SHORT COMMUNICATIONS

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The perils of *Cc* revisited

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Abstract

The space groups of 98 structures originally reported in *Cc* are revised. In 75 cases the revised space group is *C*2/*c* and the revision entails adding a center of symmetry, usually leading to large changes in bond lengths and angles. In the remaining 23 cases, where the revised space group is *Fdd*2, *R*3*c* or (in one case) $I\bar{4}c2$, the lattice type is changed but no center is added; in these cases the molecular dimensions are effectively unchanged.

1. Introduction

About 5 years ago, Baur & Kassner (1992) compiled a list of 221 structures that had originally been described in space groups of unnecessarily low symmetry. They noted that space group *Cc* had a disproportionately large representation in the list and selected, as the running title of their compilation, 'The Perils of *Cc*'; they concluded that 'possibly over 10% of all crystal structures reported in *Cc* should actually be described in a higher true symmetry'. I have now carried out a survey of the October 1995 release of the Cambridge Structural Database (1992; CSD), noting all structures reported in space group number 9 (for which the standard setting is *Cc*, but other settings such as *Bb* and *Ia* are also included). Of the approximately 1200 valid entries, I have identified 116 which, almost surely, should be described in higher symmetries (18 of these entries have previously been noted) – very close to the 10% figure predicted by Baur & Kassner (1992). The 98 new (as far as I am aware) examples of structures that have been mis-identified as belonging to *Cc* are listed in Table 1.

2. Experimental

Unit-cell dimensions and atom coordinates were recovered from the CSD and scrutinized for possible higher symmetry. This scrutiny was carried out by personal inspection rather than *via* a computer program such as *MISSYM* (Le Page, 1988) since, in several cases, matching of related atoms was obscured by disorder, by atoms (usually hydrogen) being missing, by methyl groups of related molecules having been assigned different conformations or by an occasional misprint. Coordinates of the symmetry-related (in the revised space group) atoms were then averaged and the fit to the higher symmetry was evaluated. In almost all cases the original publication was also examined and evaluated. From these evaluations I became convinced that the 98 entries in Table 1 should properly be re-formulated in the space groups noted.* In a majority of cases the revised space group is *C*2/*c*. (I have always cast it in this standard setting, no matter what setting of space group number 9 was originally specified.) Thus, a center of symmetry has been added and one must fear that the original *Cc* refinement suffered from the severe near-singularity problems that exist whenever an approximately centrosymmetric structure is described in a noncentrosymmetric space group (Ermer & Dunitz, 1970; Schomaker & Marsh, 1979). This seems to have been almost invariably the case, the matching of symmetry-related coordinates being far worse than one might expect from any reasonable e.s.d.'s. As a result, in compounds containing phenyl rings, for example, it was a typical situation that C—C distances in the *Cc* description varied from, perhaps, 1.25 to 1.55 Å. (These peculiar distances were seldom mentioned in the original papers.) In the revised *C*2/*c* structures, the distances were normal with only one exception (CHXARH; Table 1). In many cases the original authors noted difficulties in refinement, often being forced to constrain the bond lengths and angles to reasonable values.

Of the 98 entries in Table 1, 79 represent cases in which the C-centered monoclinic unit cell has $Z = 4$, *i.e.* where the asymmetric unit for the original *Cc* description contains a single formula unit (typically a discrete molecule). Accordingly, describing these structures in *C*2/*c* – as occurs in 65 of the 79 cases – requires that the molecules contain an internal symmetry element, either a C_2 axis or a center of inversion; almost invariably it is a C_2 axis. In the other 14 cases with $Z = 4$ the change in space group is to either *Fdd*2 or *R*3*c*; in *Fdd*2 the molecule must again possess a C_2 axis and in *R*3*c* a C_3 axis. Of the 19 examples with $Z > 4$, molecular symmetry is required in only three cases – CTPIRA, where two independent molecules lie on C_3 axes, JISKEB, where the molecule lies on a C_2 axis, and BIHHEF, where one of the three independent malonic acid molecules lies on a C_2 axis.

3. Discussion

I believe there are three reasons why *Cc* might be a special candidate for incorrect space-group assignment. First, any structure reported in *Cc* must be achiral and the addition of a center of symmetry (so as to create *C*2/*c*) does not affect the chirality of the system. In some other cases where the choice between a noncentrosymmetric and a centrosymmetric space group is not dictated by systematic absences – such as the pairs *P*1 and $P\bar{1}$, $P2_1$ and $P2_1/m$, and *C*2 and *C*2/*m* – adding a center of symmetry would remove the chirality and perhaps make the correct choice obvious. Second, the centrosymmetric space group *C*2/*c* can be generated from *Cc* by adding a C_2 axis, a more common (and, perhaps, less easily recognized) symmetry element than the mirror plane that is necessary to take, for

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* A lists of revised coodinates has been deposited with the IUCr (Reference: AN0533). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Structures originally described in space group Cc which are better described in higher symmetries*

Included are the 'reference codes' assigned by the Cambridge Structural Database (1992), the revised space group, the number of formula units per cell (*Z*) and the formula unit.

