

This document was produced
by scanning the original publication.

Ce document est le produit d'une
numérisation par balayage
de la publication originale.

CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 66-93

THE USE OF ANALYZING CRYSTALS IN X-RAY SPECTROGRAPHY

by

DOROTHY J. REED

MINERAL SCIENCES DIVISION

COPY NO. 15

DECEMBER 9, 1966

Mines Branch Investigation Report IR 66-93

THE USE OF ANALYZING CRYSTALS IN X-RAY SPECTROGRAPHY

by

Dorothy J. Reed*

- - -

SUMMARY OF RESULTS

The properties of quartz, LiF and EDDT crystals as dispersing elements for X-rays have been compared. The ratios of the counts obtained from these crystals has been found to vary with the intensity as well as the energy of the radiation being measured.

A question has been posed as to the effect of crystal reflectivity on interelement correction factors.

* Research Scientist, Analytical Chemistry Subdivision, Mineral Sciences Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

INTRODUCTION

In certain analyses by X-ray spectrography, determinations have been made using two different crystals to test the effect of greater separation of adjacent radiation on the results obtained. Examples of such analyses are the determination of zirconium in steels having uranium as another component and of tin in ore fractions containing antimony.

The ratios of the counts from the two crystals were calculated for interest and proved to be variable for a single characteristic radiation. The ratios were expected to vary with the energy of the radiation measured because the crystals would be penetrated to different depths. but variation with intensity was unexpected.

As time has permitted the ratios between the intensity of the characteristic radiation of a number of elements as reflected from three crystals have been determined to try to account for the variation.

CRYSTAL FUNCTION

Most X-ray spectrographic analysis is accomplished by the spatial separation of the X-rays arising from a sample into their component energies. X-rays of spectrographic interest have such short wavelengths, 0.1-10A, that it is impossible to rule gratings which can diffract them for such dispersive analysis. However, many crystal lattices are of a size that makes them suitable as dispersing agents for these radiations. The selection of such crystals is influenced by their availability, perfection, stability and reflectivity, as well as their lattice spacing.

X-rays are diffracted according to the Bragg equation: $n\lambda = 2d \sin \theta$, where n is an integer, λ the wavelength of the X-rays, d the crystal spacing and θ the angle of incidence of the rays. The angle of reflection equals the angle of incidence so characteristic radiation of an element emerges from a crystal at double the angle that satisfied the Bragg equation and is identified by this 2θ value.

The sine of an angle cannot exceed unity, so crystals can disperse only those X-rays whose wavelength does not exceed $2d$. The longest wavelength that may be diffracted is determined by the crystal lattice and this, in turn, determines the lightest element for which a crystal may be

used because the wavelengths of the characteristic radiations of the elements vary inversely with their atomic number. This variation also means that the angle at which the characteristic radiation appears and the angular separation of the radiation of adjacent elements decrease with increasing atomic number.

The highest atomic number for which a crystal may be used is limited by the geometry of the spectrograph. In the case of Philips 100kV equipment, below a goniometer setting of $6^\circ 20'$ the detector receives most of the radiation from the sample directly. The resulting high background makes identification of characteristic radiation difficult. The upper 2θ limit of 145° is a mechanical stoppage.

CRYSTALS INVESTIGATED

LiF is the most widely used crystal because of its stability, high reflectivity and suitable $2d$ spacing - 4.027 Å. It may be used for the K lines of the elements from K to W and the L lines of elements from Sn to U. The K lines of the rare earths and heavier elements are not well separated by this crystal and it is better to use the $n = 2$ radiation, if the intensity makes it possible.

For better separation of heavy element radiation the spectrograph is supplied with a quartz crystal: $2d = 2.749$ Å. Ti is the lightest element whose K lines may be detected by this crystal. The heaviest element with our equipment is Bi. This is a kV limitation. L lines of the elements from Ce to U are within its range.

For light elements EDDT (ethylene diamine dextrotartrate) is used in our spectrograph. It has a $2d$ spacing of 8.808 Å and is useful for K lines of elements from Al to Sb and L lines of those from Br to U.

There is a wide range of elements for which all three crystals may be used as far as 2θ values are concerned. For the 100kV spectrograph this is from V to Sb for $K\alpha$ lines and from Ce to U for L. The crystals have been compared using a number of elements in the $K\alpha$ range. LiF has been used as the standard of comparison for the other two because of its extensive use. All radiation has been detected with a scintillation counter.

The choice of the above crystals for the 100kV spectrograph was influenced by early work with 60kV equipment using NaCl and topaz crystals. NaCl approaches LiF in reflectivity and its $2d$ spacing of 5.641 Å makes it almost as useful. However, it is hygroscopic and in humid atmospheres its reflectivity was found to decrease rapidly. Topaz has a slightly better

lattice spacing ($2d = 2.712 \text{ \AA}$) than quartz for short X-rays, but at 60kV "ghosts" appear in the spectra of Sn and neighbouring elements reflected by it.

RELATIVE INTENSITY

The relative intensity of X-rays of varying energy reflected by the three crystals is shown in Figure 1 which presents scans of the continuum from a tungsten tube, operated at 60 kV and 20 mA, reflected from a filter paper sample. Both energy and wavelength are shown on the ordinate. The positions of certain characteristic radiations are also indicated. The intensity of the LiF scan has been halved to bring it into scale with that of the other crystals. The intensity of the high energy portion of the continuum reflected from LiF is reduced when the atomic number of the sample is increased. This is shown by the line for a Ti sample in the figure.

The Compton scatter which appears on the high wavelength side of the peaks in the LiF and quartz scans is not separated from these peaks by EDDT indicating the poorer resolution of the crystal.

Figure 2 presents the counts obtained from five metals and $\text{Sr}(\text{NO}_3)_2$ at varying potentials using the three crystals. The energy of the radiation measured is given for each sample. LiF gave the highest counts in every case. EDDT reflected more of the radiation with energies less than 10 keV than did quartz. Counts from these two crystals were similar intensity for radiation with energies from 10 to 13 keV. For 14 keV and greater energies quartz was preferable to EDDT using intensity as the criterion.

With different dispersion from the crystals it seemed possible that the range of energies reflected might differ and account partially for the crystal differences. To check the energy dispersion, pulse amplitude distribution curves were run for the K radiation of three metals using each crystal. The results in Table 1 indicate no significant difference in the range of energies reflected. Pulse amplitude, A, and width at half-height, W have been used to characterize the radiation. The curves were determined using an amplifier gain of 10 and a counter voltage of 900.

LINEARITY OF COUNTS

The response of the K α radiation of two metals to varying tube potential is shown in greater detail in Figure 3. This response plays an important part in determining the count ratios from different crystals at low and high counting rates. It is affected by such factors as critical potential, over voltage response and fluorescence yield, which determine the emitted intensity of a particular characteristic radiation. The crystal reflectivity, combined with collimation and path length, determines what portion of this radiation reaches the detector.

The intensity of the radiation in Figure 3, as represented by the counting rate, shows a slightly sigmoid response to potentials above the critical. First order regression lines, calculated from the central points for each curve, have been drawn in. The counting rate is linear from a short distance above the critical potential to a point where either the response of the radiation is no longer proportional to the over voltage or the rate is great enough to suffer electronic loss in the counting equipment. The Co counts lose linearity at 30 kV or about 10^5 cps using the LiF crystal. That this is a detector limitation is shown by the fact that the counts from the other crystals are still linear at 36 kV at lower intensities. The Cr counts all show deviation from linearity at 38 kV, though it is not obvious in the case of the quartz crystal. This is non-linear overvoltage response. The intensity from the LiF crystal is only 3×10^4 cps. Crystal ratios can be affected when counts lose linearity, whatever the cause.

INTENSITY RATIOS

Ratios were calculated for the characteristic radiation from a number of the elements, or their simple compounds, for which all the crystals could be used. A range of counting rates was obtained by varying the kV, mA or percentage of the element. The following ratios were determined: LiF/quartz, LiF/EDDT and EDDT/quartz.

Figures 4 and 5 present LiF/quartz and LiF/EDDT ratios for FeK α and MoK α radiations plotted against the LiF counting rate. In each case the ratio varies with the rate, though the range of LiF/EDDT for Fe is small. In general, MoO₃ and MoS₂ give lower LiF/quartz values than do the metallic samples. For LiF/EDDT, MoO₃ gives lower and MoS₂ higher results than the metal.

Effect of kV

In Figure 4 the points from any one sample indicate a direct response but do not lie on a straight line. There are two major sources of error responsible for this deviation: counting error and error in reproducing the geonimeter settings. By setting the kV and taking counts with the three crystals before changing the setting, the use of the LiF counts as reference reduced the inaccuracy in the kV settings to that due to fluctuations in line potential. In the case of some ratio responses to counting rate, the variation was no greater than the error introduced by the counting error; but insignificant errors in counts can result in significant ratio differences as Table 6, primarily included to typify responses to composition, illustrates.

