SHORT COMMUNICATIONS

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The determination of absolute structure. IV. Some comments on the publication of non-centrosymmetric structures.*

By PETER G. JONES, *Institut für Anorganische Chemie der Universität, Tammannstrasse* 4, 3400 *Göttingen, Federal Republic of Germany*

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Abstract

A survey of 106 non-centrosymmetric structures, all containing moderate to strong anomalous scatterers, from *Acta Crystallographica*, Section C, 1984, shows that the attempt to assign an absolute structure is mentioned in only 42 cases. The recent recommendations of Hamor, Steinfink & Willis [*Acta Cryst.* (1985). C**41**, 301–303] for the publication of non-centrosymmetric structures are discussed and an improved formulation suggested.

Introduction

Since the pioneering work of Bijvoet it has been clear that analysis of the (generally small) anomalous-dispersion effects in non-centrosymmetric crystal structures (henceforth n.c.s.) can lead to a determination of the absolute structure (Jones, 1984*a*); this method has been widely used to determine absolute configurations. It is apparently less clear that the presence of anomalous scatterers in n.c.s. *obliges* the crystallographer to analyse the anomalous-dispersion effects in order to avoid the possibility of systematic errors in atom coordinates (Cruickshank & McDonald, 1967). This communication presents a brief survey of n.c.s. reported in *Acta Crystallographica*, Volume C**40**.

Discussion

Of the 237 n.c.s. reported, 106 form the set discussed here. These have been selected according to the following criteria: (i) X-ray structures only; (ii) no pseudosymmetry, severe disorder or twinning; (iii) no structures that are apparently centrosymmetric (Jones, 1986*b*,*c*; Bocelli, Grenier-Loustalot & Marsh, 1986); (iv) only structures containing atoms with $f'' > 0{\cdot}09$ (*i.e.* P or heavier for Mo $K\alpha$ radiation).

In view of recent criticisms of reports of n.c.s. (Rogers & Allen, 1979; Jones, 1984*b*) it is unsurprising that only 42 structure reports make any reference to an attempt to assign an absolute structure (irrespective of the success or validity of the methods used). The remaining 64 structures involve anomalous scatterers ranging from P (f'' 0·095 for Mo $K\alpha$) to Bi (f'' 10·6 for Mo $K\alpha$). Since it is often possible to determine an absolute structure based on the anomalous dispersion of one P, S or Cl atom ($f'' < 0{\cdot}2$ for Mo $K\alpha$; Jones, 1984*a*), it is disappointing that no attempt was made in 17 such cases.

How serious is the neglect of anomalous dispersion? For those 21 structures lacking a polar axis, it is unlikely that serious systematic errors will be present. However, any of the other 43 structures may contain moderate to severe axial polar-dispersion errors (Cruickshank & McDonald, 1967; Flack, 1985) if the wrong absolute structure has been assumed. As a general rule, one can draw attention to the words of Flack (1985) and 'emphasize the importance of testing the absolute structure in any n.c.s., whether or not one is interested in the crystal chirality or polarity . . .'. In this respect, it is disturbing to note that the latest Checklist for Authors (1985) in *Acta Crystallographica* makes no clear reference to, or recommendations for, the determination (and subsequent reporting) of n.c.s. by the analysis of anomalous-dispersion effects; it merely refers to the use of 'independent physical measurements', 'independent' presumably implying non-X-ray methods.†

An attempt to remedy this omission has been made by Hamor, Steinfink & Willis (1985) (henceforth HSW). HSW recommend that reports of 'chirality' (*i.e.* absolute structure) should be supported by presentation of Bijvoet-pair data, but do not mention the methods of Flack (1983) or Rogers (1981) – presumably because these are not yet widely available as part of program systems. HSW also refer to the determination of the directions of polar axes and to the corresponding definition in *International Tables for Crystallography* (1983). This is likely to cause confusion for two reasons: first, the common conception of a polar axis as one along which the origin may be fixed arbitrarily (as assumed above for the discussion of axial polar-dispersion errors) is not consistent with the above definition (*e.g.* the x and z directions in crystal class m are not defined polar, yet the axes [100], [010] and [$1\bar{1}0$] in class $\bar{6}2m$ are) and secondly, it is not made clear that there are non-centrosymmetric yet achiral crystal classes with no polar axes (*e.g.* $\bar{4}2m$; Jones, 1984*a*, 1986*a*).

