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A SYSTEM OF EPMA DATA REDUCTION PROGRAMS USING BASIC FOR PERSONAL COMPUTER IN 1992

by

Shunzo Yui

(with 2 text-figures and 4 tables)

Abstract

Although computer automated or semi-automated electron microprobe analyzers have recently been common, personal programs for personal computers are useful for special treatment in EPMA data reduction. They are of course necessary for non-automated ones. Some programs are tested by sample calculations. If difference of atomic number of constituent elements is not large, the results agree well each other. Use of $AsK\beta$ and $PbL\beta$ lines instead of $AsL\alpha$ and $PbM\alpha$ gave better results for minerals containing As and Pb as main constituents. Programs originally written in BASIC for NEC-type computers were translated into GWBASIC and BASICA under MS-DOS, both for IBM-type ones.

Introduction

EPMA (electron microprobe analysis) is one of the most important methods for chemical analysis of minerals. Its basic principle is to estimate weight composition of a mineral from intensities of characteristic X-rays emitted by the elements excited with electron microbeam of about one micron in diameter. The most important and basic data in EPMA is the relative intensities to standard materials. A careful experiment using standards appropriate to the specimen is the first requisite for good result and the choice of data reduction procedure is the second or later task.

Weight ratio of an element of a specimen to standard material generally has no linear relation to X-ray intensity ratio in EPMA, therefore, the above-mentioned estimate is not easy. The most widely used method for the estimate is based on the various formulas proposed to express the behaviour of electron and X-rays in solid materials. Several physical formulas are combined to estimate weight ratio from the intensities of emitted characteristic X-rays. The numerical calculation of the combined formulas is not simple, but, due to recent development of computer, we can do it with a desk-top computer.

I published in 1986 the system of EPMA data reduction programs at the Hiro-saki University (Yui and Aoki, 1986). Then I have moved to the Hokkaido University and continue to revise some of programs although we are equipped with

computer automated EPMA (JCMA-733. JEOL). This paper presents the revision and results of test calculations.

Why personal program for personal computer in EPMA

Recently, computer automated or semi-automated electron micro-probe analyzers have been common, and they are equipped with computers of moderate size. Softwares for the computer are also provided by the EPMA manufacturer. The softwares are usually not coded by mineralogists and, therefore, we sometimes want some procedure beyond the scope of the softwares. For example, use of $K\beta$ and $L\beta$ lines are usually not considered in software of some automated microprobe but it is useful in some cases as described below, and the user is asked to modify some parts of the program. In those cases, programs written in the language familiar to EPMA users are useful.

My data reduction programs for EPMA written in BASIC (Beginner's All-purpose Symbolic Instruction Code) are useful not only for the users of non-automated EPMA but also for full-automated EPMA if the user who is less familiar with computer languages wants some procedures beyond the attached software.

Comparison of various programs.

Fundamental formulas

The most common procedure of EPMA data reduction is ZAF correction, combination of corrections for atomic number effect (Z), for absorption (A), and for fluorescence phenomena (F). Several formulas are proposed for each correction and a computer is practically essential for their numerical calculation. Combined them, many programs for computers are proposed for ZAF data reduction. The number is probably over one hundred. There should be optimum combination of a given program and a given mineral, but no one knows its general rule. Therefore, I have coded several sets of programs.

The fundamental set used in my program is Duncumb and Reed (1968) for Z , Heinrich & Yakowitz (1975) with mass absorption coefficients of Heinrich (1966) for A , and Reed (1965) with $J(A)$ values of Reed (1975) for F (Yui and Aoki, 1984). Data reduction programs involved in the computer automated EPMA of our Department is based on Philibert and Tixier (1968) for Z , Philibert (1963) for A , and Reed (1965) for F (Hirata, 1985). In order to compare the result of my program with that of the automated EPMA of our Department, I coded two programs, namely I replaced Duncumb and Reed (1968) in my 1986 program with Philibert and Tixier (1968) for Z in one program, and in another, I replaced Heinrich & Yakowitz (1975) with Philibert (1963) for A .

Oxides and oxysalts

If we can estimate the amount of oxygen, EPMA of oxide and silicate minerals

Table 1 Results of data reduction with various programs on the same cps data of kaerutite.

