

notation has the following utility. The irreducible representation with even and odd behaviour under various group elements refers to the difference structure  $C_1$  or  $C_2$ . Now the average structure is of course invariant under all group elements, so that when we add it to  $C_1$  or  $C_2$  to give the actual structure of the material at  $OO'$  etc. or  $NN'$  etc. in Fig. 1, the group elements with character  $-1$  for  $C_1$  or  $C_2$  are lost. Our subgroup symbol therefore remains as the space group of the material in the pure component form. This will remain unchanged if at lower temperatures the modulation wave squares up and we move out of the realm of sinusoidal modulation described by an irreducible representation.

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## An Experimental Determination of the Anomalous Dispersion Factor $f''$ for Sulfur

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#### Abstract

The anomalous dispersion factor,  $f''$ , of sulfur for Cu  $K\alpha$  radiation has been determined to be 0.60 (3) using measurements of Bijvoet differences of 175 selected reflections for a 6-(4-nitrobenzyl)thioinosine crystal.

#### Introduction

During the course of our study on the structure and absolute configuration of 6-(4-nitrobenzyl)-

thioinosine, a potent inhibitor of nucleoside transport, we discovered that crystals of this compound exhibited pronounced Bijvoet differences ( $\Delta I = I_H - I_{\bar{H}}$ ) for Cu  $K\alpha$  radiation. From the measured values of selected Bijvoet differences and using the method reported earlier by one of us (Parthasarathy, 1962), we have determined experimentally the absorption part of the anomalous scattering factor  $f''$  for the sulfur atom.

Direct experimental determinations of  $f''$  have been carried out only for a few atoms (see Table 1) by careful measurements of selected Bijvoet differences. The availability of synchrotron radiation of tunable wavelengths has led to accurate determinations of  $f''$  (Phillips, Templeton, Templeton & Hodgson, 1978).

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Table 1. *Experimental  $f''$  values*

Element	Radiation	$f''$	$f''$ calc.	Reference
C	Cu	0.0122 (7)	0.009	(a)
O	Cu	0.028 (5)	0.032	(b)
		0.037 (2)		(a)
		0.041 (4)†		(c)
Al	Cu	0.22 (2)	0.246	(b)
Si	Cu	0.31 (1)	0.330	(d)
		0.34 (2)		(e)
S	Cu	0.60 (3)	0.557	(f)
Cl	Cu	0.67 (5)	0.702	(g)
		0.72 (2)†		(h)
	Mo	0.155 (10)†	0.159	(i)
Co	Cu	3.59 (9)	3.608	(j)
		3.92 (2)†		(h)
	Mo	0.87 (3)	0.973	(j)
		0.962 (10)†		(i)
Ga	Cu	0.89 (3)	0.777	(k)
	Mo	1.68 (2)	1.609	(l)
Se	Mo	2.19 (3)†	2.223	(m)
Br	Mo	2.47 (10)	2.456	(j)
I	Cu	6.3 (3)‡	6.835	(n)
		6.0 (3)§		
Cs	Mo	2.22 (6)†	2.119	(o)

References: (a) Engel (1972); (b) Marezio (1965b); (c) Hope & De la Camp (1972); (d) Zachariassen (1965); (e) recalculated by Engel (1972); (f) present work; (g) Parthasarathy (1962); (h) Templeton, Zalkin, Ruben & Templeton (1979); (i) see (h); (j) Engel & Sturm (1975); (k) Marezio (1965a); (l) Marezio, Tranqui & Capponi (1975); (m) Freeman, Mair & Barnea (1977); (n) Hall & Maslen (1966); (o) Templeton & Templeton (1978).

\* From *International Tables for X-ray Crystallography* (1974).

† Determined by least-squares.

‡  $\sin \theta/\lambda = 0.5 \text{ \AA}^{-1}$ .

§  $\sin \theta/\lambda = 0.5 \text{ \AA}^{-1}$ .