To minimize the effect of counting error, regression lines for count response to kV were established for each radiation for all three crystals and the resulting counting rate for each kV calculated from the equation, i.e. from the linear response best fitted to the experimental results. The ratios fluctuation was thus smoothed, but the total error was enhanced by that of the kV settings. In Table 2 ratios for CrK α are presented as examples of ratios obtained from experimental counts and from equations calculated from the counts. The regression lines were calculated using only the central points of each line and the equation was used to determine where the sigmoid portions began by comparing calculated and experimental counts and allowing $1 S_e$ difference where S_e was the standard error of prediction calculated for each equation. The calculated ratios in parenthesis in Table 2 are from counts beyond the linear range for one or more crystals. There is no significant difference between calculated and experimental ratios. The calculated LiF/quartz and LiF/EDDT values decrease constantly with increasing kV, while the experimental ratios decrease variably. The EDDT/quartz ratio increases with potential.

All LiF/quartz and LiF/EDDT ratios were found to decrease with increasing counting rates when this increase was effected by increasing the tube potential. The EDDT/quartz values increased or decreased only slightly with kV and in many cases were almost constant, showing variation only in the third decimal place.

Effect of mA

When the count rate was increased by increasing the current to the X-ray tube, the ratios were again found to vary. Results for MoK α from MoS₂ in Table 3 show a decrease in LiF/quartz and especially in LiF/EDDT values as the mA is increased, while EDDT/quartz values are almost constant. There is little difference between the calculated and experimental

ratios. In this instance the response to mA was not linear for any crystal and second degree equations were used.

In the 100kV spectrograph the current is voltage controlled. Table 3 could not be extended beyond 25 mA at the kV used for Mo excitation. Using pure samples to determine the ratios in this investigation, the counting rate exceeded 10^5 cps at relatively low potentials. Therefore a current of 10mA was used in most cases, 20mA in a few instances.

Combined kV and mA effects are shown in Table 4 using NiK α . All ratios decrease with increasing kV. The decrease is much more pronounced at the higher mA. For LiF/quartz and LiF/EDDT the 5mA ratios are lower than the 10mA ones up to 24kV, then the reverse is true. All 5mA EDDT/quartz values are higher and the variation is greater.

Effect of Concentration

The effect on the ratios of varying the counting rate by changing the concentration of the element is shown for three steel components in Table 5. For two components LiF/quartz varies inversely with the amount present, for the other it is constant. For LiF/EDDT the variation is direct for two and again constant for Ni. All EDDT/quartz values vary inversely with the concentration, though for Ni the variation is small.

Table 6 contains the LiF/quartz ratios determined on two days for Zr in steels, which are directly proportional to the amount present. While the counting ratios duplicate satisfactorily for the two days, the minor differences cause significant ones in the ratios for the higher concentrations. For Ge in steels LiF/quartz also varied directly with the concentration. These Ge and Zr results are early ones and instigated the comparison of crystals.

Sn in ores was also determined using two crystals by the 2% SiO₂ dilution technique (1) prior to this investigation. Table 7 presents the results of varying concentration and of background. The ratio varies inversely with the amount of Sn present and the background has no effect except on the lowest concentration where the greatest effect would be expected.

Effect of Background

The effect of background is also shown in Table 8 for AgK α radiation. Ag was chosen to check this factor because, of the elements used in this investigation, it is subject to the highest tube continuum and the background

effect should be greatest. It is improbable that EDDT would ever be used to determine this metal, but all crystals were used for all the elements selected to make the survey complete. The counting rate was varied by changing potential, not changing concentration as with the Sn results above. LiF/quartz and EDDT/quartz ratios confirm the Sn results that the background has no significant effect, but for LiF/EDDT the net counts give a higher ratio - the background has a much greater effect on the low EDDT counting rate than on the high LiF rate.

Effect of Atomic Number

To determine how the ratios varied with atomic number, a standard basis for comparison was necessary because the ratios were subject to the above variables.

The ratios were first related to the LiF counting rate. The potentials required to produce 5×10^4 cps using this crystal were calculated from the equations for each element and substituted in the equation for the other crystals to give their corresponding counting rates. For the lighter elements the calculated potential was greater than that of the upper limit for linearity of the regression lines. The basis for comparison was reduced to 3×10^4 cps for the LiF crystal. This resulted in the potential for the heavier elements being less than that required for a linear response from the other crystals.

To achieve comparable secondary X-ray production from the elements, a potential derived from the product of two factors was used. The factors were calculated from the fluorescence yield, ω , and the critical potential, CP, for each element. The factor for ω was calculated to give all elements a relative ω_k of 0.400. The yields of the elements used (2,3), given in Table 9, have a mean of 0.445. The ω factor was multiplied by 2.5 CP (4) to give the final kV value in Table 9. The CP multiplier was chosen to keep all final potentials within the linear response range of the elements for all three crystals. The counting rates were calculated for each crystal using this value and the ratios derived from them.

Figure 6 presents the crystal ratios plotted against the energy (5) of the characteristic radiations using as bases 5×10^4 and 3×10^4 cps for LiF, where applicable, and the comparable production factor. Where more than one compound was used for an element, the mean ratio has been plotted. The energy of the radiation, as listed in Table 9, rather than the atomic number of the element has been used to make it possible to include ratios for WLa_1 and $PbL\beta_1 + 2$. There is good agreement for LiF/EDDT and EDDT/quartz regardless of the basis used for comparison. For LiF/quartz the ratios based on 3×10^4 cps were in general higher than those

calculated to the other bases.

The LiF/EDDT ratios increase with increasing keV. The other ratios increase rapidly up to 6 or 7 keV then decrease. Neither quartz nor EDDT have as good reflectivity as LiF for any element considered. LiF/quartz is least for the heavy elements where quartz is used to separate overlapping peaks. LiF/EDDT is least for the light elements where EDDT is likely to be employed.

CRYSTAL STRUCTURE

During the course of these investigations Ag_2SO_4 was scanned using the three crystals to show the separation of the α and β radiation of Ag - the heaviest element used for crystal comparison. The scans are reproduced in Figure 7. In addition to showing poor separation of $\text{AgK}\beta$ at $6.47^\circ 2\theta$ from $\text{AgK}\alpha$ at 7.31° , EDDT gave double peaks. The scans of a selection of lighter elements made with this crystal are shown in Figure 8. The separation of the α and β peaks is complete at Zn. For all elements in the figure both peaks show complex structure.

These scans recalled the topaz "ghosts" found for elements from Cd to Te in much earlier work with the 60 kV equipment, though there was little resemblance in the complexity of the scans. These "ghosts" as represented by those for Cd in Figure 9, were well separated from the characteristic peaks and small in comparison with them, but they could be mistaken for radiation from traces of neighbouring elements or the rare earths as indicated in the figure. The ghosts were assumed to arise when radiation of sufficient energy to penetrate the complex rhombic topaz crystal to a depth that enabled it to be diffracted from crystal planes other than those parallel to the surface was so diffracted. This assumption was based on the fact that the positions of the ghosts relative to the characteristic peaks remained constant from element to element.

The complex structure in the EDDT scans is not separate from the characteristic peak and might be regarded as evidence of twinning, except that it is not found in a scan of Ti - Figure 10. The multiple peaks are probably due to the complexity of the tartaric monoclinic crystal. They could arise from deep within the crystal where crystal planes not parallel to the surface could diffract the deeper penetrating, more energetic radiation of the heavier elements. They do not occur with the elements for which the crystal is commonly used, i.e. those lighter than Ti. The use of simple cubic crystals, whenever possible, is recommended.

DISCUSSION

In this investigation the crystals have been compared beyond the range of elements for which they would normally be used. This was necessary to give a good comparison range. It is unlikely that EDDT would be used for elements heavier than Fe or quartz for those lighter; though in specific analyses, such as the use of quartz for high temperature alloys, these limits have been exceeded.

The ratios determined in this investigation apply only to the three crystals used. They may be used as a general guide for crystal comparison, but are not absolute values. Reflectivity varies among individual crystals of the same kind and in some instances may be improved by polishing - as may resolution (6).

Detector response varies with keV (7) but it should be constant for any specific energy and the ratios for any characteristic radiation should not be affected by it.

The change in ratios with keV, or Z, was expected. It was assumed that X-rays of different energies would penetrate each crystal to different depths and hence absorption and in-phase reinforcement would vary. Considering this response, the crystals must give a different individual response or the ratio would not change. In Figure 6 the curves with quartz as the ratio denominator are similar. Therefore it could be assumed that the response of LiF and EDDT are similar or change in a manner toward quartz that is complementary; but LiF and EDDT do not give the same response as their ratios show. A mathematical analysis of the six possible ratios might give an answer, but such an answer would apply only to our crystals. With other EDDT and quartz crystals, the positions of the Se and Pb lines for these crystals in Figure 2 could easily be reversed.

