

# EDAX

## LABORATORIES

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Division of EDAX International, Inc.  
(Formerly Nuclear Diodes, Inc.)

4509 Creedmoor Road, Raleigh, North Carolina 27612  
Telephone (919) 787-3988

### EDIT/7.EP OPERATORS MANUAL

#### Keyboard

IN/OUT			SPECTRUM				COUNTS	QUANT
READ	TAPE	CLEAR	PEAK	WINDOW	ADD	SUBT	P/B	MANL
!	"	#	\$	%	&	'	(	)
ENTER	DISPLAY	LABELS	SMOOTH	STRIP	BLANK	BGSUB	INT	ZAF KVAL
1	2	3	4	5	6	7	8	9 0

EDIT/7.EP is an 8k program for use with a 707 series analyzer. It is intended for use with energy-dispersive x-ray spectra generated by electron bombardment, and contains specific quantitative programs appropriate to the analysis of bulk specimens such as are normally encountered in the SEM or microprobe. The programs are selected from the teletype keyboard, using the upper row of keys and the identifications shown on the keyboard nameplate reproduced above. For instance, the SMOOTH program is selected with the "4" key, and the CLEAR program with the "#!" key (capital 3).

The programs are conceptually divided into four categories: (a) transferring spectra in and out of the system; (b) operating on spectra individually; (c) reducing the spectral data to elemental counts; and (d) calculating concentration information. In typical operation, the various individual steps for dealing with a spectrum to reduce it to the desired final answer need not be selected one by one by the operator, but instead will be used as needed by the other programs. Nevertheless, the separate programs are available for use in unusual or experimental situations. Descriptions of the programs and their uses follow. The dialogue between the user and the program is shown with the user responses underlined. The symbol \* indicates that the next program selection can be made.

#### IN/OUT PROGRAMS

The EDIT/7.EP Program incorporates facilities to store up to four (4) 400 channel spectra, which are identified as memories A B C and D. Any of the programs in EDIT/7.EP can be used with any of the stored spectra. If an 800 channel spectrum is stored, it occupies two memory locations, and is identified by the first of them. For example, if a spectrum is stored in memories C and D, it would be selected as memory C. Instructions to memory D will be ignored.

Enter

\*ENTR MEM:B # ANY 16 CHAR LBL

This program enters a spectrum into the memory selected from the 707A analyzer. It also reads the energy range information from the analyzer display automatically for analyzers with serial number 322 and higher. For older analyzers, the question ER is typed and the user must answer with a digit from 1 to 9 as listed below:

ER#	energy range	eV/channel
1	0-40 keV	100
2	0-20 keV	50
3	0-40 keV	50
4	0- 8 keV	20
5	4-12 keV	20
6	8-16 keV	20
7	12-20 keV	20
8	0- 4 keV	10
9	0-16 keV	20

The user can enter any identification number or label (up to sixteen characters long), which will be kept within the spectrum and typed out with subsequent data outputs. This label is terminated with a carriage return. An 800 channel spectrum is automatically stored in the memory selected and the next one, so that the last memory (D) cannot be selected in this case. If the analyzer is storing data in a preset mode, the transfer will take place when the analysis is completed.

Display

\*DISP MEM:B PREV. ENTERED LBL

This program transfers a stored spectrum back to the 707A analyzer, types out the previously entered label, and if the analyzer is so equipped, it also resets the display to the proper energy scale. For older analyzers, the energy range number is typed out.

Labels

\*LABL  
A: PREV. ENTERED LBL  
B: ETC  
C: ETC  
D: ETC

This program types out the labels previously entered for each spectrum.

Read

\*READ MEM:C

This program reads back into memory the tapes produced by the TAPE program. The tapes should be placed in the reader with the blank leader in position, and the reader turned to start after the memory has been selected.

Tape

\*TAPE MEM:A

This program stores a spectrum on paper tape, using the teletype punch. All information including the label and energy range are included. The teletype punch should be turned on when this program is selected. The program automatically generates leader and trailer for the tape. Since the data are punched in binary code to reduce tape length, the symbols produced by the teletype printer have no meaning during this operation. A 400 channel spectrum produces a tape 90 inches long, requiring 1.5 minutes to punch and to read back in.