### Experimental

Single crystals of 6-(4-nitrobenzyl)thioinosine were obtained by slow evaporation from ethyl alcohol solution. The crystal ( $C_{17}H_{17}N_5O_6S$ ) is monoclinic, space group  $P2_1$  with cell constants  $a = 14.718$  (1),  $b = 8.678$  (1),  $c = 7.269$  (1) Å,  $\beta = 93.55$  (1)°,  $V = 926.6$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.49$ ,  $D_x = 1.503$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 19.3$  cm<sup>-1</sup>,  $T = 294$  K,  $F(000) = 436$ . Using 2205 reflections  $I \geq 2\sigma(I)$  to  $2\theta \leq 164^\circ$ , the crystal structure was determined and refined to  $R = 0.042$  (Soriano-Garcia & Parthasarathy, 1974; Soriano-Garcia, Parthasarathy, Paul & Paterson, unpublished results).

Using a GEXRD-3 diffractometer equipped with Ross filters and a full  $\omega$  circle, the integrated intensities for a set of  $hkl$ ,  $\bar{h}\bar{k}\bar{l}$  and  $h\bar{k}l$ ,  $\bar{h}k\bar{l}$  reflections were collected. The crystal used had dimensions  $0.23 \times 0.36 \times 0.18$  mm. The four symmetry-related reflections of this crystal were measured in succession at room temperature using Cu  $K\alpha$  radiation and manual mode. The integrated intensities were measured by an  $\omega-2\theta$  step-scan procedure. The steps were equally spaced  $\omega$  angles of  $0.05^\circ$ , ranging up to  $1^\circ$ . For most reflections, the peak was contained within  $0.5^\circ$ . During the scan procedure, balanced filters were used to allow for background. For each step, the counts were measured for a constant time of 10 s. The numerical integration for obtaining the integrated intensity was carried out on a computer using the trapezoidal method.

Table 2. *Distribution of Bijvoet ratios*

Number of reflections	Range of Bijvoet ratios (%)
106	$10 < \Delta I/I < 20$
58	$20 < \Delta I/I < 30$
28	$30 < \Delta I/I < 40$
18	$40 < \Delta I/I < 50$
8	$50 < \Delta I/I < 60$
3	$60 < \Delta I/I < 70$
19	$70 < \Delta I/I < 190$

Table 3. *A sample of reflections showing large calculated and measured Bijvoet ratios and the corresponding  $f''_{\text{eff}}$  for selected reflections*

$h k l$	$(\Delta I/I)_{\text{th}}(\%)$	$(\Delta I/I)_{\text{exp}}(\%)$	$f''_{\text{eff}}$
0 2 0	174.75	153.14	0.49
-2 3 5	-64.46	-66.46	0.57
2 6 1	73.06	72.46	0.55
-3 2 3	-54.78	-55.14	0.56
-3 2 4	-63.20	-64.01	0.56
-4 1 5	57.37	52.78	0.51
-6 1 0	55.40	49.37	0.50
8 2 1	-53.17	-54.29	0.57
10 1 3	-77.47	-66.43	0.48
13 3 4	53.64	52.26	0.54

We measured 240 sets of  $(\Delta I/I)$  that were expected to give values greater than 0.1; in most cases  $(\Delta I/I)$  was greater than 0.20. Under these experimental conditions, the integrated intensities for the four symmetry-related reflections were measured and the  $(\Delta I/I)$  values for the direct and inverse reflections were evaluated as an average of two values.

Detailed absorption corrections, calculated by an analytical integration (Coppens, 1970) were applied. For this correction, the crystal shape was defined by eight plane faces. The transmission coefficient for reflections ranged from 0.527 to 0.616.

### Determination of $f''$

The procedure and other precautions involved in the determination of  $f''$  have been outlined earlier (Parthasarathy, 1962; Engel, 1972). Table 2 shows the distribution of the Bijvoet ratios and it can be seen that few reflections have  $(\Delta I/I) > 0.6$ .

The calculated residuals for reflections related by the twofold symmetry and for reflections related by the center of inversion are, respectively, 0.024 and 0.203. These values clearly show that systematic errors are minimal and the enantiomer-sensitive reflections show significant deviation from equivalence.