The variation in the ratios with the counting rate was not anticipated. For pure metals and simple compounds LiF/quartz increased or decreased, only slightly in many cases, with no relationship to any apparent variable. Because EDDT/quartz is almost constant for many energies, it might be argued that the variation in the other ratios is due to LiF response. For complex samples the ratios in some instances varied directly, not inversely, with the counting rate when the changing rate was due to per cent composition. This was the case for LiF/EDDT for Cr and Mo radiation from steels, Table 5. and for LiF/quartz for Zr in steels, Table 6; but not for LiF/quartz for Cr and Mo in steels. If such ratio response inversion is due solely to interelement effects, should it not be similar for all crystals?

Crystal response appears to be a relatively unimportant variable compared with others such as tube operating conditions and counter voltage. It seemed sufficient at first to establish only that it varied with the counting rate, regardless of the means used to change the rate, and that the variation could not be predicted readily. However, on further consideration the importance of crystal response increased. It is obvious from Figure 6, where the effect of counting rate has been minimized, that the response of one or more crystals must change with keV or the ratios would remain constant. Such variation with the energy of X-radiation could affect the evaluation of interelement effects and explain why Traill and Lachance find experimental α 's not in agreement with those calculated (8) and why Lucas-Tooth and Price's k 's (9) sometimes show negative absorption when positive would be expected.

In these mathematical corrections the assumption has been made that the radiation leaving the sample reaches the detector unchanged. Only interelement enhancement and absorption have been considered in the calculations; no other factors have been introduced. If the crystal reflectivity changes with keV, then it also affects the intensity of the radiation reaching the detector and hence the interelement effect interpretation. Some interelement effects may have been falsely interpreted for this reason. Crystal effect would explain why corrections have been found satisfactory for small ranges of sample variation when appropriate standards are used, but not for wider ranges, and why standards must cover the sample range.

If crystal reflectivity is a variable affected by both the intensity and the energy of X-radiation, a crystal parameter should be added to Birks' fundamental approach (10) to the calculation of the composition of a sample.

It is hoped to be able to prove or disprove the variability of crystal reflectivity in the near future. While the exact measurement of radiation entering and leaving a crystal is beyond the scope of our present equipment, proof that it changes with energy and intensity may be possible.

CONCLUSIONS

Crystal ratios for characteristic radiation vary with the energy of the radiation or the atomic number of the element.

Crystal ratios vary with the counting rate whether it is varied by kV or mA to the X-ray tube or by the per cent of the radiating element in the sample.

For pure elements or simple compounds LiF/quartz and LiF/EDDT vary inversely as the counting rate, while EDDT/quartz may vary directly or indirectly.

In complex systems the ratios may vary directly or indirectly with the counting rate.

Simple cubic crystals should be used as dispersing agents for crystals whenever possible.

REFERENCES

1. D. J. Reed and W. R. Inman, Fifth Ottawa Symposium on Applied Spectroscopy, September, 1958.
2. J. Laberrie-Frolow and P. Radvanji, J. Phys. and Radium, 17, 144, (1956); (Green and Cosslette, Proc. Phys. Soc. 78, 1206 1961).
3. J. Sherman, Spectrochim Acta 7, 283, 1955 - Arend's formula.
4. H. A. Leibhafsky, H. G. Pfeiffer, E. H. Winslow and P. D. Zeman, "X-ray Absorption and Emission in the Analytical Chemistry", p.307, John Wiley and Sons, New York, (1960).
5. A.S.T.M. Data Series, "X-ray Emission Line Wavelengths and Two-Theta Tables", D.S. 37 (1965).
6. W.D. Fisher, Fifth National Meeting, Society for Applied Spectroscopy, Chicago, June 1966.
7. J. Taylor and W. Parrish, Rev. Sci. Instr. 26, 367 (1955).
8. R. J. Traill and G. R. Lachance, Can. Spectry. 11, 63 (1966).
9. H. J. Lucas-Tooth and B. J. Price, Metallurgia, 64, 149 (1961).
10. L. S. Birks, Fifth National Meeting, Society for Applied Spectroscopy, Chicago, June 1966.

TABLE 1

Characteristics of Radiation Reflected from Three Crystals

	Cr K α		Ni K α		Mo K α	
	A	W	A	W	A	W
LiF	10.3	7.2	15	8.4	36.9	13
Quartz	10.2	7.0	15	8.4	37.2	13
EDDT	10.3	7.2	15	8.4		

TABLE 2

Experimental and Calculated Crystal Ratios for Cr K α Radiation
from Cr Powder

kV	Experimental			Calculated		
	LiF/quartz	LiF/EDDT	EDDT/quartz	LiF/quartz	LiF/EDDT	EDDT/quartz
12	6.87	2.77	2.48	(14.00)	(12.88)	(1.09)
14	8.35	3.17	2.99	8.49	3.87	2.19
16	7.45	2.79	2.33	7.60	3.21	2.37
20	6.95	2.76	2.52	7.03	2.84	2.48
24	7.24	2.72	2.67	6.82	2.70	2.52
28	6.68	2.64	2.52	6.71	2.64	2.54
30	6.62	2.60	2.54	6.67	2.61	2.55
34	6.59	2.55	2.58	6.61	2.58	2.56
38	6.71	2.67	2.51	6.58	2.56	2.57
42	6.67	2.59	2.58	6.55	2.55	2.57
46	6.70	2.53	2.56	(6.53)	(2.53)	(2.58)
50	6.55	2.59	2.52	(6.51)	(2.52)	(2.58)

TABLE 3

Ratios for MoK α Radiation from MoS $_2$ with Varying Current

mA	Experimental			Calculated		
	LiF/quartz	LiF/EDDT	EDDT/quartz	LiF/quartz	LiF/EDDT	EDDT/quartz
5	7.51	17.39	0.432	7.61	17.54	0.434
10	7.03	16.07	0.438	6.95	15.98	0.435
15	6.58	15.19	0.433	6.59	15.11	0.436
20	6.26	14.29	0.439	6.30	14.40	0.437
25	6.04	13.77	0.432	6.03	13.74	0.439

TABLE 4

Calculated Ratios for NiK α Radiation - Varying kV and mA

kV	10 mA			5 mA		
	LiF/quartz	LiF/EDDT	EDDT/quartz	LiF/quartz	LiF/EDDT	EDDT/quartz
16	13.93	5.47	2.545	11.12	4.26	2.608
18	12.06	4.74	2.543	10.74	4.14	2.597
20	11.23	4.42	2.542	10.55	4.07	2.591
24	10.46	4.11	2.542	10.37	4.01	2.586
28	10.09	3.97	2.542	10.28	3.98	2.584
32	9.88	3.89	2.541	10.22	3.96	2.582
36	(9.74)	(3.83)	2.541	10.19	3.95	2.581
40	(9.65)	(3.80)	2.541	(10.16)	(3.94)	2.580

TABLE 5

Ratios from Steel Samples for K α Radiation of Components

%	Experimental			Calculated		
	LiF/quartz	LiF/EDDT	EDDT/quartz	LiF/quartz	LiF/EDDT	EDDT/quartz
<u>Cr</u>						
2.12	7.68	2.25	3.41	7.87	2.25	3.50
4.66	7.94	2.37	3.34	7.73	2.39	3.24
6.02	7.50	2.43	3.08	7.69	2.41	3.19
16.56	7.86	2.48	3.16	7.59	2.48	3.06
16.68	7.46	2.48	3.01	7.58	2.48	3.05
<u>Ni</u>						
7.12	10.50	3.61	2.71	10.36	3.70	2.799
10.07	10.25	3.79	2.70	10.36	3.70	2.796
13.37	10.21	3.71	2.75	10.36	3.70	2.795
14.36	10.50	3.75	2.80	10.36	3.70	2.795
15.48	10.37	3.65	2.84	10.36	3.70	2.794
<u>Mo</u>						
0.84	8.86	7.38	1.20	9.25	7.00	1.18
1.50	8.73	9.14	0.96	8.86	8.98	0.99
2.80	8.69	10.01	0.87	8.60	10.02	0.86
4.61	8.67	10.87	0.80	8.50	10.63	0.80
8.26	8.36	11.03	0.76	8.41	11.11	0.76

TABLE 6

Ratios for ZrK α Radiation from Steels: 2 days, total counts

% Zr	LiF cps	Quartz cps	Ratio
0.015	1452	258	5.62
	1452	261	5.56
0.033	1664	288	5.78
	1656	272	6.09
0.071	2195	299	7.34
	2187	291	7.51

TABLE 7

Ratios for SnK α Radiation from SiO₂ Dilution Standards

% Sn (in 2% dilution)	LiF/quartz	
	Total cps	Net cps
78.76	2.56	2.54
39.38	3.58	3.60
7.88	4.19	4.22
1.58	4.79	5.39