	Weight %			(Standards)
	(1)	(2)	(3)	
SiO ₂	39.68	39.29	39.33	(SiO ₂)
Al ₂ O ₃	14.00	13.96	13.90	(Al ₂ O ₃)
TiO ₂	4.79	4.81	4.85	(TiO ₂)
Fe ₂ O ₃	16.05	16.29	16.49	(Fe ₂ O ₃)
MgO	10.30	10.36	10.29	(MgO)
MnO	0.23	0.23	0.23	(MnO)
NiO	0.02	0.02	0.02	(NiO)
CaO	11.02	11.02	11.01	(CaSiO ₃)
Na ₂ O	2.93	2.96	2.97	(NaAlSi ₃ O ₈)
K ₂ O	1.53	1.53	1.52	(KAlSi ₃ O ₈)
Total	100.55	100.47	100.62	

Z: atomic number correction, *A*: absorption correction, *F*: fluorescence correction for characteristic X-rays.

(1) *Z*: Philibert & Tixier (1968), *A*: Philibert (1963), *F*: Reed (1965) (computer automated JCMA of JEOL).

(2) *Z*: Duncumb & Reed (1968), *A*: Heinrich & Yakowitz (1975), *F*: Reed (1965, 1975).

(3) *Z*: Philibert & Tixier (1968), *A*: H&Y, *F*: Reed (both same as (2))

Table 2 Results of data reduction with two programs on the same cps data of carbonate from Hayaseno, Aomori Prefecture.

X-ray	Stds.	Counts per second		Weight %		Weight % (normalized)		
		(1)	(2)	(3)	(4)	(5)	(6)	
MnK	MnO	1592.6	710.9	MnO	48.34	47.29	49.03	49.03
FeK	Fe ₂ O ₃	1500.2	2.7	FeO	0.17	0.17	0.17	0.17
ZuK	ZnO	1221.1	74.7	ZnO	6.94	6.84	7.04	7.09
CaK	CaSiO ₃	5269.1	591.2	CaO	5.07	4.92	5.14	5.10
MgK	MgO	7173.9	3.8	MgO	0.11	0.11	0.12	0.11
				CO ₂	37.95	37.12	38.49	38.49
Total				98.58	96.45	100.00	100.00	

Stds: standards.

(1) and (2) Cps on standards and sample, respectively.

(3) *Z*: Philibert & Tixier (1968), *A*: Heinrich & Yakowitz (1975), *F*: Reed (1965, 1975).

(4) *Z*: Duncumb & Reed (1968), *A*: Heinrich & Yakowitz (1975), *F*: Reed (1965, 1975).

(5) and (6) Normalized values to total 100% of (3) and (4), respectively. (*Z*, *A*, *F*. see Table 1).

is not so difficult because we can get good oxide or silicate standards and the amount of oxygen, the common element of sample and standards is generally larger than 30% in weight and exceeds 50% in some minerals. A cps data of kaerutite with JCMA of our Department is treated by several programs. The results are shown in Table 1. They agree well compared with accuracy of EPMA itself. On the other hand, in the case of rhodochrosite, the results do not agree in weight % but they agree quite well, if normalized to total 100%.

Sulfides

Sulfide and similar minerals generally consist of sulfur and heavier element, and we can measure intensities of the characteristic X-rays of all constituents by EPMA of sulfide minerals, but unfortunately a few good standard substance are available. In addition, some of sulfide minerals contain so heavy elements such as Pb and Bi that a general expression of the interactions between electron and mate-

Table 3 Results of data reduction with various programs on the same cps data of galena-clausthalite and altaite.

	Galena-clausthalite			Altaite			(X-rays, Standards)
	Weight %						
	(1)	(2)	(3)	(1)	(2)	(3)	
Au	0.05	0.05	0.05	0.30	0.30	0.30	(AuM, Au ₅₀ Ag ₅₀)
Pb	75.83	76.98	77.46	60.50	61.58	62.32	(PbM, PbS)
Ag	0.06	0.06	0.06	0.09	0.09	0.09	(AgL, Au ₅₀ Ag ₅₀)
Sb	0.06	0.06	0.06	0.30	0.31	0.32	(SbL, Sb ₂ S ₃)
Te	1.96	2.03	2.03	31.87	32.77	32.96	(TeL, Te)
Se	15.79	15.80	16.00	3.32	3.32	3.38	(SeK, Se)
S	4.78	4.85	4.88	0.04	0.04	0.04	(SK, PbS)
Total	98.54	99.83	100.54	96.15	98.15	99.13	
Number of atoms (Te+Se+S=1)							
Au	0.001	0.001	0.001	0.001	0.001	0.001	
Pb	1.004	1.012	1.009	0.996	0.990	0.995	
Ag	0.001	0.001	0.001	0.003	0.003	0.003	
Sb	0.001	0.001	0.002	0.008	0.009	0.009	
Te	0.043	0.045	0.044	0.852	0.856	0.854	
Se	0.554	0.550	0.552	0.143	0.139	0.141	
S	0.402	0.405	0.404	0.005	0.005	0.005	
Au-Sb	1.007	1.015	1.012	1.008	1.002	1.007	