Clear

\*CLR MEM:C TO USE W/:A

This program clears the memory selected, for instance to use in peak generation for manual stripping operations. The memory is then set up to have the same energy scale as another selected memory, with which the artificial generated peaks are to be used.

SPECTRUM MANIPULATION

Smooth

\*SMTH MEM:B

This program smooths the statistical channel-to-channel fluctuations in the spectrum with a seven point polynomial. It is particularly useful for low count spectra, or to assist the user in recognizing small peaks. It can be used repeatedly without adversely affecting the spectrum.

Strip

\*STRP MEM:B @=CU/K KA=12345. KB=2177.

This program strips a series of peaks from the spectrum to aid in detecting or properly integrating overlapped peaks. The user is asked to specify the line he wishes to strip by element (the atomic symbol) and shell (K,L,M). The program automatically generates Gaussian peaks of the proper energy and width, and strips each away until the background level is reached. Peak ratios for  $\beta$  and  $\alpha$  lines are used. The intensity removed is typed out for the user's information. Perfectly overlapped peaks (e.g. Pb M and S KA) will not be separated by this program. It works well when the overlapped peaks are at least 100 eV apart.

Blank

\*BLNK MEM:C SUBT MEM:B NORM @ 6.4 WIDTH 11

This program subtracts one spectrum from another. The memory to be subtracted is multiplied in its intensity scale so that exact cancellation is obtained in a particular energy window. This is useful in two types of situations. One is for

removing a blank spectrum measured from a substrate (e.g. the specimen support stub or mounting medium). In this case, the spectrum background is largely independent of the specimen, and is primarily from the substrate. By setting a window at an energy where there are no characteristic peaks from elements in the specimen, the background or blank spectrum will be scaled in intensity before subtraction to reduce the counts in that window to zero. This will effectively remove the substrate contribution to the background under the characteristic elemental peaks. The memory in which the blank spectrum is stored, the energy (in keV) of the window to be set, and its width in channels must be entered by the user.

This program is also useful for peak stripping in cases where one peak is completely overlapped by a peak from another element, but there is another peak that is unobstructed. For example, if the Pb M line must be stripped to reveal a S KA line, the "blank" spectrum could be a S-free specimen containing Pb. The normalizing window would be set on the Pb LA peak, and the proper ratio of the Pb M peak would be automatically subtracted.

#### Background Subtract

\*BGSB MEM:B

This program removes the spectral background (Bremsstrahlung) leaving net peaks with a statistical confidence limit of 95%. It is called automatically by several other programs.

#### Peak

\*PEAK MEM:C HEIGHT:14793 KEV:6.398

This program generates a Gaussian peak at the energy specified with the proper width and the peak height specified and adds it to the spectrum in the memory selected.

#### Window

\*WNDW MEM:A FROM 2.04 TO 6.1 12345.

This program totals the counts in the spectrum between the two energy values specified. It can be used for integrating portions of background when using the peak-to-background model for quantitative concentration calculations. It is used by the BLANK program to integrate the range over which the two spectra are to be normalized.

#### Add

\*ADD MEM:A X 3.147 TO C

This program adds the spectrum in one memory to that in another memory, first multiplying the intensity value by the constant specified. The constant can have any value up to 100. Any channel value exceeding ( $2^{16} - 1$ ) is reduced to that value.

Subtract

\*SUBT MEM:A X 0.255 FROM D

This program subtracts the spectrum in one memory from that in another memory, first multiplying the intensity values by the constant specified. The constant can have any value up to 100. Any channel value that becomes negative is replaced by zero.

COUNT INTEGRATION

Intensity

\*INT MEM:A  
ELEM LINE  
FE KA 12345.  
ZN KB 617.  
PB L2 1074.