TABLE 8

Effect of Background on AgK α Experimental Ratios

kV	LiF/quartz		LiF/EDDT		EDDT/quartz	
	total	net	total	net	total	net
28	7.28	7.25	19.91	20.97	0.36	0.35
30	7.02	7.02	20.58	21.38	0.34	0.33
32	6.75	6.75	19.04	19.54	0.36	0.35
34	6.54	6.55	18.94	19.37	0.34	0.34
36	6.30	6.31	17.57	17.90	0.36	0.35

TABLE 9

Factors for Equivalent Secondary X-ray Production

Element	Z	keV	CP	ω_k	factor $\omega_k = 0.400$	2.5 CP x factor
V	23	4.95	5.462	0.216	1.8518	25.29
Cr	24	5.41	5.987	0.245	1.6326	24.44
Mn	25	5.90	6.535	0.278	1.4388	23.51
Fe	26	6.40	7.109	0.308	1.2987	23.08
Co	27	6.93	7.707	0.340	1.1765	22.67
Ni	28	7.48	8.329	0.374	1.0695	22.27
Cu	29	8.05	8.978	0.405	0.9876	22.16
Zn	30	8.62	9.657	0.436	0.9174	22.15
Se	34	11.20	12.649	0.556	0.7194	22.75
Sr	38	14.16	16.101	0.654	0.6116	24.62
Mo	42	17.47	19.996	0.730	0.5479	27.39
Ag	47	22.16	25.509	0.799	0.5006	31.92

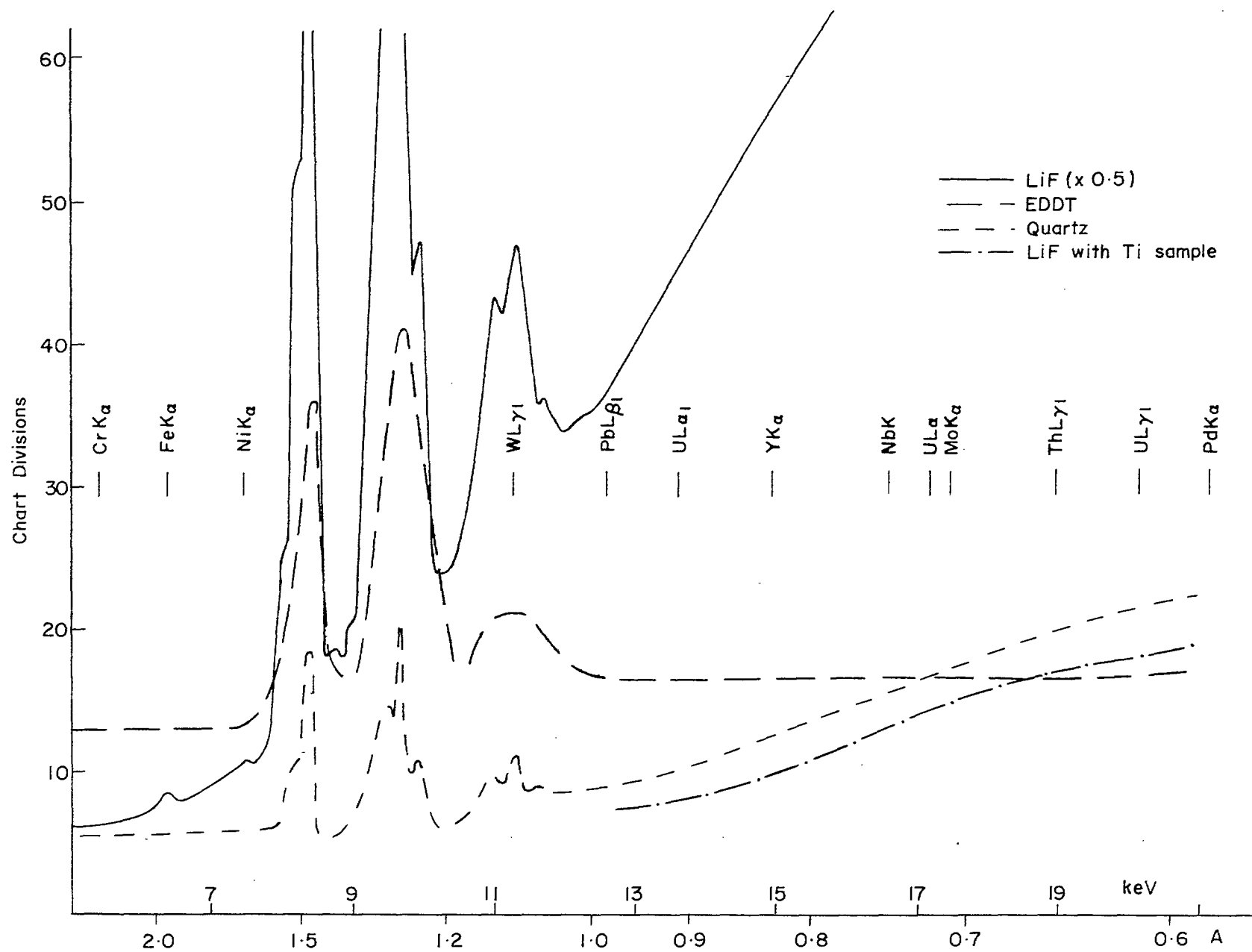
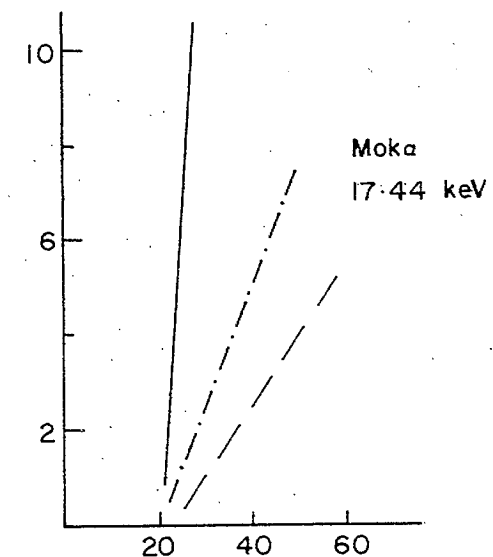
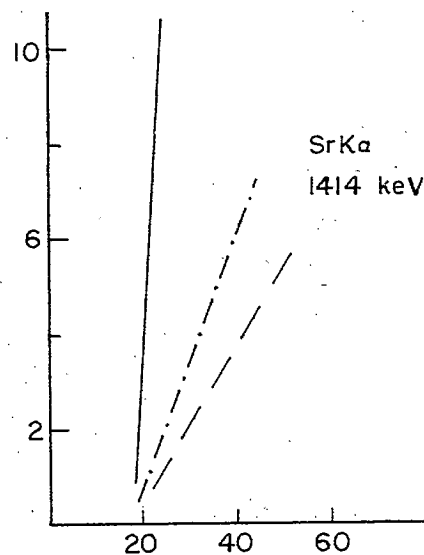
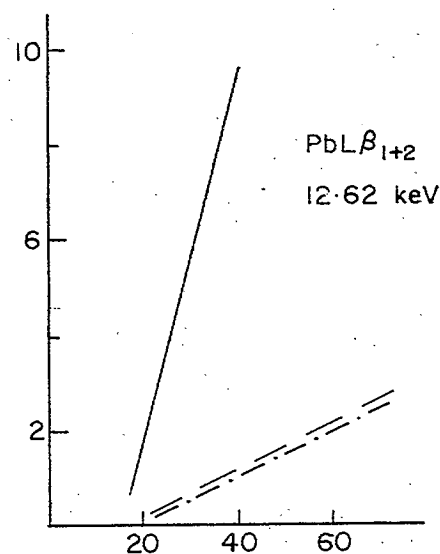
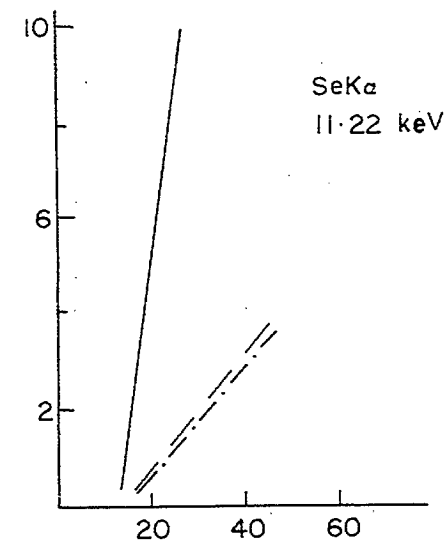
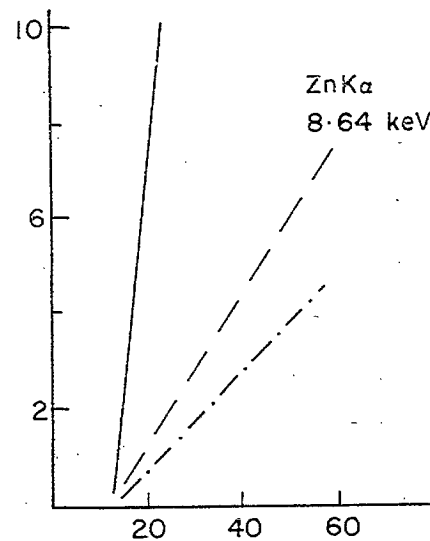
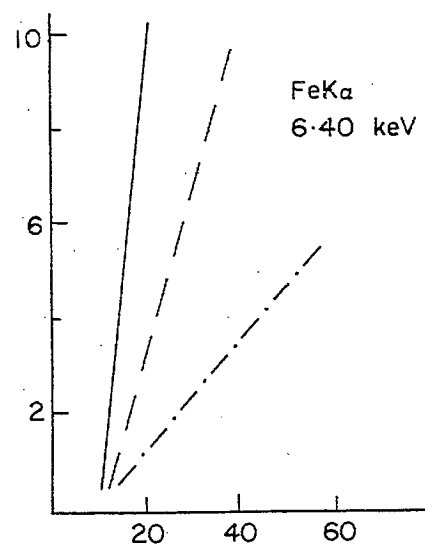


Figure 1. Reflection from Filter Paper of the Continuum.



