation of backscatter
ABCO		Mass absorption coefficient
AC(J,I)	μ_A^B	Mass absorption coefficient for line of element I in element J
AN(IZ)	A	Atomic weight of element whose atomic number is IZ
B		Intermediate step in computation of backscatter
BK(I)		Measured background of element I.
<u>C1</u> (M), C2(M), C3(M)		Coefficients for computing the absorption edge energies
COSEC	$\csc\psi$	Cosecant of the x-ray emergence angle
CO		Variable used in determining if there is a characteristic fluorescence correction
CRLF		Teletype carriage return-line feed
E(I)	$E_{q,m}$	Energy of the measured line of element I
EC(M, I)	E_q	Energy of each absorption edge of element I
EL(J, I)	$E_{q,m}$	Energy of each line of element I

<u>Program Variable Name</u>	<u>Common Symbols</u>	<u>Remarks</u>
EO	E_o	Operating kilovoltage
EQ(I)	E_q	Energy of the absorption edge of the measured line of element I
FAC(J,I)	μ_{A}^B	Absorption coefficients needed in characteristic fluorescence correction
FC(I)	$\underline{1/f(\chi)}, \underline{1/f}_p$	$1/f_p$ of the standard for the p pure element I
H(I)		Voltage dependent term in expression for f_p
IADTTY		Address of our teletype (do not use)
IBUF(50)		Input buffer
IDUM		Dummy storage location
IFDB(2) IPRI		Variables needed for our output (do not use)
IS		Error condition of output buffer (applicable to our system)
ISPC(4)		4 rubouts and 2 carriage return-line feeds on teletype
IZ(I)	Z_j	Atomic number of element I as an integer
JI	J	Mean ionization potential in Bethe's equation
K KK K5		Variables in test for characteristic fluorescence correction
KEY(J,I)		The value of this variable defines which characteristic fluorescence corrections have been found

<u>Program Variable</u> <u>Name</u>	<u>Common Symbols</u>	<u>Remarks</u>
L or LI(I)		Code defining the x-ray lines: <u>1</u> = $K\alpha$ -line, 2 = $L\alpha$ -line, 3 = $M\alpha$ -line, 0 = element not analyzed
LS		L^2
MESS(40)		Message array — used for all output
MHD(5)		Date (must be loaded from your computer's clock if you want it)
N		Number of elements in specimen
<u>N1</u>		Number of elements analyzed
NFSW		NFSW=0 for the initializa- tion of variables needed during the measurement of the standards
NW		Number of characters to be output to the output buffer R10S2
NYEAR		The year (1973)
NZ	Z	Atomic number
OM(I)	ω_q	Fluorescence yields of ele- ments which excite second- ary emission of other elements
P(I)	P(k,j)	Parameter in Reed's equation for $L\rightarrow K$ and $K\rightarrow L$ fluorescence
PB(I)		Peak intensity - background intensity for the standard for element I
IK(I)		Peak intensity for the standard for element I

<u>Program Variable Name</u>	<u>Common Symbols</u>	<u>Remarks</u>
PST		Do not use
Q		Intermediate step in computation of backscatter
R(J,I)	R	Backscatter factors for radiation I and element J
$\underline{R1}(I)$	$(\frac{r-1}{r})$	Jump ratio for characteristic fluorescence correction
RFC	$f(\chi)$	$\underline{1}/FC(I)$
S(J,I)	S	Stopping powers
SC(I)		Counts measured in scaler I
SETSW TAB		Do not use
U(I)	E_o/E_q	Overvoltage of element I
X(I)	E_o/E_q	Overvoltage needed in characteristic fluorescence correction
XL(I)		Number of peak measurements made on standard I
XXK(I)		Number of background measurements made for element I
Y(I)	E_o/E_q	Overvoltage of element I
Z(I)	Z_j	Atomic number of element I
ZL	$\ln(Z_j)$	Natural log of atomic number

