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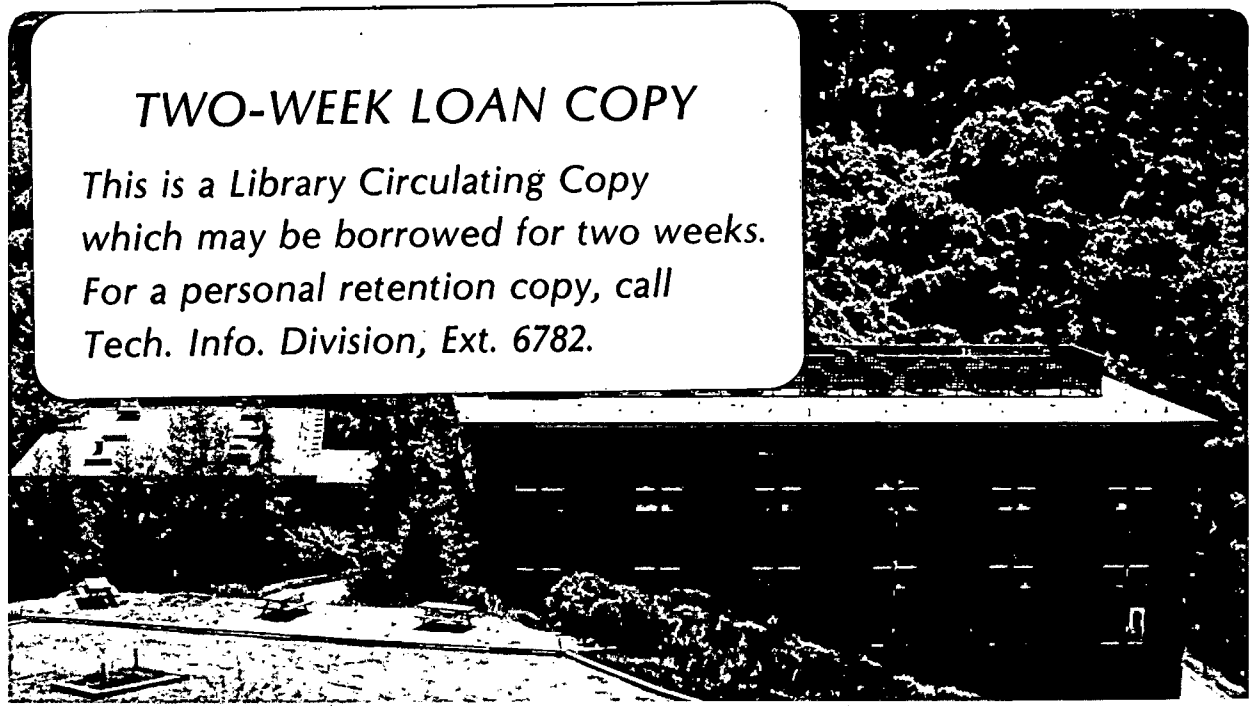
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ANOMALOUS SCATTERING OF X-RAYS BY CESIUM AND COBALT  
MEASURED WITH SYNCHROTRON RADIATION

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Both real and imaginary components of the anomalous X-ray scattering were measured by single-crystal diffraction experiments with synchrotron radiation at wavelengths through the region of the three L absorption edges of cesium, the first such experiment for any element at the L edges. Near the  $L_3$  edge  $f'$  varies between -26.7 and -13.9 electrons and  $f''$  between 4.0 and 16.1 in a wavelength interval of 0.008 Å. Similar but smaller changes occur near the  $L_2$  edge, and still smaller ones at  $L_1$ . Fine structure in the  $f''$  curve corresponds to that observed in an absorption curve and also, by a dispersion relation, to fine structure in the  $f'$  curve. These effects offer promise as a substitute for isomorphous replacement for solving the phase problem for macromolecular crystal structures. Similar experiments with cobalt near the K edge

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give  $f'$  values in agreement with measurements by other workers for nickel and copper by different methods at corresponding wavelengths; the lowest value observed is  $f' = -7.5 e$ .

## INTRODUCTION

Synchrotron radiation provides a continuous spectrum of X-rays intense enough for single-crystal diffractometer experiments at arbitrary wavelengths, even when a very narrow band of wavelengths is selected with a silicon or germanium crystal monochromator. We are interested in this technique as a means of measuring anomalous scattering effects near absorption edges at high resolution, both to confront the physical theory of the scattering process and to lay a foundation for applications of this new radiation source in solving complex crystal structures. A demonstration by photographic technique of the utility of synchrotron radiation and K-edge effects for studying protein structure was reported by Phillips, Wlodawer, Goodfellow, Watenpaugh, Sieker, Jensen & Hodgson (1977) with a review of some of the previous applications of anomalous scattering to the phase problem. It was recognized very early that anomalous scattering effects are larger at L edges than K edges, and Brentano and Baxter (1934) measured a reduction of 10.1 electrons in the magnitude of scattering by tungsten at a wavelength 6.5% longer than the  $L_3$  edge. In spite of the wide interest in anomalous

scattering among crystallographers and the fact that large effects can be predicted from existing data for absorption near L edges, there does not appear to have been any direct study of the variation of scattering closer to L edges until our work with cesium in cesium hydrogen tartrate. The method is to derive the anomalous scattering terms  $f'$  and  $f''$  by least-squares adjustment using diffraction data measured with a crystal whose structure is known accurately from work at another wavelength (Templeton and Templeton, 1978). The first such experiments with synchrotron radiation (Phillips, Templeton, Templeton and Hodgson, 1978) showed an unprecedented decrease in scattering power of cesium at the  $L_3$  edge,  $f' = -27.1 \pm 0.9$ . In the present paper we report more extensive measurements of cesium at all three L edges and some similar experiments with cobalt near its K edge.