Ordinates kV Abscissae 10⁴ cps — LiF - - - EDDT - · - · - quartz

Figure 2. Relative Intensity of Characteristic X-rays.

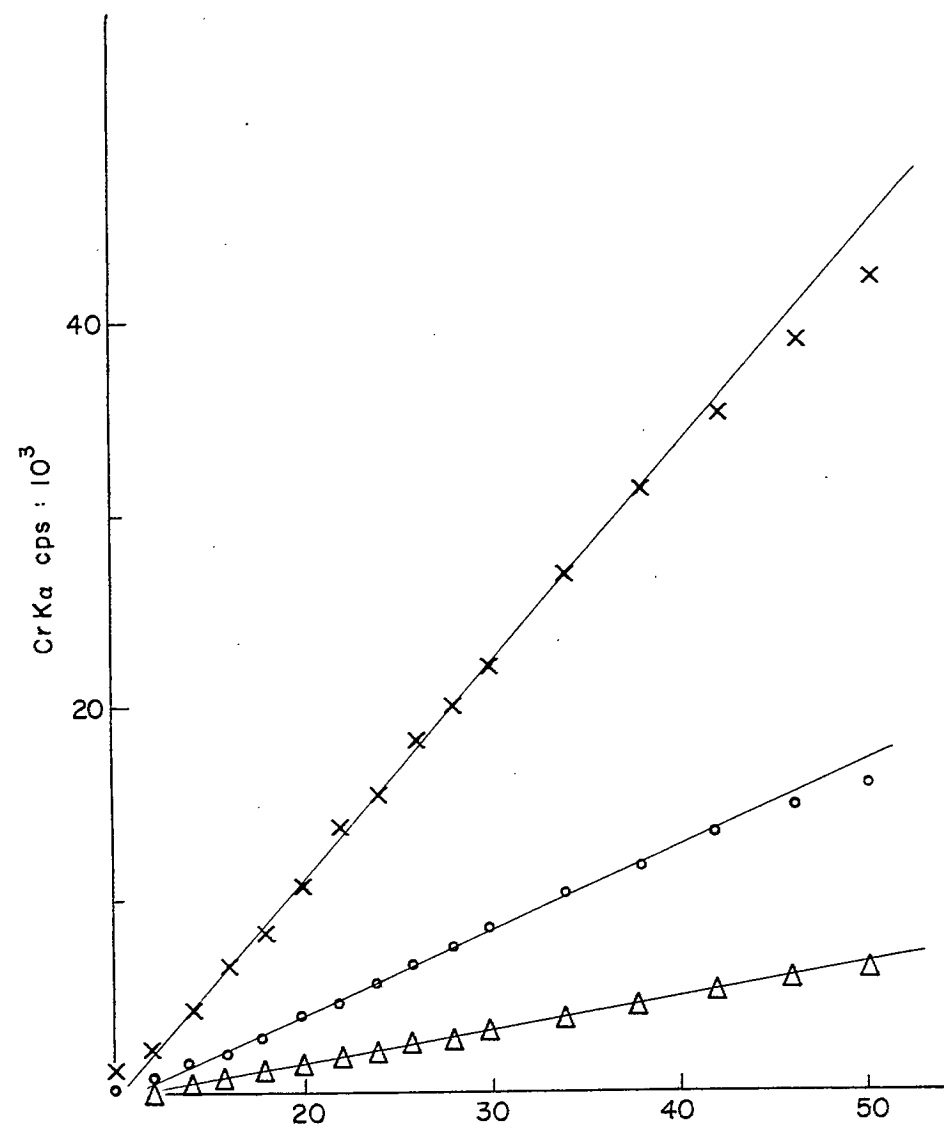
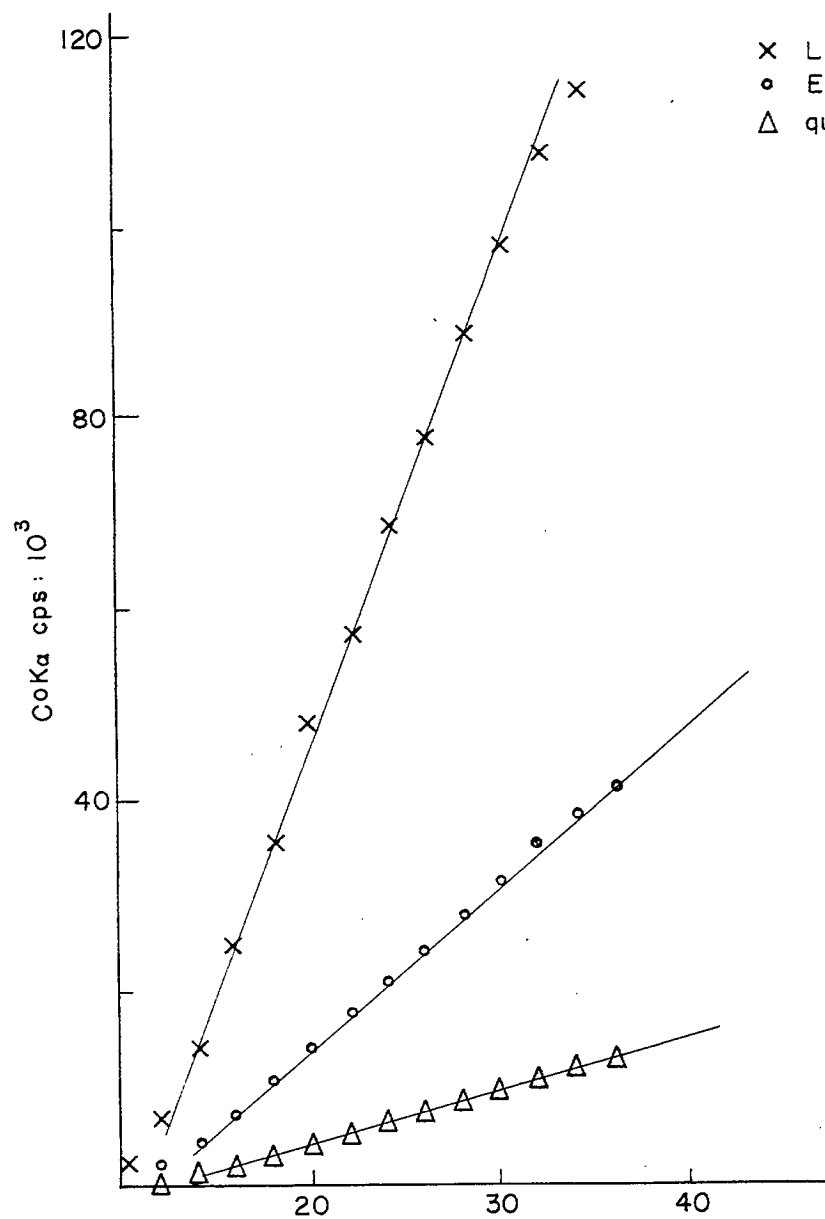


Figure 3. Response to Applied Potential.