Z: atomic number correction, *A*: absorption correction, *F*: fluorescence correction for characteristic X-rays.

(1) *Z*: Duncumb & Reed (1968), *A*: Philibert (1963), *F*: Reed (1965,1975).

(2) *Z*: D&R (same as (1)), *A*: Heinrich & Yakowitz (1975), *F*: Reed (same as (1)).

(3) *Z*: Philibert & Tixier (1968), *A*: H&Y, *F*: Reed (both same as (2)).

rials, and between X-ray and materials are not simple and they result in the difficulty of estimate of weight composition from X-ray intensity ratios. Difference in the results of data reduction due to the different set of formulas used is not so small for some sulfide and similar minerals.

Cps data on altaite and galena-clausthalite (Shiga and Urashima, 1989) are also tested with various sets of formulas as shown in Table 3.

Treatment of not measured elements.

In the Earth's crust, the most common elements is oxygen and the next is silicon, therefore, silicate and oxide minerals are common. Unfortunately for mineralogists, the wavelength of the characteristic X-ray of oxygen is larger than 15 Å, and we can not precisely measure its intensity in EPMA. We must estimate indirectly the amount of oxygen based on various assumptions, for example, stoichiometry of chemical composition of minerals.

The estimate is particularly difficult for the minerals containing elements such as iron and manganese which may be present in more than one valence state. In such a case, we must add some amount of oxygen to that assumed by data reduction formulas. If the amount is small, recalculation following EPMA data reduction is not difficult (Droop, 1987). However, if the added amount of oxygen is over 10 wt%, the matrix effect of oxygen give so serious effect to the results of calculation, therefore, the amount of oxygen should be added in normalization to total 1 (100%) at start of each iteration loop of calculation.

On the other hand, the ratio of metal atoms to oxygen require some limitation to the calculation. Some sets of my programs (Z** ADJST. HOK where ** represents DR or PT) can handle the calculation on these condition.

For hydrous minerals, we cannot measure the water content with EPMA, therefore, we must use other method to analyze it. If we cannot get enough amount of pure sample of the mineral, the direct estimate of water content of the mineral is generally impossible. In those cases, we can do the calculation based on assumption but the results are often not so precise. The amount of water should also be added at start of each iteration loop of calculation.

Overlapped peaks in EPMA

On the contrary to oxides and oxysalts, we can measure the characteristic X-ray intensities of almost all constituent elements of sulfide and alloy minerals, but we cannot discriminate some characteristic X-rays such an AsK α and PbL α emitted by the minerals containing both As and Pb. In such a case, we must use, for example, AsL α or AsK β lines for As and PbM α or PbL β lines for Pb but fluorescent excitation by AsK and PbL lines must be considered by special treatment different from the case of silicates and oxysalts.

Yui and Aoki (1986) examined which X-rays should be used on what condition

by sample calculation and concluded that if As and Pb are main constituents of the mineral, use of AsK β and PbL β lines has some advantage to AsL and PbM lines because correction factors which might magnify the error in measurement of X-ray intensities and data reduction are smaller for AsK β and PbL β lines than for AsL and PbM lines. Ozawa (in preparation) used AsK β and PbL β lines in analysis of Pb-As-S minerals to get good results.

Conclusion : Present system of data reduction programs.

As stated above, it is practical to treat data on silicates and oxysalts, and those on sulfides and alloys with different sets of programs with personal computers of relatively small size. The number of program for EPMA data reduction is probably over one hundred, and no one knows the optimum combination of programs and minerals. Therefore, I have coded four sets of programs for normal ZAF data reduction, two for oxide and oxysalts, and two for sulfides and alloys. The difference between the two is in the used formulas for the atomic number correction. The functions of those programs are shown in Text-fig. 1.