Cesium was chosen for the first experiments because its L absorption edges occur at wavelengths which are a compromise between higher-energy photons which better penetrate air and beam-line windows, and lower-energy ones which are produced more intensely by the storage ring in its frequent low-energy mode of operation. Cesium hydrogen (+)-tartrate was chosen because of its desirable physical properties and favorable symmetry. It crystallizes in space group  $P2_12_12_1$  with one molecule in the asymmetric unit. A non-centric structure improves the determination of  $f''$ . The 222 point symmetry makes it convenient to measure equivalent reflections and Bijvoet pairs on the same side of the crystal, whatever its orientation. The chiral tartrate molecule guarantees that the

crystal is a pure enantiomer. The location of all atoms in general positions gives variety to their contributions to structure factors and tends to decrease correlations between different parameters. The lattice parameters, atomic coordinates and thermal parameters are known accurately ( $R = 0.016$ ) from an experiment with Mo  $K\alpha$  radiation by conventional methods (Templeton and Templeton, 1978).

Cobalt was studied as a check of the method at a  $K$  absorption edge by comparison with measurements of nearby elements by other techniques and as a test of the diffraction apparatus at a wavelength near Cu  $K\alpha$ . Crystals of (+)-tris(ethylenediamine)cobalt(III) chloride (+)-tartrate were at hand and the necessary structural parameters had been determined by Templeton, Zalkin, Ruben and Templeton (1979) using Mo  $K\alpha$  radiation ( $R = 0.022$ ).

#### EXPERIMENTAL

The experiments were done at the Stanford Synchrotron Radiation Laboratory with X-rays produced by the SPEAR storage ring. Measurements were carried out with the SSRL diffractometer system which includes a doubly-curved mirror and two-crystal germanium (111) monochromator to focus the beam and select the wavelength (Hastings, Kincaid and Eisenberger, 1978), an Enraf-Nonius CAD4 diffractometer with scintillation counter to measure diffraction intensities, an ionization chamber to monitor the incident beam intensity, and a PDP 11/34 computer to record the data and to control the operation

and mechanical adjustments of the equipment. A detailed description of the system and its performance has been given by Phillips, Cerino and Hodgson (1979). The diffractometer is enclosed in a steel box ("hutch") for radiation safety. It is turned on its side so that diffracted beams are measured in a vertical plane, at right angles to the electric vector of the horizontally polarized synchrotron radiation. Some of the experiments at the cesium  $L_1$  edge were done with a modified collimator system and different but similar mirror and monochromator on another beam line at a slightly greater distance from the storage ring.

The measurements reported here were made with three spherical crystals of  $\text{CsHC}_4\text{H}_4\text{O}_6$  with diameters about 0.33, 0.28 and 0.38 mm and a crystal of  $\text{Co}(\text{C}_2\text{H}_8\text{N}_2)_3\text{Cl}(\text{C}_4\text{H}_4\text{O}_6) \cdot 5\text{H}_2\text{O}$  with six well-defined faces and dimensions  $0.102 \times 0.084 \times 0.110$  mm, each mounted on a glass fiber with epoxy glue.

The monochromator angle is set very precisely by a stepping motor with 1200 or 2000 steps per degree, but it lacks a permanent absolute zero. Consequently the wavelength scale was determined frequently by measuring absorption curves with a sample inserted between the ion chamber and the diffractometer scintillation counter (set at zero angle with the beam stop removed) and passing the X-rays through the diffractometer collimator. The wavelengths are assumed to be 2.1673, 2.3139 and 2.4740 Å for the cesium L edges and 1.6081 Å for the cobalt K edge (Bearden, 1967).



These experiments were done over a period of 20 months under a variety of conditions of accelerator operation with electron energies in the storage ring ranging from 1.9 to 3.6 GeV. Most of the useful results were obtained with electron energies above 2.3 GeV.

Diffraction intensities were recorded in the  $\omega$ - $2\theta$  scan mode with scan speed set to achieve typically 4% statistics. A typical procedure was to measure 40 different reflections, sometimes with repetitions, which after averaging of equivalent ones yielded two centric reflections and 9 Bijvoet pairs. These reflections, with  $(\sin\theta)/\lambda$  in the range 0.26 to 0.36, were selected so that cesium and light atoms added with a variety of phase angles to avoid high correlation among  $f'$ ,  $f''$  and the scale factor. The cobalt experiments were done with 34 reflections (17 Bijvoet pairs) with  $(\sin\theta)/\lambda$  between 0.214 and 0.234.