This program automatically totals the net counts in a window set at the proper energy and with the proper width (to the nearest whole channel) for the elements and lines specified. The line designations recognized are: KA (K-alpha), KB (K-beta), LA (L-alpha), LB (L-beta-one), L2 (L-beta-two), LG (L-gamma), and MA (M-alpha). The user can continue to enter as many elemental symbols and lines as desired. When finished, the list is ended with a carriage return instead of an elemental symbol. The totals printed out are the summation of channel-by-channel values in the window, and do not correct for peak overlaps unless prior steps were taken to strip overlapping peaks. Setting a window for an element and getting a total does not insure the element's presence; any portion of another peak lying in the window will be counted. Background subtraction is automatically performed if it has not been used already.

Peak-to-Background

\*P/B MEM:C  
ELEM LINE  
FE KA .123456E-01  
ZN KB .604317E-03  
PB L2 .216411E-02

This program is similar to the Intensity Program, except that the total net counts for each window are divided by the total number of counts in the background removed by the background-subtraction program. The values are printed in scientific notation. They can often be used with linear calibration plots constructed with standards to obtain quantitative weight fraction analysis in organic matrices.

### QUANTITATIVE ANALYSIS

#### ZAF-Automatic Concentration Data

\*ZAF MEM:A V=25.0 S=15 T=38  
ELEM LINE STD  
CU K Q=100.  
AG L  
AU M  
\*\*\*\*\*

54.875%CU  
31.474%AG  
14.103%AU

Do you have to enter P values?  
Do you have to set windows  
for each element?  
What if an unknown amount of  
Oxygen is present, how will this  
effect Q?

This program performs a ZAF-type correction for interelement effects to convert measured intensities to concentrations for up to 8 elements, using the method of Yakowitz, Myklebust and Heinrich (FRAME). The user is asked to enter the accelerating voltage V, the sample tilt S, and the x-ray takeoff angle T. He then indicates the elements by symbol, and whether the K, L, or M line is to be used. If fewer than eight elements are entered, the list is ended with a carriage return. The program will automatically collect the intensities by stripping the original spectrum, which is destroyed in the process. It is usually wise to enter the elements in approximate descending order of concentration, particularly if peak overlaps are present, since this is the order in which the peaks are stripped.

In the example shown above the user entered Q for the type of standard. This indicates quantitative analysis without standards, by assuming that the elements total approximately to some fixed amount (in this example 100%). The program then normalizes the relative intensities (K values) to this total to start the calculation. This does not force the final answers to total to that amount.

When standards are used the user can reply to STD with P (pure element) or R (ratio to pure element). An example is shown below.

ELEM LINE STD  
CU K P=12345.  
AG L R@MO/LA S=0 T=41 I=47481.  
AU M S

The P value entered by the user is the intensity on the same pure element under identical conditions of voltage and geometry. This is the typical method for microprobe analysis with flat polished samples in a non-tilting stage. An answer of R allows a different element and/or geometry to be used, and computes the effective pure element intensity for the element being analyzed. The answer of S (same) instructs the program to use the same R standard for that element as the one used above. Any combination of P and R can be used. ?  
? explore

One element can be calculated by difference by entering it as the last element, and answering LINE with N (none), as illustrated below:

ELEM	LINE	STD
FE	K	R @ CU/K S=25 T=52 P=67834.
CR	K	S
MN	K	S
C	N	

*can you do this with Oxygen*

#### Manual ZAF Calculation

*MANL	V=10.0	S=0	T=52.5
ELEM	LINE	INT	STD
NA	K	.0281	P=1
MG	K	.0593	P=1
AL	K	.0363	P=1
SI	K	.2360	P=1
CA	K	.1110	P=1
O	N	--	--
+++			

3.872%NA  
7.228%MG  
4.353%AL  
26.901%SI  
12.071%CA  
45.574%O

The Manual Program is identical to the ZAF Program except that intensities are entered from the teletype instead of being collected from the spectrum automatically. Microprobe K-values can be entered as intensities by using P=1.0 for the standard, as illustrated above, or the same P, R, S, or Q options described before can be selected.