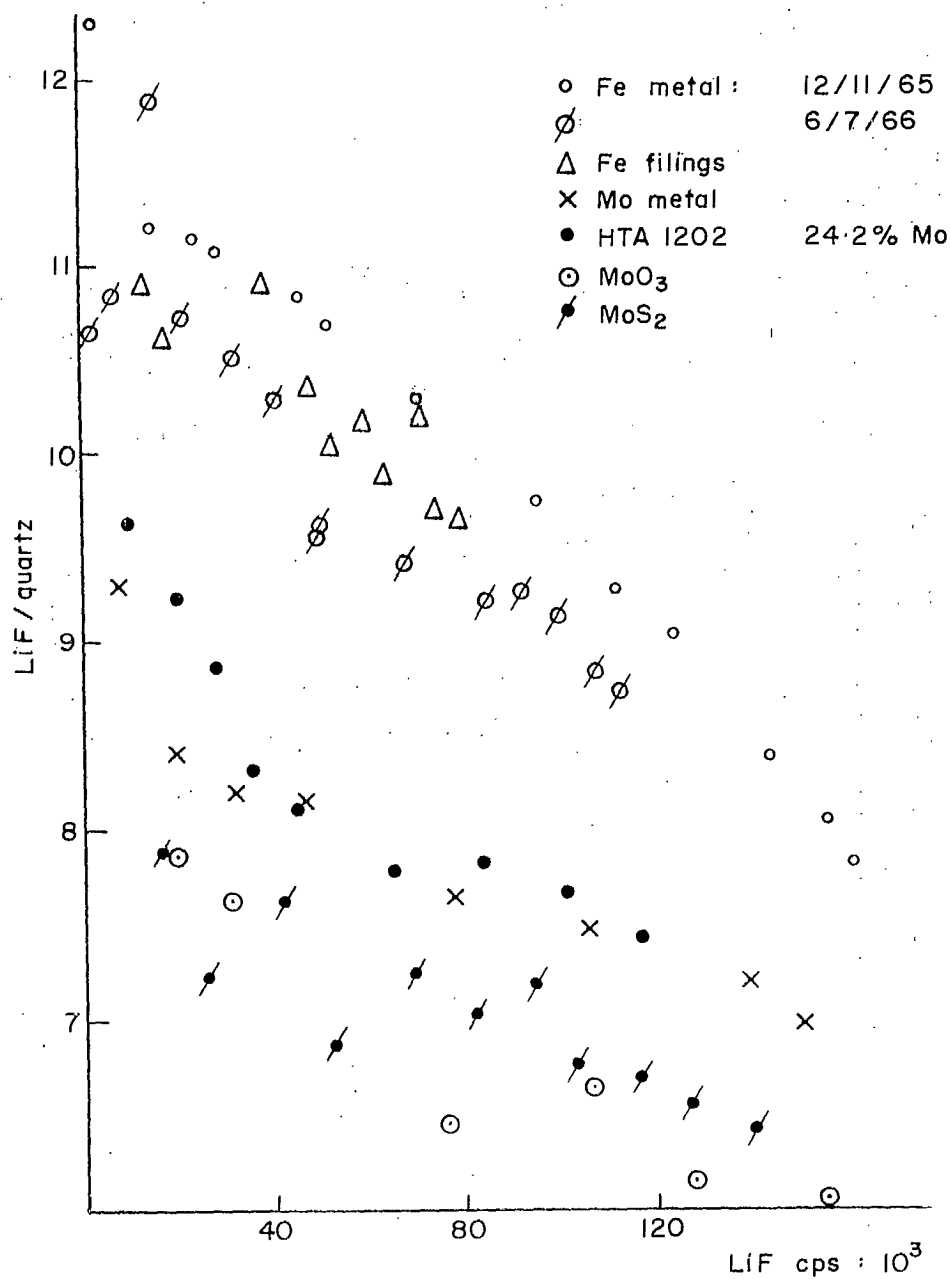


Figure 4. LiF/quartz for FeK α and MoK α at Different Counting Ratio.

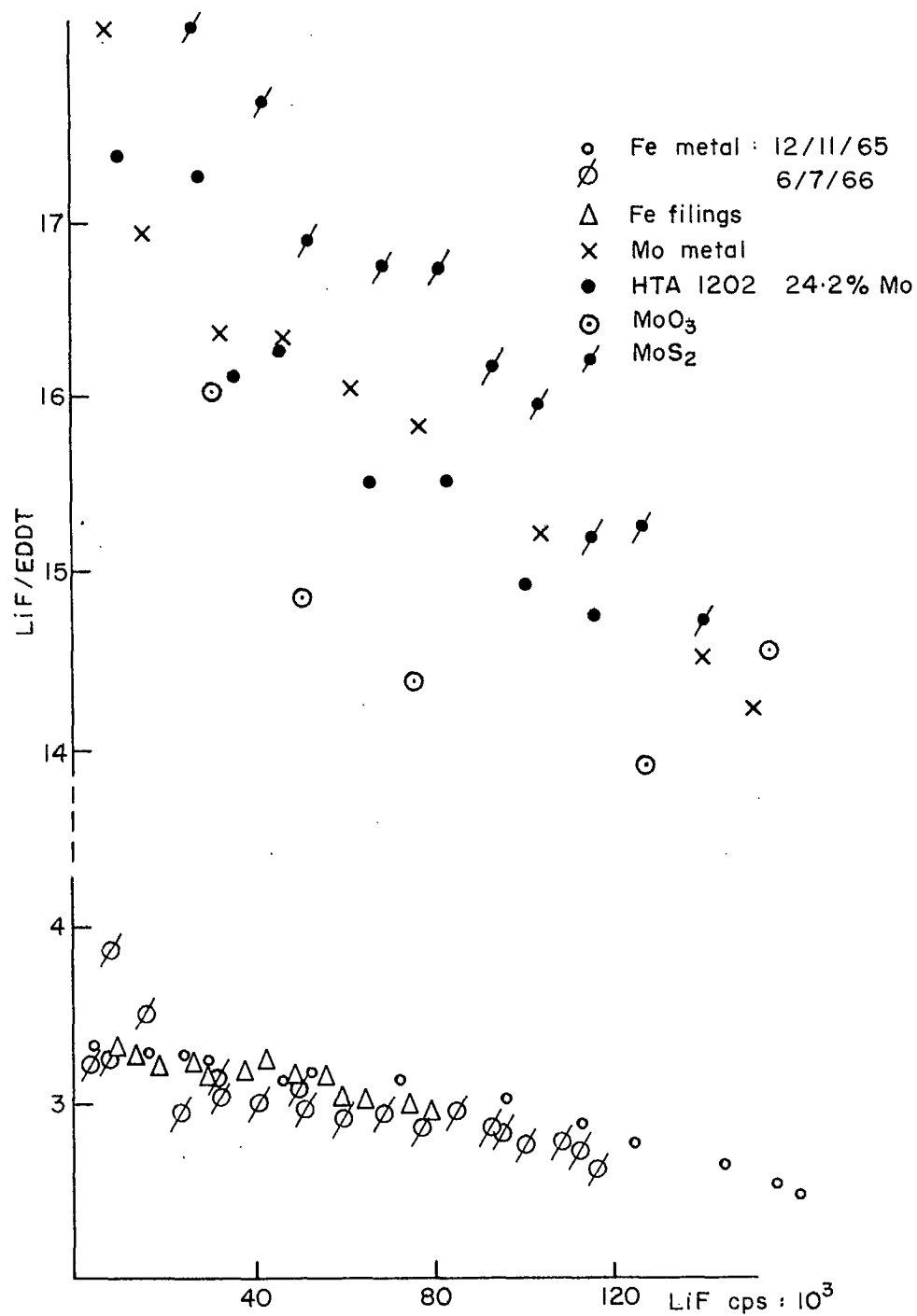


Figure 5. LiF/EDDT for $\text{FeK}\alpha$ and $\text{MoK}\alpha$ at Different Counting Ratio.