Refcode	Space group	*Z*	Formula unit	Reference	Note
AMESNC	*C*2/*c*	4	$C_6H_{12}N_2O_2Cl_2Sn$	Harrison, King & Healy (1979)	
BIHHEF	*C*2/*c*	20	$C_3H_4O_4KF$	Emsley, Jones & Kuroda (1982)	
BORGUK	*C*2/*c*	4	$C_{42}H_{64}N_{10}O_8Co_2.2H_2O$	Robinson, Flohr, Kempe, Pannhorst & Rétey (1983)	(*a*)
BRMENP10	*C*2/*c*	4	$C_{20}H_{24}Br_4$	Oku, Harada, Yagi & Shirahase (1983)	
BUVYIA	*Fdd*2	16	$C_{16}H_{13}N_2O_4Cl_3S.H_2O$	Fletton, Humber, Roberts, Owston & Henrick (1983)	
CAGROR	*Fdd*2	8	$C_{36}H_{46}N_6O_2Ni.2PF_6$	Morrison, Thompson & Trotter (1983)	
CASWOI10	*C*2/*c*	4	$C_{40}H_{30}NO_5P_2F_6Rh$	Dobson, Moore, Robinson, Galas & Hursthouse (1985)	(*b*)
CHXARH	*C*2/*c*	4	$C_{14}H_{21}N_2O_8Cl_2Rh.2H_2O$	Filippova, Polynova, Il'inskii, Porai-Koshits & Ezerskaya (1981)	(*c*)
CIVLIC10	*Fdd*2	16	$C_{11}H_{14}O_6$	Irngartinger, Reimann, Garner & Dowd (1988)	
CLTPOC	*Fdd*2	8	$C_{36}H_{30}O_2Cl_2P_2Co$	Mangion, Smith & Shore (1976)	
CPOECU10	*C*2/*c*	8	$C_{16}H_{21}NOCl_3CuP$	Newton, Caughman & Taylor (1974)	(*d*)
CTPIRA	*R*3*c*	12	$C_{24}H_{33}Cl_3P_3Ir$	Robertson & Tucker (1981)	
CUACPY	*C*2/*c*	4	$C_{14}H_{16}N_2O_4Cu.C_5H_5N$	Anzenhofer & ten Rouwelaar (1967)	(*e*)
CUWSES	*C*2/*c*	4	$C_{34}H_{20}O_{11}P_2Cr_2$	Zeiher, Mohyla, Lorenz & Hiller (1985)	
DAWDIO	*C*2/*c*	4	$C_{32}H_{72}Si_4$	Matsumoto, Takatsuna, Minemura, Nagai & Goto (1985)	
DAWWED	*C*2/*c*	4	$C_2H_5Cl_5Sn.2(C_4H_{12}N)$	Paseshnitchenko, Aslanov, Jatsenko & Medvedev (1985)	(*f*)
DAWWIH	*C*2/*c*	4	$C_4H_9Br_5Sn.2(C_4H_{12}N)$	Paseshnitchenko *et al.* (1985)	(*g*)
DELWEW	*Fdd*2	8	$C_{17}H_{18}N_4O_2P_2$	Neidlein, Degener, Gieren, Weber & Hubner (1985)	
DERKOA10	*C*2/*c*	4	$C_4H_8S_{10}Mo_2Fe.3(C_8H_{20}N)$	Zhigui, Chunxiao, Yuguo & Fengshan (1986)	(*h*)
DESZUW	*R*3*c*	6	$C_{12}H_9O_3PSe$	Allen, Nowell & Taylor (1985)	
DIKWAV	*C*2/*c*	4	$C_{20}H_{36}N_4Cl_2Pd_2$	Yamamoto & Yamazaki (1985)	
DIOXGC	*C*2/*c*	4	$C_4H_8O_2Cl_2Ge$	Kulishov *et al.* (1970)	
DIRVEF	*C*2/*c*	4	$C_{32}H_{46}S_6Cr_4Mn$	Pasynskii, Eremenko, Gasanov, Struchkov & Shklover (1984)	
DIRVIJ	*C*2/*c*	4	$C_{32}H_{46}S_6Cr_4Fe$	Pasynskii *et al.* (1984)	
DISKEV	*C*2/*c*	4	$C_{20}H_{38}N_4O_7$	Dvorkin *et al.* (1985)	(*i*)
DOHVOL	*C*2/*c*	4	$C_{40}H_{88}Si_4$	Matsumoto, Minemura, Takatsuna, Nagai & Goto (1985)	
DOTJIF	*C*2/*c*	4	$C_{15}H_{11}O_2Cl_4Sb$	Gérard, Lucken & Bernardinelli (1986)	
DUNPEH	*C*2/*c*	4	$C_{40}H_{74}O_8P_4Rh_6$	Arif, Heaton & Jones (1986)	
DUYGAF	*R*3*c*	6	$C_{21}H_{15}.C_{42}H_{30}Br_3Ni_2$	Cecconi, Ghilardi, Midollini, Moneti & Orlandini (1986)	