Absorption effects in these experiments are large and require careful attention. At an absorption edge the absorption coefficient  $\mu$  is a very sensitive function of wavelength, and in principle its value is unknown in advance; it is directly related to  $f''$  which is one of the objects of the experiment. Sometimes it is necessary to repeat the data analysis, using a value of  $\mu$  based on the result of the first iteration. However, since the scale factor is also one of the parameters which is varied, it is only necessary that the ratios of absorption corrections for different reflections be correct. These ratios are much less sensitive to  $\mu$  than are the absolute

values of the transmission factors. In most of our calculations  $\mu$  was estimated accurately enough in advance to make iteration unnecessary. For a spherical crystal the ratios of transmission factors become independent of  $\mu$  in the limit of large  $\mu R$ , and limitation of the measurements to a small range of Bragg angles makes the corrections nearly the same for all reflections. For computational convenience we derived an expression which is valid within about 1% for  $\mu R > 5$ :

$$\begin{aligned}
 T = 1 - \frac{\cos^2 \theta}{\sin \theta} \ln \tan \left( \frac{\pi}{4} + \frac{\theta}{2} \right) + \frac{1 + \cos 2\theta}{(\mu R)^2} \\
 + \left[ \frac{0.64}{\mu R} - \frac{1}{(\mu R)^2} \right] \sin 2\theta - \frac{0.20}{(\mu R)^2} \sin 4\theta \\
 - \frac{0.02}{\mu R} \sin 6\theta
 \end{aligned} \tag{1}$$

where  $T$  is the transmission at Bragg angle  $\theta$  relative to that at  $\theta = 90^\circ$  for a sphere of radius  $R$  and absorption coefficient  $\mu$ . The first two terms give the limiting value of  $T$  for infinite  $\mu R$ , the third term is an asymptotic expression for  $T$  at  $\theta = 0^\circ$  and goes to zero at  $\theta = 90^\circ$ , while the remaining terms are an approximation adjusted to results of numerical integrations in the range  $\mu R = 5$  to 40 for intermediate values of  $\theta$ .

For the cesium salt the value of  $\mu$  ranged from 360 to 1320  $\text{cm}^{-1}$ , and correction factors ( $T^{-1}$ ) from 1.7 to 3.4 in the extreme case.

Absorption corrections for the prismatic cobalt crystal were calculated by the analytical method with our program ABSOR (Templeton and Templeton, 1973); they ranged from 1.25 to 1.34 for  $\mu = 30 \text{ cm}^{-1}$  (the minimum value) and from 2.15 to 2.77 for  $\mu = 110 \text{ cm}^{-1}$  (the maximum value).

Because of the highly polarized nature of synchrotron radiation and the geometry of the experiment (scattering in the plane perpendicular to the electric vector of the X-ray) no correction was made for polarization effects.

The method of least squares was used to derive  $f'$  and  $f''$  for the heavy atom from the data at each wavelength. The scale factor was the only other variable. Anomalous scattering terms for the light atoms, which are small but not negligible, were interpolated from the tables of Cromer and Liberman (1970). The structural parameters were taken from Templeton and Templeton (1978) and Templeton, Zalkin, Ruben and Templeton (1979).

#### CESIUM ANOMALOUS SCATTERING

The results for cesium are listed in Table 1 and plotted in Fig. 1. Our first experiments (Phillips, Templeton, Templeton and Hodgson, 1978) already showed  $f'$  values as negative as -26 electrons at the  $L_3$  edge, and the new data confirm that sharp dip. Points are measured on the sharp peaks of  $f''$  which occur at both the  $L_2$  and

$L_3$  edges, showing that diffraction experiments can be carried out with sufficient wavelength precision to exploit such fine-structure effects. Additional modulation of  $f'$  on the short wavelength side of the edge, already familiar in absorption measurements, is also observed. One sees that the  $f'$  values reflect these same effects (modified by the dispersion relation) with substantial amplitude. The main features observed in both  $f'$  and  $f''$  at the  $L_3$  edge are reflected with about half the amplitude at the  $L_2$  edge, indicating little difference in the electronic transitions involved in the photoelectric effect at these edges except the multiplicity difference of the  $p_{3/2}$  and  $p_{1/2}$  vacancy states of the L shell. The resemblance of this structure at the two edges is also evidence in support of the validity of the experimental method.

The anomalous scattering terms  $f'$  and  $f''$  are related to the absorption cross section  $\sigma$  by the relations (James, 1962; Wagenfeld, 1975):

$$f''(\omega) = \frac{m c \omega \sigma(\omega)}{4 \pi e^2} \quad (2)$$

$$f'(\omega) = \frac{2}{\pi} \int_0^{\infty} \frac{\omega' f''(\omega') d\omega'}{\omega^2 - \omega'^2} \quad (3)$$

where the Cauchy principal value is taken for the integral in (3),

which is one of the forms of the Kramers-Kronig dispersion relation. These expressions refer to the values of  $f'$  and  $f''$  for forward scattering, whereas our experiments measure them at finite scattering angles. The difference is likely to be only a few percent or less, but is one of the reasons for measuring them in actual diffraction experiments. To test the applicability of relation (2) values of  $f''/\omega$  are plotted in Fig. 2, together with an absorption spectrum (minus a linear background) of cesium hydrogen tartrate. As the absorption spectrum was not on an absolute scale it has been adjusted by a scale factor to coincide with the extreme values of  $f''$ . The good fit indicates that absorption curves will be useful guides for prediction of  $f''$  in other materials. Strong absorption lines like the one seen here have been called "white lines" because of their appearance when recorded on photographic plates.