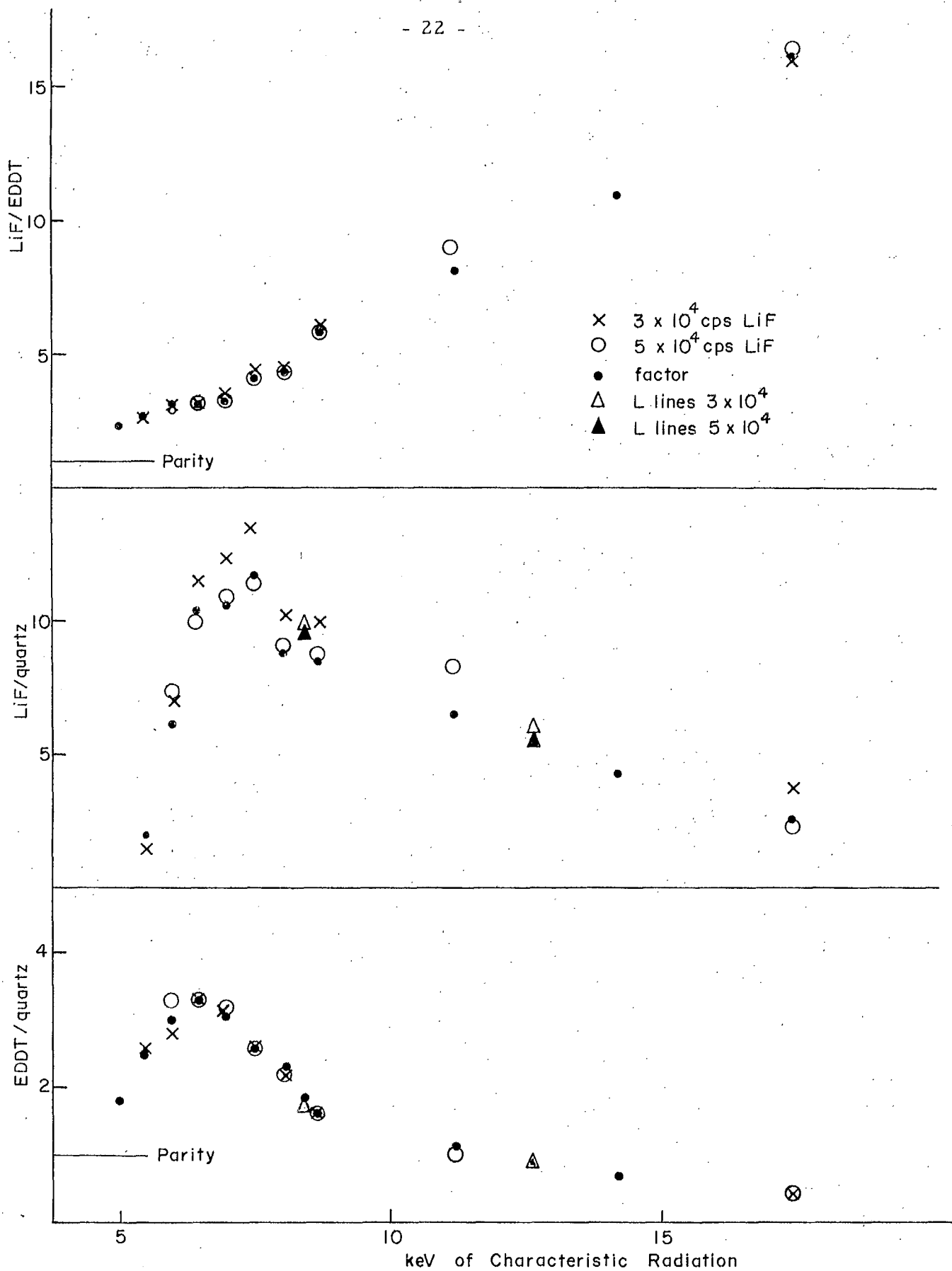


Figure 6. Variation of Crystal Ratios with keV.

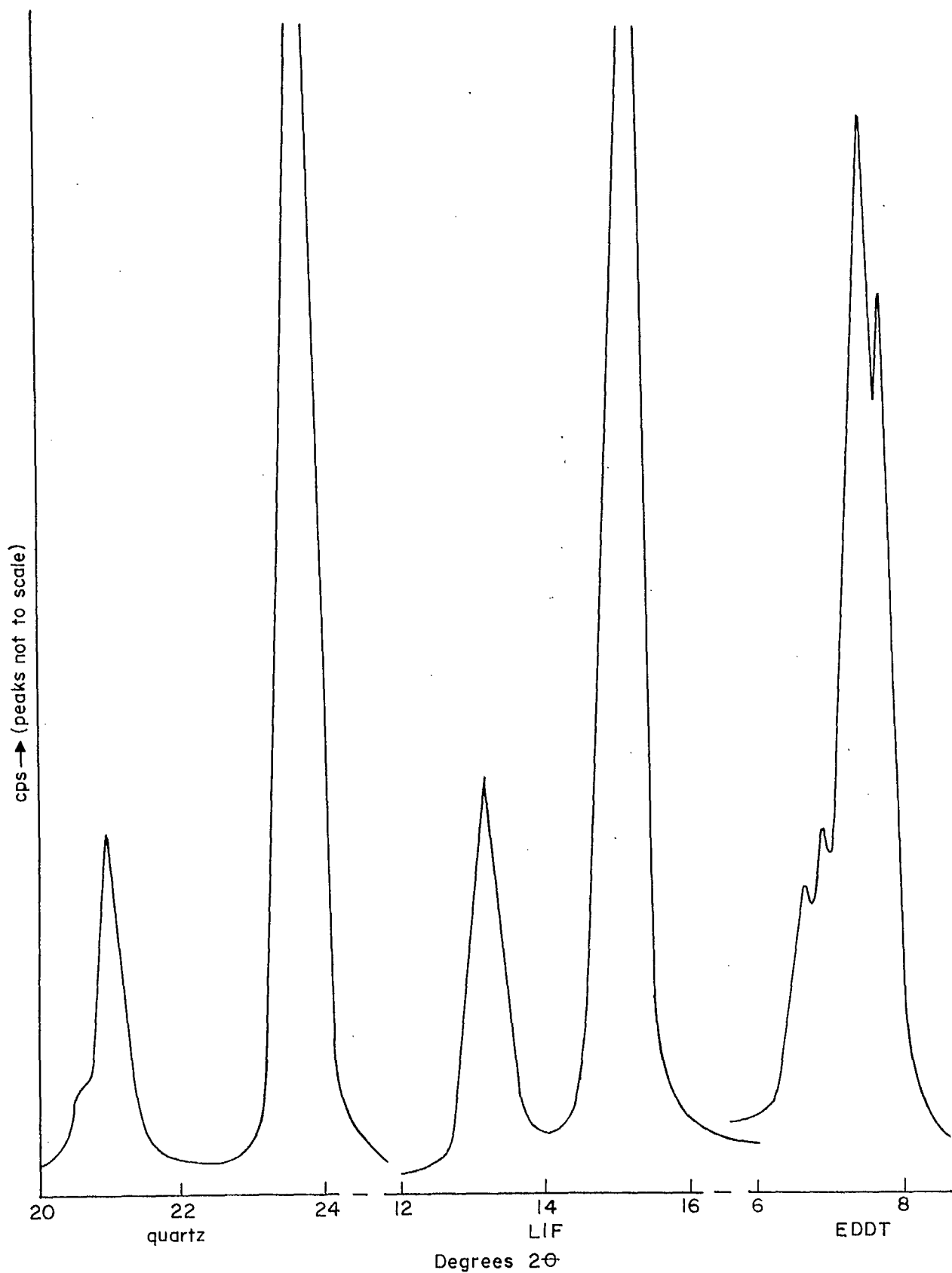


Figure 7. Scan of Ag_2SO_4 using Three Crystals.

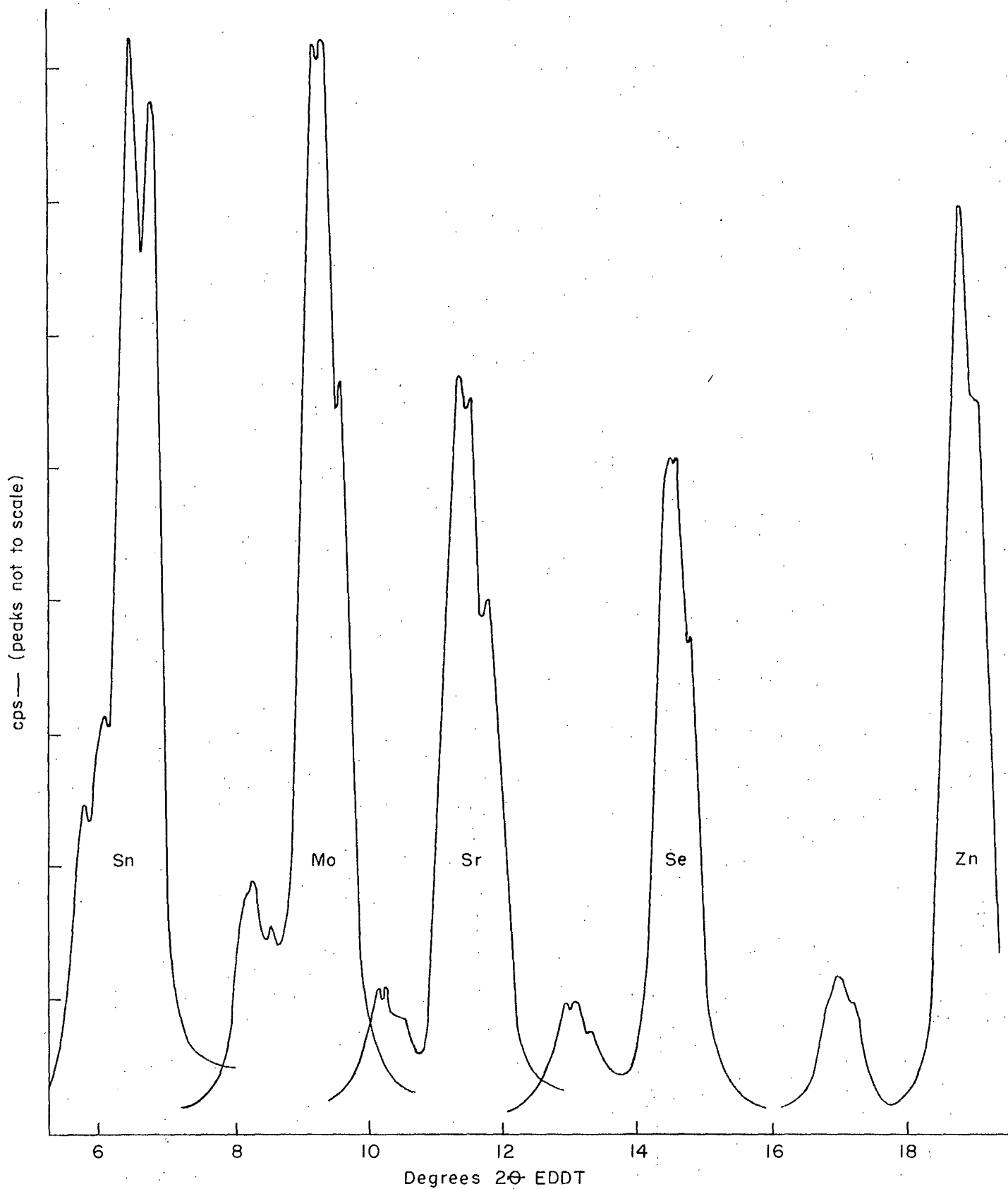


Figure 8. EDDT "Ghosts" of Various Elements.