FAWBAG	*C*2/*c*	4	$C_{36}H_{36}N_6O_4Cu$	Ishida, Hatta, Yamashita, Doi & Inoue (1986)	(*j*)
FIXWIS	*Fdd*2	8	$C_{16}H_{16}O_2$	Cosmo, Hambley & Sternhell (1987)	
FUZYII	*C*2/*c*	4	$C_{19}H_{47}N_3O_7S_{19}Cu_6Mo_6$	Xintao, Bing, Yifan & Jiaxi (1988)	(*k*)
GEGXEV	*C*2/*c*	8	$C_{20}H_{32}O_5$	Schultz, Macielag, Podhorez, Suhadolnik & Kullnig (1988)	
GEJMAJ	*C*2/*c*	4	$C_{32}H_{38}O_{14}V_2.C_7H_8$	Lee, Nakanishi, Chiang, Frankel & Spartalian (1988)	(*l*)
HAGTIS	*C*2/*c*	4	$C_{12}H_{12}O_{14}BCs$	Zviedre, Fundamensky & Schwartz (1992)	
HALJUZ	*C*2/*c*	4	$C_{15}H_{23}NO_2$	de Ridder, Schenk & Döpp	(*m*)
HAXJIZ	*C*2/*c*	4	$C_{18}H_{22}N_4O_2S_2Co.2NO_3.H_2O$	Carballo & Castiñeiras (1983)	
INOLPT	*C*2/*c*	8	$C_{54}H_{38}O_6P_2Pt$	Hunt, Kemmitt, Russell & Tucker (1979)	
JABJOL	*C*2/*c*	4	$C_{20}H_{38}N_2O_4Co$	Colborn, Garbauskas & Hejna (1988)	
JAHRAL	*C*2/*c*	4	$C_{20}H_{12}N_8Ag.NO_3$	Nasielski, Nasielski-Hinkens, Heliporn, Rypens & Declercq (1988)	
JANNOB	*C*2/*c*	4	$C_{40}H_{36}N_2OP_2ClRh$	Cooper *et al.* (1989)	(*n*)
JEGGIL	*C*2/*c*	4	$C_9H_{16}O_5Si_2$	de Ruiter, Benson, Jacobson & Verkade (1990)	
JEVTOT	*C*2/*c*	4	$C_{14}H_{18}N_8O_4S_2Cu_2.2NO_3.H_2O$	Qin *et al.* (1989)	
JISKEB	*I*$\bar{4}$*c*2	8	$C_9H_{29}O_2Sn_3I$	Perevalova *et al.* (1990)	
JOGVUW	*Fdd*2	16	$C_{10}H_{14}N_2O_9V.NH_4.2.5H_2O$	Shimoi, Saito & Ogino (1991)	(*d*)
JOHBUD	*C*2/*c*	4	$C_5H_6N_2O_4$	Wade, Kondracki & Carroll (1991)	
JUXFUD	*C*2/*c*	4	$C_{32}H_{39}N_3O_4Sr$	Mösges, Hampel, Kaupp & von Ragué Schleyer (1992)	
KAXYOX	*C*2/*c*	4	$C_7H_{10}NS_2F_6Cu$	Willert-Porada, Burton & Baenziger (1989)	
KEPGIV	*Fdd*2	8	$C_{32}H_{48}O_{14}Na_2I_2$	Lu *et al.* (1990)	
KEVWUD	*R*3*c*	6	$C_{24}H_{67}N_{16}Si_4.3N_3$	Woning, Daniels & Verkade (1990)	
KEYKUU	*C*2/*c*	4	$C_{44}H_{37}OP_2ClRu$	Rickard, Roper, Taylor, Waters & Wright (1990)	(*o*)
KEZGOL	*C*2/*c*	8	$C_{44}H_{80}Si_5$	Kabe *et al.* (1990)	
KIBZAW	*C*2/*c*	4	$C_{12}H_{30}N_6O_5W_2.S_2O_6.4H_2O$	Schreiber, Wieghardt, Nuber & Weiss (1990)	
KIDTIA	*C*2/*c*	4	$C_{24}H_{20}O_{12}Rh_6$	Hou, Wakatsuki & Yamazaki (1990)	
KIFKIT	*C*2/*c*	8	$C_{25}H_{26}N_9S_3Y$	Bombieri *et al.* (1989)	
KIFKOZ	*C*2/*c*	8	$C_{25}H_{26}N_9S_3Eu$	Bombieri *et al.* (1989)	
KIMSUU	*Fdd*2	8	$C_{15}H_{14}N_2$	Zyss, Ledoux, Bertault & Toupet (1991)	
KIXCAV	*C*2/*c*	4	$C_{37}H_{31}O_2P_2Ag$	Lan-Sung, Hua-Hui & Qian-Er (1991)	(*p*)
KIYPIR	*C*2/*c*	4	$C_{36}H_{28}F_{12}S_2Pt$	Debaerdemaeker, Roth & Brune (1991)	
KIZKIN	*Fdd*2	8	$C_{26}H_{38}ClSi_2Ti_2As$	Fenske, Grissinger, Hey-Hawkins & Magull (1991)	