The on-line treatment of data reduction is possible with ***MES** programs if the interface between EPMA and the personal computer is available (** and **

Programs for EPMA Data Reduction(ZAF) assuming a valence state for an element

Data Selection	Data Exchange	Measurement (Cts Input)	Cps Input	Data Reduction	Wt% Input	Tabulation of Results
***DTSEL.EXC						
		***MES**.BAS				
					EPTABSUL.BAS EPTABOXD.BAS	
***CAL**.BAS ***VAC**.BAS						

Calculation of Numbers of Atoms of Two Valence States

Data Selection	Cps Input & Data Reduction	Tabulation of Results
Z**ADODT.HOK	Z**ADJST.HOK	

Work	Not Work

Text-figure 1 Functions of various programs for each step of EPMA data reduction.

*** represents ZAS (ZAF for sulfides and alloys) or ZOZ (ZAF for oxides and oxysalts).

** represents DR (Duncumb and Reed, 1968) or PT (Philibert and Tixier, 1968).

DTSEL.EXC means data selection and exchange. MES means measurement. CAL means calculation. VAC means accelerating potentials varied by element or group of elements.

represent ZAS or ZO_X and DR or PT, respectively), ZAS means Zaf Sulfide, ZO_X means Zaf OXysalts, DR means Duncumb and Reed, and PT means Philibert and Tixier. Some programs can select the necessary data for calculation form those stored as DATA statements (Table 4), and the selected ones are kept as disk file for repeated or later use.

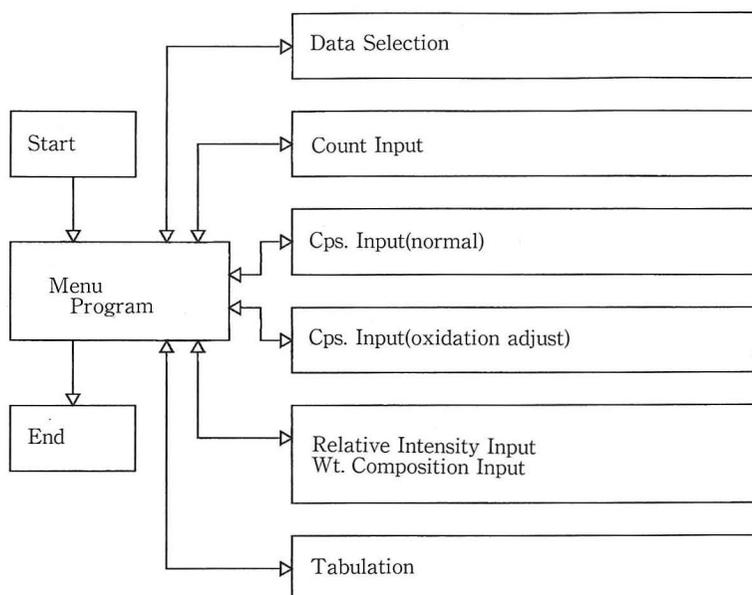
Table 4 X-rays and materials constants of which necessary for data reduction are stored in programs.

Program	X-rays and materials stored in DATA statement
ZASCAL**. BAS	SK, TiK, VK, CrK, MnK, FeK, CoK, NiK, CuK, ZnK, GaK, GeK, AsK, SeK, MoL, AgL, CdK, InL, SnL, SbL, TeL, ReL, AuL, HgL, TlL, PbL, BiL, AsL, SeL, AuM, HgM, TlM, PbM, BiM. (34 α lines)
ZASDTSEL. EXC	AsK β , PbL β (in addition to the above data)
ZOXDTSEL. BAS ZOXCAL**. BAS Z**ADODT. HOK	NaK, MgK, AlK, SiK, PK, SK, ClK, KK, CaK, TiK, VK, CrK, MnK, FeK, CoK, NiK, CuK, ZnK, AsK, SrL, ZrL, NbL, MoL, AgL, SnL, SbL, BaL, TaL, WL, PbL, (30 α lines), C, O, F, H ₂ O, OH.

** represents DR or PT.

DR: Duncumb and Reed (1968).

PT: Philibert and Tixier (1968).



Text-figure 2 System of programs for IBM-type computers.