The anomalous scattering terms near the  $L_3$  edge are shown in Fig. 3 on an expanded wavelength scale for comparison with values calculated by Cromer (1978) according to the method of Cromer and Liberman (1970) with a change in the numerical integration procedure to make it valid for wavelengths very close to the edge. Also plotted is a curve of  $f'$  calculated from  $f''$  according to equation (3) with integration limits  $7.44 \text{ \AA}$  to  $0.022 \text{ \AA}$ . Our experimental values of  $f''$  were used near the L edges, with some interpolation guided by the behavior of absorption curves. Values at more remote wavelengths were based on the calculations of Cromer

and Liberman (1970). The agreement of absolute scale is excellent. The oscillation of the experimental points is greater than that of the calculated curve and suggests that we have underestimated the sharpness of the fine structure of  $f''$ . This result gives encouragement that useful estimates of  $f'$  can also be derived from absorption measurements.

The model of Cromer and Liberman (1970) lacks modulations of the density of states in the continuum and neglects transitions to vacant bound states. The edge discontinuity is assumed to be very sharp. Thus the calculated curve for  $f''$  is featureless except for the edge discontinuity. Except for absence of the white line at the edge and lack of modulation the curve agrees well with our results. The corresponding curve for  $f'$  is higher than our experimental values at longer wavelengths and lower at shorter wavelengths because of the neglect of the substantial effect of the white line. If the edge is assumed to be infinitely sharp,  $f'$  goes to negative infinity according to a logarithmic singularity. In reality, finite levels widths place a limit on the sharpness of the edge and a bound on the negative excursion of  $f'$ . Thus this model does not give a useful comparison for the extreme values of  $f'$ .

#### COBALT ANOMALOUS SCATTERING

The results for cobalt are listed in Table 2, and those nearest the K absorption edge are plotted in Fig. 4. Also plotted are values

for  $f'$  for copper derived by Freund (1975) from absolute intensity measurements and for nickel measured by interferometry by Bonse and Materlik (1976). The nickel values were estimated from the published curve. The values for the three elements are brought to the same abscissas by plotting the ratios of wavelengths to the respective K-edge wavelength. The solid lines indicate the Cromer and Liberman (1970) values of  $f''$ . The measurements of  $f'$  by the three techniques are in excellent agreement. The disagreement with theory for  $f''$  is no greater than one expects considering that some fine structure features are present in the absorption curve. Beam time did not permit these to be explored in more detail. In contrast to the cesium  $L_3$  edge, there is little advantage in going to this K edge rather than  $Cu K\alpha$  if one wishes to exploit  $f''$ . The reduction by about 5 electrons in  $f'$  in this wavelength interval is large enough to be useful for some purposes, but is not competitive with the changes observed with cesium or expected for rare earth  $L_3$  edges.

#### APPLICATIONS TO THE PHASE PROBLEM

A major objective to this work was to provide a foundation for the use of synchrotron radiation in solving the phase problem for macromolecular crystals. The use of Bijvoet differences, which are approximately proportional to  $f''$ , is well established as a tool for this purpose. It is obvious that the power of this technique is enhanced if one can collect diffraction data at the peak of a

white line, but the gain relative to a well-chosen characteristic line source may be relatively modest. For example, for cesium we observe  $f'' = 16$  electrons as the maximum value near the  $L_3$  edge, compared with about 13 e at  $CrK\alpha$  or 11 e at  $FeK\alpha$ . The tunability of synchrotron radiation is more important if one seeks to use the changes in  $f'$ ; the largest effects are confined to rather narrow wavelength intervals not accessible for most elements with characteristic line sources, and less use has been made of  $f'$  to solve diffraction problems. The difference of 26 electrons which we observe for cesium between  $CuK\alpha$  and the  $L_3$  edge is more than double any difference found for any element in the table of Cromer and Liberman (1970) for five  $K\alpha$  wavelengths.

The effect of  $f'$  on diffraction data is approximately like that of a change of atomic number. Thus if experiments are done at wavelengths chosen to give large changes in  $f'$ , the result is like an isomorphous substitution with the advantage that the isomorphism is perfect. A change of 26 electrons is equivalent to the normal scattering of an iron atom at zero angle or a strontium atom at  $(\sin\theta)/\lambda = 0.3$  and thus is in a range which has been used to solve protein structures. Naturally it is more powerful to use the combined effects of  $f'$  and  $f''$ .

A familiar diagram in the complex plane, Fig. 5, illustrates the structure factor algebra for determining the phase of an X-ray reflection. If the heavy atom structure is known one can calculate the magnitude and phase of  $F_H$ , the structure factor for the heavy atoms. The structure factor vector  $F_L$  for the light atoms terminates



on a circle with center at  $-F_H$  and radius equal to the measured structure factor magnitude  $|F|$ , since  $F = F_H + F_L$ . If the same reflection is measured at another wavelength where the anomalous scattering terms are different, a new circle with a different center and perhaps different radius is determined. Measurements of the other member of the Bijvoet pair determine circles with centers placed symmetrically on the opposite side of  $F_H^0$ , the heavy atom structure factor if  $f' = f'' = 0$ . In general three measurements are enough for a unique solution at the point where the three circles intersect. In the figure the two curved lines are the loci of centers for wavelengths near the  $L_3$  edge, drawn to scale for our cesium measurements. Two small circles at the left indicate centers for measurements at  $CuK\alpha$ . It is evident that the power for phase determination depends on how far apart the centers are and that the use of Bijvoet pairs doubles the effect of a large  $f''$  value. Whether changes in  $f'$  or in  $f''$  are more effective for a particular reflection will depend on the phase relations in that case. One or the other will always have a suitable phase.