Assuming that  $f''$  for lighter atoms may be neglected, it can be shown that (Parthasarathy, 1962):

$$(f''_{\text{S}})_{\text{eff}} = (f''_{\text{S}})_{\text{th}}[(\Delta I/I)_{\text{exp}}/(\Delta I/I)_{\text{th}}]. \quad (1)$$

This method measures the deviations of  $f''_{\text{S}}$  from its theoretical expected value (Engel, 1972). The measured values of the Bijvoet ratios are calculated using the expression

$$(\Delta I/I)_{\text{exp}} = [(I_{hkl} - I_{\bar{h}\bar{k}\bar{l}})]/0.5 [(I_{hkl} + I_{\bar{h}\bar{k}\bar{l}})]. \quad (2)$$

and  $(\Delta I/I)_{\text{th}}$  can be readily calculated from the structure-factor expressions. The values of  $(\Delta I/I)_{\text{th}}$  and

$(\Delta I/I)_{\text{exp}}$  are listed in Table 3.\* A comparison of calculated and measured Bijvoet ratios show good agreement, both in their signs and magnitudes. These reflections were chosen based on the selection criteria used by Parthasarathy (1962).

Using the value of 0.557 for  $f''_{\text{S}}$  obtained from *International Tables for X-ray Crystallography* (1974), the values of  $(f''_{\text{S}})_{\text{eff}}$  were calculated from (1). The values so obtained are listed in Table 3; the average value of  $f''_{\text{S}}$  is 0.58 (3). Using (7) of Engel (1972), we also calculated  $f''_{\text{exp}}$  from the value of the effective  $f''_{\text{eff}}$  of S with respect to the light atoms in cell; it was found to be 0.60 (3).

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\* A full table of reflections and  $(\Delta I/I)_{\text{th}}$ ,  $(\Delta I/I)_{\text{exp}}$  and  $f''_{\text{eff}}$  values has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39263 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### Erratum

*Acta Cryst.* (1984). **A40**, 484

**Graphic representation and nomenclature of the four-dimensional crystal classes. II. The individual symmetry operations: erratum.** By E. J. W. WHITTAKER, *Department of Geology and Mineralogy, Oxford University, Parks Road, Oxford OX1 3PR, England.*

(Received 5 March 1984)

#### Abstract

Parentheses have been incorrectly placed in the first equation of page 62 of Whittaker [*Acta Cryst.* (1984), **A40**, 58–66]. The right-hand side of the equation should consist of two  $4 \times 4$  matrices. The correct equation is given.

#### Reference

WHITTAKER, E. J. W. (1984). *Acta Cryst.* **A40**, 58–66.

The first equation on page 62 of Whittaker (1984) should be:

$$\begin{pmatrix} 0 & \bar{1} & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 \\ 0 & 0 & \bar{1} & 0 \end{pmatrix} = \begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 1 \\ 0 & 0 & \bar{1} & 0 \end{pmatrix} \begin{pmatrix} 0 & \bar{1} & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}.$$

### International Union of Crystallography

*Acta Cryst.* (1984). **A40**, 484

#### Commission on Journals Changes in Editorial Boards

There have been several recent changes in the Editorial Boards of the IUCr journals and there will be further changes later in 1984.

Professor G. A. Jeffrey completed his term of office in September 1983 and Professor D. H. Templeton has resigned on his election as President of the American Crystal-

lographic Association. Their successors are Professor James A. Ibers, Professor C. E. Nordman and Professor H. Steinfink. In addition, Professor J. Drenth and Professor C. E. Bugg have been appointed as the Netherlands and US Co-editors of *Acta Crystallographica*.

Professor M. Hart and Dr F. R. Ahmed complete their terms of office as *Editor of Journal of Applied Crystallography* and *Co-editor of Acta Crystallographica*, respectively, at the XIIth Congress of Crystallography. The names of their successors will be announced in due course.