It seems that HSW have failed to address the main problem, namely that many n.c.s. are published with no

* Part III: Jones (1986*a*).

† *Editorial note.* See Commission on Journals announcement in *Acta Cryst.* (1985). C**41**, 1280.

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mention of an attempt to determine an absolute structure; this will mean that, in at least some cases, no such attempt has been made and that the structure may contain systematic errors. A simple general recommendation for reports of n.c.s. could be formulated as follows: Any report of an n.c.s. should state *either* the methods used to assign an absolute structure and the results thus obtained *or* that the anomalous-dispersion effects were too small to allow such an assignment, with evidence for this statement in the case of structures containing atoms heavier than (say) Si.

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Acta Cryst. (1986). C**42**, 925

Structure of bis(triphenylphosphine)iminium bis(cyano)(phthalocyaninato)ferrate(III), $[N\{P(C_6H_5)_3\}_2][Fe(CN)_2(C_{32}H_{16}N_8)]$, at 180 K: erratum.

By H. KÜPPERS, *Mineralogisches Institut der Universität, Olshausenstrasse* 40, *D*-2300 *Kiel* 1, *Federal Republic of Germany* and W. KALZ and H. HOMBORG, *Institut für Anorganische Chemie der Universität, Olshausenstrasse* 40, *D*-2300 *Kiel* 1, *Federal Republic of Germany*

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Abstract

Fig. 2 printed in the paper by Küppers, Kalz & Homborg [*Acta Cryst.* (1985), C**41**, 1420–1423] on the structure of $[PNP][Fe^{III}(CN)_2Pc]$ is in error. The anion presented there belongs to the structure of the corresponding Fe^{II} compound, $[PNP]_2[Fe^{II}(CN)_2Pc].CH_2Cl_2$, which was subsequently published in *Z. Naturforsch. Teil B* (1986), **41**, 44–47. The correct figure is given.

An *ORTEP* (Johnson, 1976) drawing of the $[Fe^{III}(CN)_2Pc]^-$ anion showing the bond lengths and angles is given in Fig. 1.

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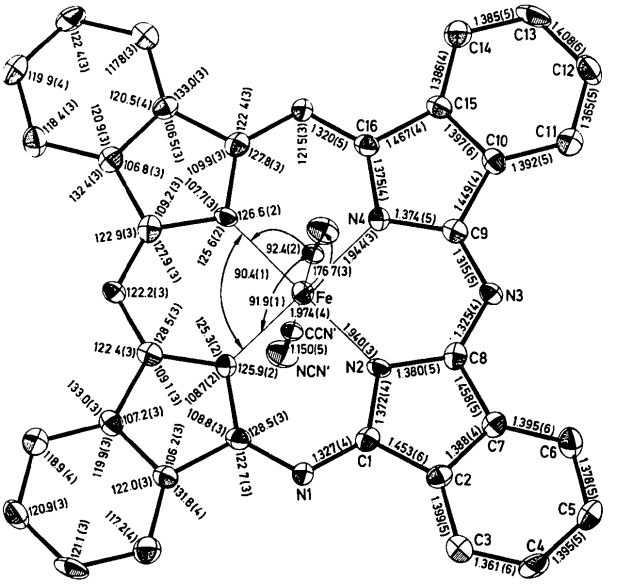

Fig. 1. *ORTEP* (Johnson, 1976) plot of the $[Fe^{III}(CN)_2Pc]^-$ anion with bond lengths (Å) (right part of the centrosymmetrical anion) and angles (°) (left part). Thermal ellipsoids are at the 50% probability level.

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