My programs are originally written using the N88BASIC, a BASIC for the NEC-type computer under MS DOS because it is most common in Japan and the NEC company distribute a basic compiler program. Recently I translated my programs into both GWBASIC and BASICA under MS DOS for IBM-type personal computer because some Chinese scientists are interested in my programs and NEC-type computers are not common in China. They can work separately as stand-alone programs or connected with a menu program as shown in Text-fig.2 and appendix. Some of them were tested in China (at Chinese Academy of Geological Sciences and Institute of Geology, Chinese Academy of Science) with the IBM-type computer in 1991.

The programs will be sent on request accompanied with a floppy disk (5.25 inch or 3.5 inch) formatted under MS DOS. In order to adapt a given program to an available computer or to modify to a specified problem, we need a careful documentation on the program. An example of documentations on my programs is shown as appendix.

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Appendix.

An example of document on a set of programs for oxides and oxysalts (BASICA version)

Most important and basic data for EPMA are relative intensities to standard materials. You should get the most accurate relative intensities as possible. Careful experiment with the standards appropriate to the specimen is the first requisite for a good result.

EPMA data reduction is estimate of weight composition from X-ray intensities based on various assumptions (formulas) with inevitable errors. The closer composition of standards used to that of the specimen, the smaller error. A certain formulae give better results for a certain set of standards and specimen. But no one knows the optimum combination.

Programs for EPMA data reduction saved in this disk are modified ones of Yui and Aoki (1986) for oxides and oxysalts. Original ones were written in N88BASIC for NEC, some are too big for some computers, therefore, all are modified to BASICA for IBM and some are divided into several small programs which are connected with a menu program. The extensions of the program name were changed to BAA. Twenty points of measurement (nos. 1 to 5 on standard, nos. 6 to 20 on specimen) are possible, at present. Increase of points is easy if memory size of the computer allows. Background measurement on both sides of peak is possible.

The programs are EPMAMENU. BAA (MENU means menu), ZOXTSEL. EXC (ZOX means ZAF oxides; DT, data; SEL, select; EXC, exchange), ZOXCAL**. BAA (CAL means calculation), ZOXTSEL. BAA (REL means relative intensities), and ZOXTSEL. BAA. (MES means measurement) where** stands for one of DR and PT. DR means atomic number correction after Duncumb & Reed (1968). PT means Philibert & Tixier (1968). Absorption correction is after Heinrich & Yakowitz (1975) and fluorescence correction is after Reed (1965) with J (A) value after Reed (1975) in both DR and PT.

Data reduction using ZOXCAL** and ZOXTSEL** need the calculated relative intensities of standards to pure 'elements', which are calculated with ZOXTSEL** program. The above three programs work with common data file prepared with ZOXTSEL. EXC program which has necessary data for data reduction on 30 X-rays (NaK-PbL) and 5 materials (C, O, F, H₂O, OH). (See Yui and Aoki, 1986). Addition of data of other (usually less common) elements also can be handled with ZOXTSEL. EXC program. The selected data are stored on disk for later use.

Duplicated files are automatically made, one with the specified name and another with a default name. If data reduction follows, the data is read from disk by just kick RETURN key once without input of any file name. The file with the default name is renewed by every selection.

As to intensity of characteristic X-ray, ZOXTSEL** can handle counts which are input from any of the interface to EPMA (on-line), keyboard and disk which

are selected interactively (by keyboard input in answering instruction on screen). In my program, communication protocol for the on-line mode is still remain for N88BASIC and supposed as OPEN "COM: N72NN" in lines 930 and 960. Please check your interface and revise the above lines.

In the on-line mode, takeoff angle and dead time were put in program, but they can be easily replaced with any values interactively. In the case of accident or mistake, the operator can omit the wrong data. Homogeneity indices of all measured X-rays are calculated just after measurement on a point. If even one of the indices is larger than a given value (usually 3), the operator is alarmed with buzzer and asterisk on screen and he can repeat measurement.

When measurements on all points are finished, a table of cps after dead time correction is shown on the screen. If doubtful values are present, the operator can go back to partial remeasurement on the values. After check of the cps table, results of data reduction calculation are almost immediately given on screen and to printer. Number of atoms for a given basis are interactively calculated.

After the calculation, if doubtful values are found, partial measurement can be repeated on doubtful element on standard and samples, then the partially revised data are reduced to weight rations again.

ZOXCAL** programs need cps, and ZOXRREL** need relative intensities to 'element'. After data reduction, X-ray intensities are also stored on disk for repeated and/or later use. Two files are made in the same manner as the above common data. In the case of repeated re-calculation with same data, we can use those data without input of the file names.