For simplicity this discussion neglects the anomalous scattering of the light atoms, which is not insignificant at the wavelengths used in this work. While awkward to describe in the vector diagram, this effect is easy to include in an algebraic solution.

This method has already been tested by Hoppe and Jakubowski (1975) with a crystal of erythrocrucorin (containing one iron atom per 16,000

daltons) using two wavelengths near the iron K edge. They determined phases for many reflections with an accuracy of about  $50^\circ$  with anomalous scattering effects smaller by a factor of 4 than those reported here.

While the objective of solving the phase problem is enough reason to explore these anomalous scattering effects, a more fundamental result may be in improvement in the accuracy of determinations of macromolecular structure. Refinements of non-centric structures are hampered by a circularity: structure is derived in part from phases and then phases are derived from the new structure. A least-squares refinement based on data measured at several appropriate wavelengths rather than just one is likely to be more reliable simply because of the greater information content of the data. We conjecture that it will converge more rapidly and be more discriminating with regard to false minima or near-minima because of the phase information contained in the data. The same could be said of simultaneous refinement of data for isomorphous derivatives if the isomorphism were perfect, but unfortunately it rarely if ever is for complicated structures.

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Table 1. Anomalous scattering terms for cesium and agreement index  $R = \Sigma|\Delta F|/\Sigma|F|$  for each data set. Standard deviations of last digits are given in parentheses. The values at 2.2895 Å may be compared with -10.742 and 12.919 at Cr  $K\alpha_1$  (2.2896 Å) calculated by Cromer and Liberman (1970).

$\lambda(\text{Å})$	$f'$	$f''$	R
2.7021	-9.8(5)	4.0(4)	0.020
2.5021	-14.7(3)	3.6(2)	0.019
2.4909	-15.9(3)	3.6(3)	0.022
2.4891	-16.0(2)	3.8(2)	0.017
2.4874	-16.2(3)	3.5(2)	0.023
2.4856	-16.8(3)	3.6(2)	0.022
2.4838	-17.1(4)	3.4(3)	0.027
2.4824	-17.1(2)	3.7(2)	0.037
2.4793	-19.2(2)	4.0(2)	0.025
2.4786	-19.1(5)	4.0(3)	0.049
2.4751	-24.8(3)	5.8(1)	0.036
2.4740	-24.6(7)	7.4(4)	0.049
2.4737	-26.7(3)	8.9(3)	0.048
2.4722	-16.8(6)	16.1(8)	0.044
2.4703	-13.9(10)	10.8(6)	0.056
2.4680	-15.3(9)	10.3(8)	0.079
2.4638	-16.2(4)	11.3(4)	0.037
2.4612	-13.8(3)	10.7(3)	0.014
2.4579	-14.0(3)	9.7(3)	0.017
2.4545	-13.9(4)	9.9(4)	0.018

Table 1. Continued

2.4492	-14.2(3)	10.2(3)	0.023
2.4242	-13.4(4)	9.7(3)	0.023
2.3992	-12.3(4)	10.0(4)	0.017
2.3743	-11.6(4)	10.3(4)	0.019
2.3495	-11.8(4)	9.8(3)	0.014
2.3246	-12.8(2)	9.6(2)	0.012
2.3169	-14.3(3)	9.5(3)	0.016
2.3146	-18.1(3)	10.5(3)	0.023
2.3139	-17.3(8)	11.1(6)	0.047
2.3134	-16.0(9)	14.9(11)	0.052
2.3123	-14.5(7)	16.1(8)	0.043
2.3118	-14.9(12)	13.7(10)	0.058
2.3109	-12.2(5)	13.7(6)	0.032
2.3103	-13.1(10)	12.9(8)	0.047
2.3091	-12.6(5)	12.5(6)	0.032
2.3070	-13.2(6)	13.1(7)	0.035
2.3041	-11.7(14)	13.7(15)	0.066
2.3013	-11.4(5)	12.9(7)	0.040
2.2895	-11.1(6)	12.8(8)	0.047
2.2495	-9.4(6)	12.7(6)	0.024
2.1995	-8.9(6)	12.4(5)	0.025
2.1800	-9.7(12)	11.0(12)	0.054
2.1730	-8.8(9)	11.9(9)	0.037

2.1698	-9.5(8)	11.5(8)	0.034
2.1673	-12.2(9)	12.2(8)	0.037
2.1671	-11.1(5)	14.1(6)	0.074
2.1653	-11.7(11)	12.7(10)	0.050
2.1639	-11.2(10)	13.4(9)	0.048
2.1633	-9.4(9)	13.7(9)	0.035
2.1563	-8.8(10)	13.1(10)	0.041
2.1402	-7.4(10)	12.6(10)	0.038
1.601	-2.8(7)	8.1(5)	0.033

Table 2. Anomalous scattering terms for cobalt.

$\lambda(\text{\AA})$	$f'$	$f''$	R
1.541 <sup>a</sup> (Cu $K\alpha_1$ )	-2.464	3.608	
1.542 <sup>b</sup> (Cu $K\alpha$ )	-2.36(2)	3.92(2)	0.029
1.546	-2.7(4)	3.9(5)	0.061
1.597	-5.7(5)	5.1(5)	0.084
1.605	-7.0(3)	4.6(2)	0.069
1.607	-7.5(3)	4.1(4)	0.067
1.609	-7.4(4)	1.0(4)	0.069
1.614	-4.9(3)	0.7(3)	0.051

<sup>a</sup>Calculated by Cromer and Liberman (1970).