Subroutine MAC

<u>Program Variable Name</u>	<u>Common Symbols</u>	<u>Remarks</u>																						
C	C_i^A	Coefficient in the expression: $\mu_B^A = C_i^A (\lambda_B)^{n_i^A}$																						
CN(4)	n_i^A	The value of n in the above expression																						
D1(4), D2(4), D3(4)		Coefficients for computing the necessary value of C.																						
MI		Number for selecting the proper coefficients for computing C																						
		<table border="1"> <thead> <tr> <th><u>M</u></th> <th><u>MI</u></th> </tr> </thead> <tbody> <tr><td>1</td><td>1</td></tr> <tr><td>2</td><td>2</td></tr> <tr><td>3</td><td>2</td></tr> <tr><td>4</td><td>2</td></tr> <tr><td>5</td><td>3</td></tr> <tr><td>6</td><td>3</td></tr> <tr><td>7</td><td>3</td></tr> <tr><td>8</td><td>3</td></tr> <tr><td>9</td><td>3</td></tr> <tr><td>10</td><td>4</td></tr> </tbody> </table>	<u>M</u>	<u>MI</u>	1	1	2	2	3	2	4	2	5	3	6	3	7	3	8	3	9	3	10	4
<u>M</u>	<u>MI</u>																							
1	1																							
2	2																							
3	2																							
4	2																							
5	3																							
6	3																							
7	3																							
8	3																							
9	3																							
10	4																							
R(10)		Energy jump for each edge																						

Subroutine QUDAT

<u>Program Variable</u> <u>Name</u>	<u>Common Symbols</u>	<u>Remarks</u>
AS	μ_A^*	Absorption coefficient of the specimen
AT	μ_B^*	Absorption coefficient needed in characteristic fluorescence correction
C(I)	C_j	Concentration of element I
EK	k_j	Computed intensity ratio for element I
ES	ΣC_j	Sum of concentrations
FCS(I)	$\frac{1}{f(\chi)}^*, \frac{1}{f_p}^*$	Absorption factor, f_p of the specimen for element I
K(I)		Number of times the background is measured for element I
L(I)		Number of times the peak is measured for element I
NFLAG		NFLAG=0 while standards are being measured and =1 while the specimen is being measured
NFSW		NFSW=1 after the first measurement is made on a standard.
NSW		Switch to change from standards (NSW=0) to the specimen (NSW=1)
RFC	$f(\chi)^*, f_p^*$	$\frac{1}{FCS(I)}$
RS	R^*	Backscatter from the specimen
SF(I)		Factors for compound standards

<u>Program Variable</u> <u>Name</u>	<u>Common Symbols</u>	<u>Remarks</u>
SS	S*	Stopping power for the specimen
V	$\left(\frac{3.33 \times 10^5}{E_o^{1.65} - E_q^{1.65}}\right) \left(\frac{1}{\mu_B^*}\right)$	V term in characteristic fluorescence correction
W(I)	RS*/SR*	Atomic number factor
XK(I)	k _i	Measured intensity ratio for element I
XKF(I)		Characteristic fluorescence factor for element I
XU	$\frac{\mu_B^*}{\mu_A} \csc\psi$	u term in characteristic fluorescence correction
XUV		$\frac{\ln(1+u)}{u} + \frac{\ln(1+v)}{v}$

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET		1. PUBLICATION OR REPORT NO. NBS TN-796	2. Gov't Accession No.	3. Recipient's Accession No.
4. TITLE AND SUBTITLE FRAME: An On-Line Correction Procedure for Quantitative Electron Probe Microanalysis			5. Publication Date October 1973	6. Performing Organization Code
			8. Performing Organization	
7. AUTHOR(S) Harvey Yakowitz, Robert L. Myklebust, and Kurt F. J. Heinrich		10. Project/Task/Work Unit No.		
9. PERFORMING ORGANIZATION NAME AND ADDRESS NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234		11. Contract/Grant No.		
		13. Type of Report & Period Covered Final		
12. Sponsoring Organization Name and Address Same as No. 9.		14. Sponsoring Agency Code		
		15. SUPPLEMENTARY NOTES		
16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.) A procedure using a mini-computer for on-line correction of x-ray data from electron microprobe analysis has been developed. This program, called FRAME, allows mass fractions to be computed on-line by the ZAF approach. Besides x-ray intensities, the only input data required are the atomic numbers of the elements present, the analytical line being used, and the operating voltage. Other required parameters such as atomic weights and x-ray mass attenuation coefficients are stored or calculated by the program. FRAME is in FORTRAN IV, and requires about 4K of computer core. Results from FRAME are very close to those of COR2, which is considered to be the most accurate program available for quantitative electron probe microanalysis.				
17. KEY WORDS (Alphabetical order, separated by semicolons) Electron probe; mini-computer; on-line correction; quantitative microanalysis.				
18. AVAILABILITY STATEMENT <input checked="" type="checkbox"/> UNLIMITED. <input type="checkbox"/> FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NTIS.		19. SECURITY CLASS (THIS REPORT) UNCLASSIFIED		21. NO. OF PAGES 51
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