Results of data reduction are given in the forms of element wt. ratios, their normalized ones to total 1, atomic ratios and numbers atoms for a given basis of which multiple calculations are interactively possible for various bases, and then in the forms of oxide wt. ratios, their normalized ones to total 1, and formular ratios. They are output to screen, printer, and disk where two files are prepared. In this program, weight ratios of elements for an analyzed point are printed on both screen and paper in horizontal arrangement.

For the vertical tabulation of result of ZOXMES** and ZOXCAL** programs, use EPTABOXD. BAA program. Data input is possible from any of keyboard and disk. ZOXCAL** programs can contain tabulation.

The programs for adjust of two valence states of Fe, Mn etc. consist of Z** ADODT. BAA (** stands for PT or DR) for selection of common data, and of Z** ADJST. BAA for data reduction using the selected data. ADJST means calculating mixed valence states for an elements, for example, Fe (III) and Fe (II), under the requirements of mineralogy such as ratio of metal atoms to oxygen.

Ratio of the two valence state of an element, for example, Fe (III)/TFe and basis for atom number calculation are input from keyboard interactively during the calculation, or put into the program as additional lines for the more rapid calculation. The lines for braunite, jacobsite, pumpellyite, spinel, todorokite, and hollandite are in the program in July, 1991.

If you need to revise the calculation formula or to use some options, check lines 7200-8000 and revise necessary parts. If you wish to add lines for other minerals, you must revise the following lines, 2890, 3290-92, 3680, 3870-75, 4950-95, 4735, 4810, 4835, and 7050-7110 of Z** ADJST program.

Practice

The above-mentioned programs are connected by EPMAMENU. BAA program. You should start the program and follow the below instruction shown on screen.

Programs are grouped into the following four.

- 1 Sulfides & alloys with Z after Duncumb & Reed
- 2 Sulfides & alloys with Z after Philibert & Tixier
- 3 Oxides & oxysalts with Z after Duncumb & Reed
- 4 Oxides & oxysalts with Z after Philibert & Tixier

Input (1/2/3/4)

If you input 4 from keyboard, for example, the followings appear on screen.

Oxide & oxysalts

- 1 Data selection
- 2 ZAF data reduction with count input
- 3 ZAF data reduction with cps. input from keyboard
- 4 ZAF data reduction with cps. input from disk
- 5 ZAF oxidation adjust, (cps. input from keyboard)
- 6 ZAF data reduction with rel. int. or wt. ratio input
- 7 Tabulation of results
- 8 End

Input (1/2/3/4/5/6/7/8)

If you select item 1, ZOXTSEL. BAA starts and you are asked interactively to input X-rays and materials concerned, then data necessary for data reduction using PT will be selected from data stored as DATA statements, because you have selected item 4 for the first instruction. After data is selected and saved in disk, the menu program starts again and the above second instruction appears. If you select 2, ZOXTMESP. BAA will start and the following is shown on screen.

Input file name of common data for ZAF (If same as last, RET only) or n
(new selection) or d (another drive) (File name/RET/n/d)

The data selected and saved by the above program, will be automatically transferred from disk into memory of the computer if RET (RETURN key) is kicked.

On-line mode will be selected by input 1 for the next instruction on screen as shown below if your EPMA and computer are connected with appropriate interface. In my program, communication protocol is supposed as "COM: N72NN". If your computer is not connected with EPMA, please select item 2 or 4 depending on whether your data is recorded on paper or on disk. The items 2 and 3 are used in training of how the program works with counts of sample measurement stored on disk.

- 1 X-ray measurement with EPMA and data reduction
- 2 Input of counts from disk without start switch of EPMA
- 3 Input of counts from disk with start switch of EPMA
- 4 Input of counts from keyboard

Input one of above nos. (1/2/3/4)

Sample name or standard, counting time, number of repeat, and X-rays measured with each channel will be input interactively from keyboard. After set of EPMA, X-ray intensities are measured and transferred to the computer.

After data reduction is completed, and the results are saved in disk, you will go back to the second instruction on screen of the menu program. If you wish to arrange vertically the result, select item 7 of the instruction and move to EPTABOXD. BAA program, in which data files with default names can be selected by kick of RETURN key only and transferred to the computer. After tabulation, select item 8 in the menu program and the job comes to end.

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