<sup>b</sup>Measured by Templeton, Zalkin, Ruben and Templeton (1979).



FIGURE CAPTIONS

- Fig. 1. Anomalous scattering terms for cesium determined from diffraction data in the region of the  $L_1$  absorption edge (left),  $L_2$  edge (center) and  $L_3$  edge (right).
- Fig. 2. Absorption coefficient (solid curve) for cesium hydrogen tartrate at the  $L_3$  edge, less a linear background, plotted on an arbitrary scale, and values of  $f''/\omega$  derived from the diffraction experiments (circles with error bars), scale normalized and shifted to fit at extreme points.
- Fig. 3. Anomalous scattering terms measured near the  $L_3$  edge (circles) and calculated (broken lines, Cromer, 1978). The solid curve for  $f''$  is drawn through the experimental points. The solid curve for  $f'$  is calculated by the dispersion relation as described in the text.
- Fig. 4. Anomalous scattering terms near the K edge measured in this work for cobalt, by Freund (1975) for copper, and by Bonse and Materlik (1976) for nickel and extrapolated for cobalt from the calculations of Cromer and Liberman (1970).
- Fig. 5. Vector diagram in the complex plane for phase determination with structure factor magnitudes measured at several wavelengths (see text).

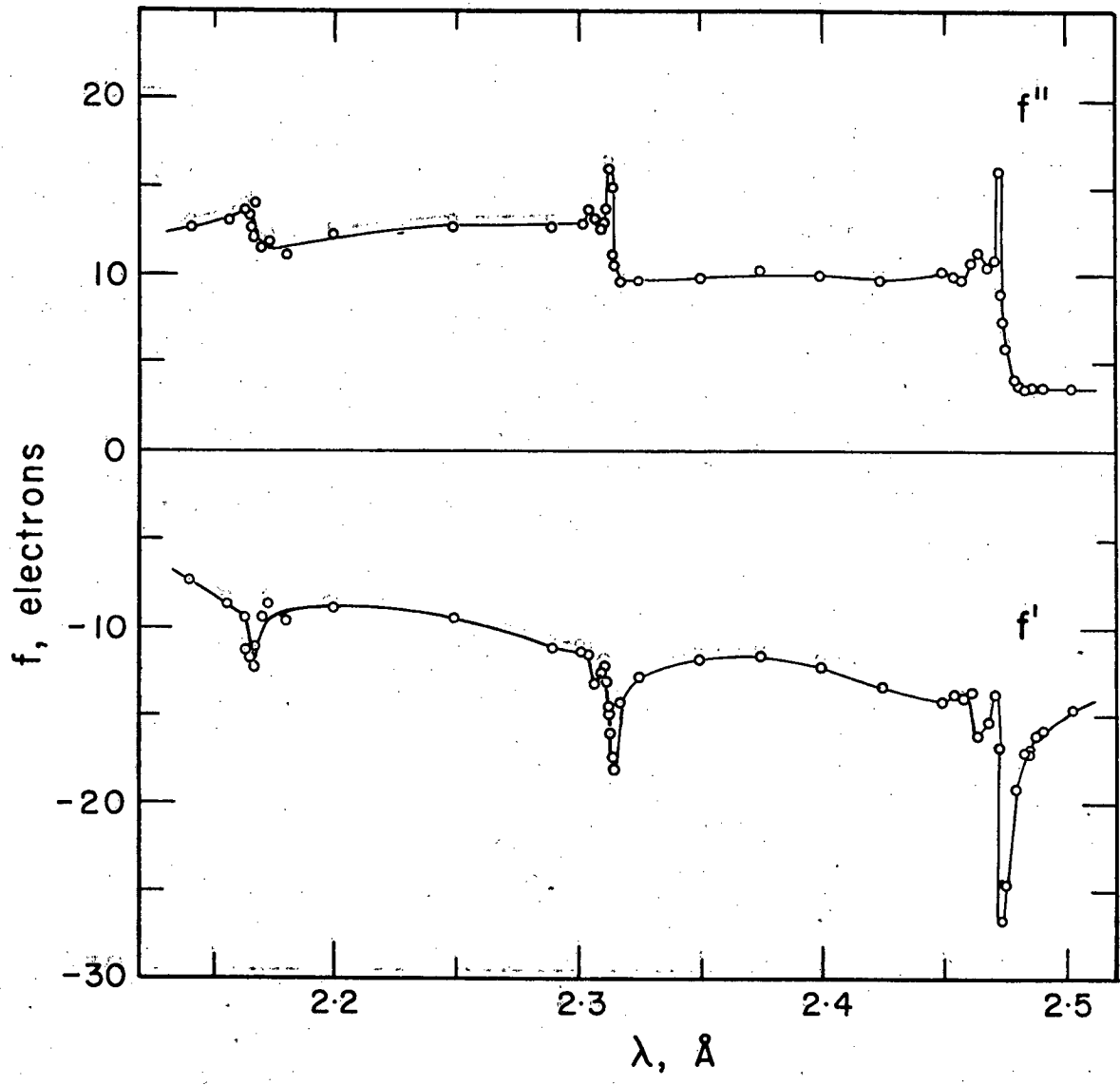
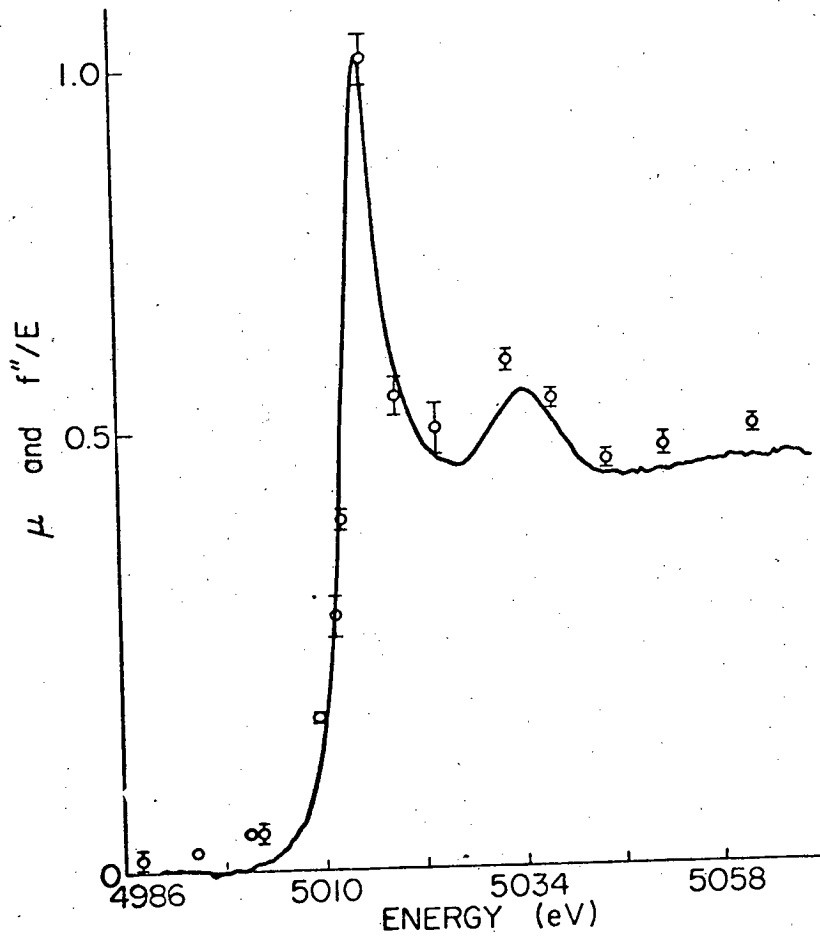