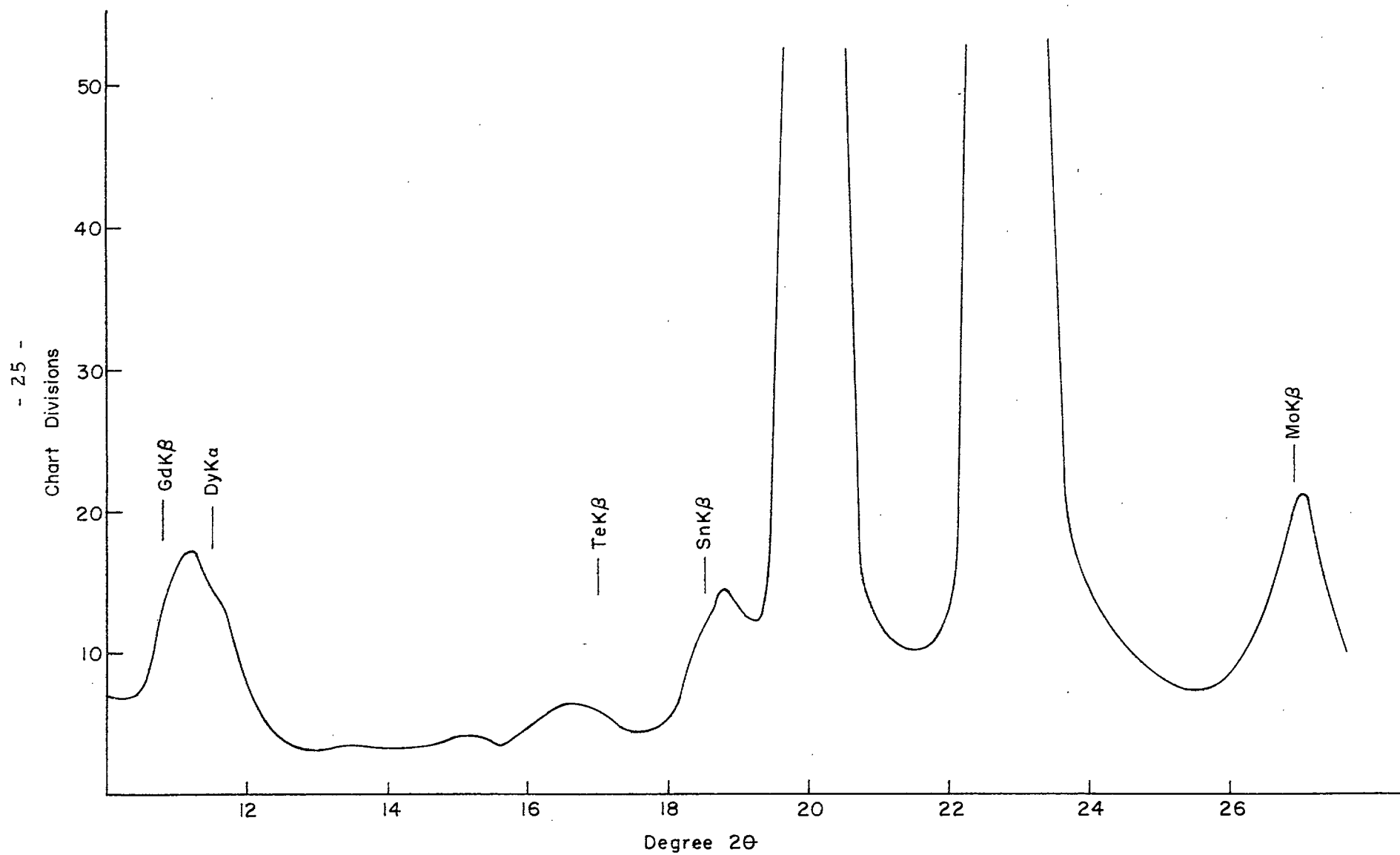


Figure 9. Topaz "Ghosts" of Cadmium.

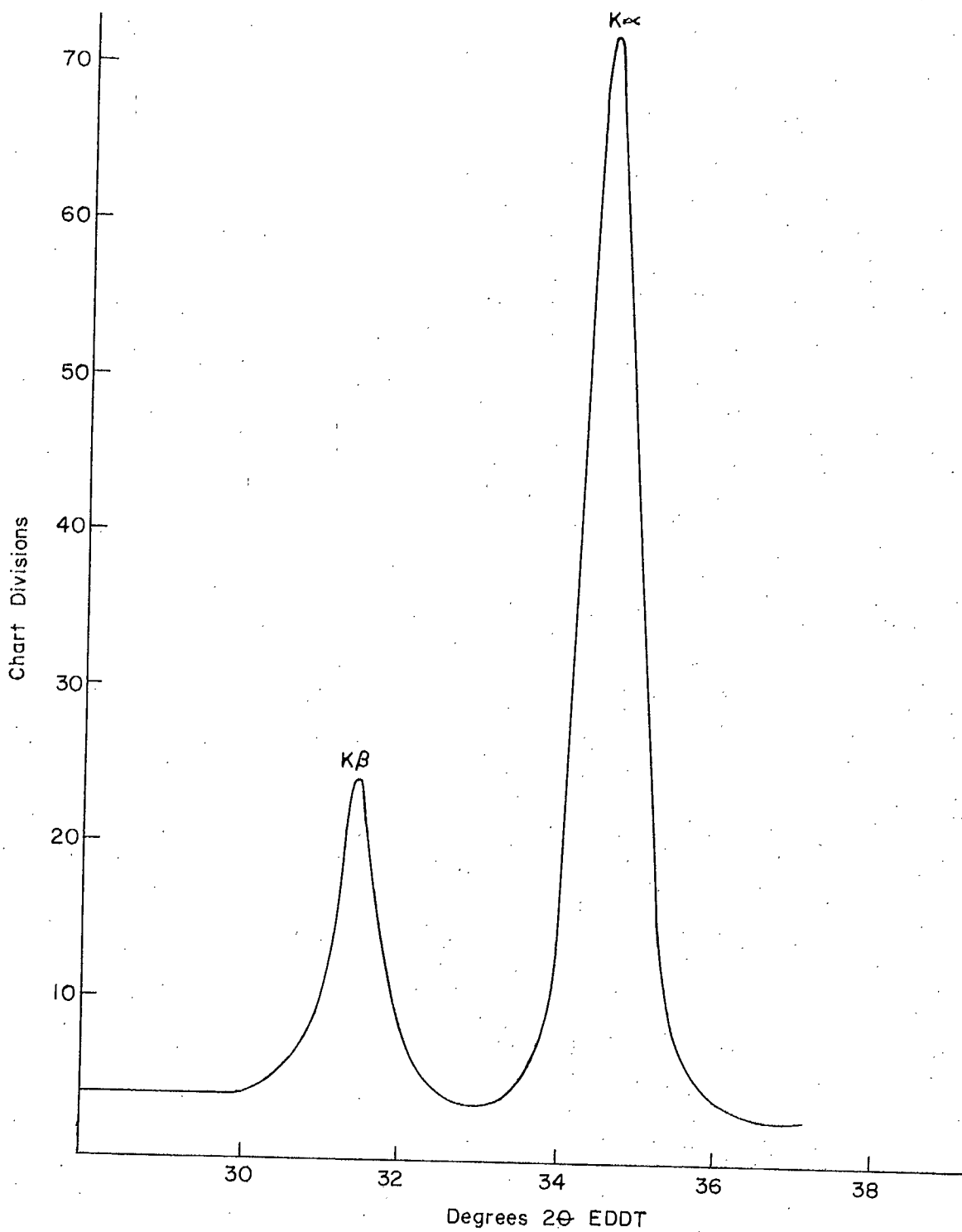


Figure 10. Scan of Titanium using FDDT.