#### Reverse ZAF Calculation

*KVAL	V=25	S=0	T=52.5
ELEM	LINE	%	
CU	K	41.1	
AU	L	59.8	
+++			

CU: 0.453  
AU: 0.529

This program computes K values from concentrations. It is especially useful for using complex standards such as minerals, since the known concentrations can be entered and used to compute K values which can then be divided into the measured intensities to get effective pure element intensities.

### ERRORS

Certain types of commands will result in the message ERROR and a return to the symbol \* indicating that the program was aborted and another one can be used. In general, this will result if an energy is specified that is not in the range of the spectrum, or if routines using two spectra (e.g. ADD) are used with spectra having different energy ranges, or if a memory is specified that is the second half of an 800 channel spectrum. The only program that can be used for the second half memory is ENTER. This means that it is possible to load a new spectrum over the second half of an 800 channel spectrum. When the user does this he should clear the first half to avoid confusion.

When entering numerical values, the use of any key other than a digit 0-9 or decimal point (or E if using scientific notation) will cause a ? to be typed. The value must then be re-entered. If a value is entered in error, a rubout can be used to delete it. All numerical values are terminated with the carriage return key, and can have any number of digits before or after the decimal point. Integer values do not require the decimal point.

### LOADING THE PROGRAM - PAPER TAPE

The so-called "bootstrap loader" that lets the computer read in the more elaborate loader program is normally present in the computer memory. If it has been inadvertently erased, it can be entered manually with the data switches, by setting the data switches to each of the values listed below (zero = down, 1 = up) and pressing the control switch indicated.

<u>Set switches to</u>	<u>(Octal)</u>	<u>and press</u>
0 001 111 111 101 111	017757	examine
1 010 110 100 100 000	126440	deposit
0 110 011 110 001 000	063610	deposit next
0 000 000 111 111 111	000777	deposit next
0 110 000 101 001 000	060510	deposit next
1 010 111 001 000 000	127100	deposit next
1 010 111 001 000 000	127100	deposit next
1 000 111 000 000 011	107003	deposit next
0 000 000 111 111 010	000772	deposit next
0 000 001 100 000 000	001400	deposit next
0 110 000 001 001 000	060110	deposit next
0 000 100 111 110 110	004766	deposit next
0 100 100 100 000 010	044402	deposit next
0 000 100 111 110 100	004764	deposit next

After this is done, set the switches to 0/001/111/111/111/000 (octal 017770), place the program tape in the reader, turn the reader control to start, press the START button on the console. This will read in the first segment of tape, which is a more sophisticated loader used to load the remaining tape.

When the tape stops, set the switches to 0/001/111/111/111/111 (octal 017777) and again press START. The large portion of the tape will now read in. Then

the message

707 ANALYZER SERIAL NUMBER?

will be typed out, and the user should respond with the serial number from the 707 being used. Next the user is asked for the resolution of his spectrometer

SPECTROMETER RES. @ MN KA IN EV?

This number can be taken from the test sheets received with the system.

Finally, set the data switches to 0/000/000/000/011 (octal 000003), and lock the computer panel. The computer can now be controlled by the STOP/START buttons on the console. The \* indicates that the program is ready.

LOADING FROM CASSETTE

Place the tape cassette into the cassette drive and press REWIND. STOP the computer and enter the following instructions from the data switches:

<u>Set switches to</u>	<u>(Octal)</u>	<u>and press</u>
0 000 000 011 111 110	000376	examine
0 110 000 001 011 100	060134	deposit
0 000 000 011 111 111	000377	deposit next
1 000 000 011 111 110	100376	reset, start

The system will respond by reading in a loader program from the cassette, which will ask # meaning the number of the particular program desired on the cassette (which can hold several programs). If, for example, the program EDIT/7.EP is identified on the cassette label as #3, the user should respond with 3 and carriage return. After the program has read in from the cassette the computer will halt. The user should then set the data switches to 0/000/000/011/111/111 (000377) and press START. The questions and procedure described above will then be followed. The tape cassette should be rewound before removal.