Fig. 1

XBL 799-11274



XBL 794-9137

Fig. 2

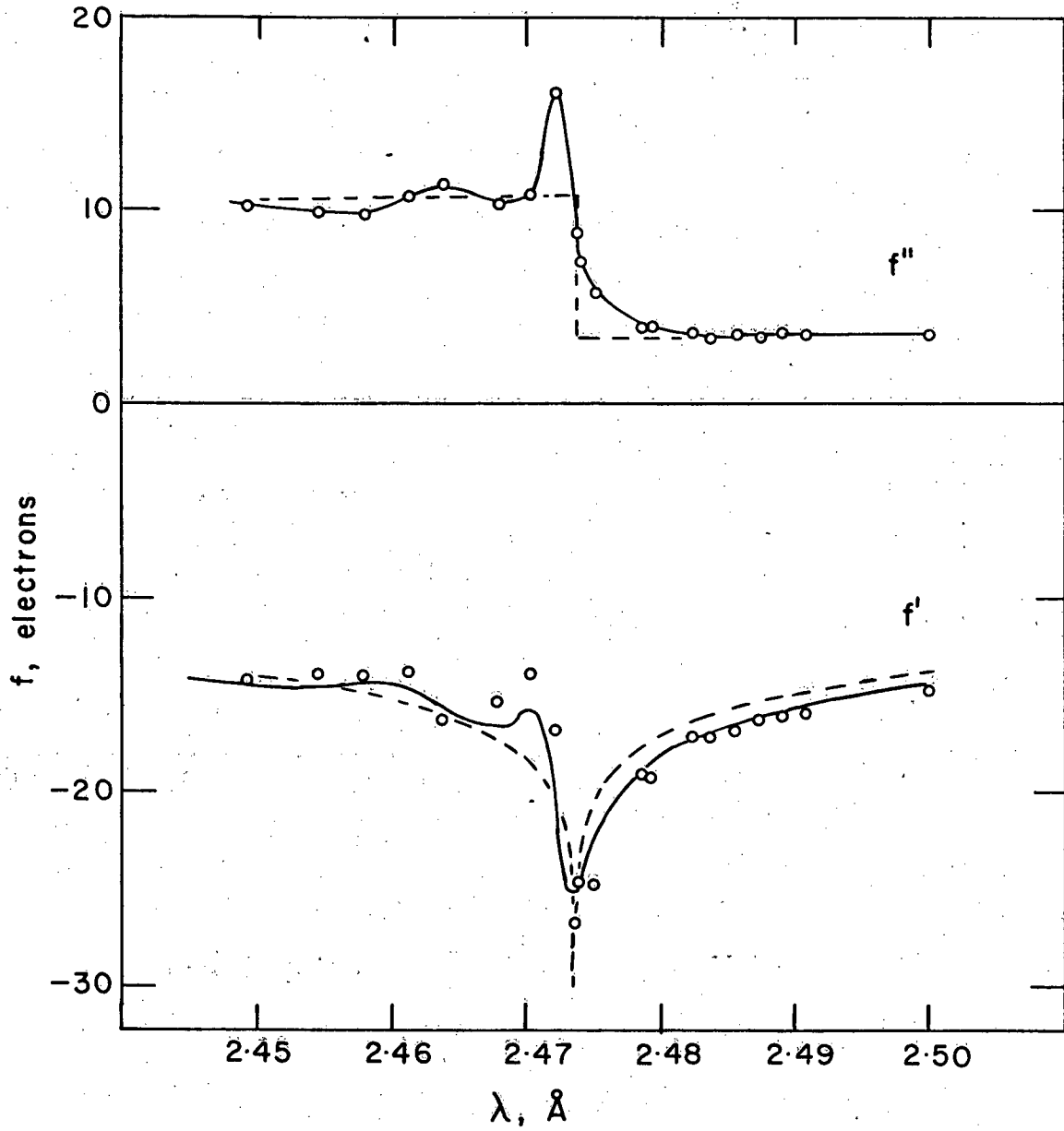
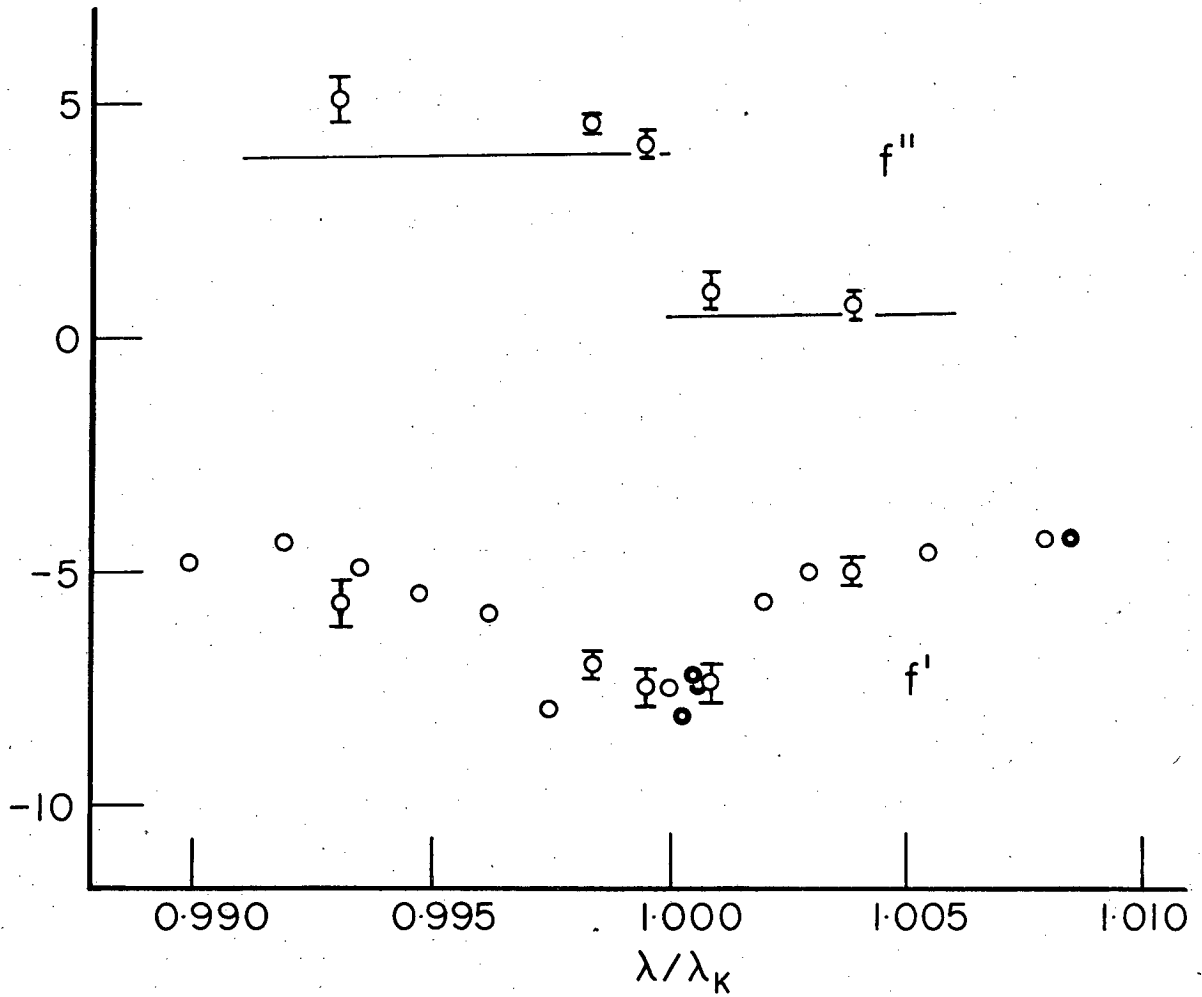


Fig. 3



- $\bar{\circ}$  Templeton, Templeton, Phillips & Hodgson, Co
- Freund, Cu
- Bonse & Materlik, Ni
- Cromer & Liberman

Fig. 4



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