Table 1 (*cont.*)

Refcode	Space group	*Z*	Formula unit	Reference	Note
KOWDAB	*C*2/*c*	4	$C_{14}H_{18}N_4O_4Ni.2(ClO_4)$	Zhanfeng, Guoxiong, Ling, Fengshan & Yuguo (1990)	
KUBRAA	*C*2/*c*	4	$C_{20}H_{32}N_2O_4$	Krakowiak *et al.* (1992)	
LELYEG	*C*2/*c*	4	$C_2N_2S_2ClHg.2(C_{10}H_8S_8)$	Konovalikhin *et al.* (1992)	
LEWZAO	*C*2/*c*	4	$C_{16}H_{28}O_4Ni$	Walther, Schmidt, Klettke, Imhof & Görls (1994)	
LEZTAL	*C*2/*c*	8	$C_{28}H_{35}OPSi_2Ru$	Tobita, Wada, Ueno & Ogino (1994)	
NONPIN	*C*2/*c*	4	$C_{18}H_{39}O_2P$	Bello (1973)	
NPHLCU	*C*2/*c*	4	$C_{28}H_{24}N_5O_3Cu.C_4HO_4Cl_6$	van Meerssche, Germain, Declercq & Wilputte-Steinert (1981)	
OCYAXM	*C*2/*c*	8	$C_{25}H_{28}O_4$	Precigoux, Busetta, Courseille, Hospital & Miquel (1978)	
OFAFSB10	*C*2/*c*	4	$C_4F_{12}O_5Sb_2$	Bullivant, Dove & Haley (1980)	
OXIBZN10	*C*2/*c*	4	$C_{12}H_{10}N_2O_7I_2$	Alcock & Countryman (1979)	
PAKKER	*C*2/*c*	4	$C_{52}H_{44}OFP_4Mo.BF_4$	Cotton, Eglin & Wiesinger (1992)	(*q*)
PEKKOF	*C*2/*c*	4	$C_{26}H_{30}O_2Yb$	Ji-Zhu, Zhong-Sheng, Wen-Qi & Yin (1993)	
PENRAB	*R*3*c*	6	$C_{15}H_{45}N_3Si_4Ti$	Ovchinnikov *et al.* (1992)	(*r*)
PENREF	*R*3*c*	6	$C_{15}H_{45}N_3Si_3GeTi$	Ovchinnikov *et al.* (1992)	(*r*)
PERDIZ	*C*2/*c*	4	$C_{12}H_{12}N_2Cl_2Pt$	Miskowski, Houlding, Che & Wang (1993)	
PIHXAF	*Fdd*2	16	$C_{24}H_{36}SIr_2$	Jones & Chin (1994)	
SAZPEO	*C*2/*c*	4	$C_{14}H_{18}N_2Cu.ClO_4$	Munakata, Kitagawa, Shimono & Masuda (1989)	(*d*)
SBFOXS	*C*2/*c*	4	$C_2O_4F_6Na_2Sb_2$	Escande, Tichit, Ducourant, Fourcade & Mascherpa (1978)	
SEMJUP	*C*2/*c*	4	$C_{28}H_{26}O_4Cl_4$	Mahato *et al.* (1988)	
SEYGEI	*C*2/*c*	4	$C_{37}H_{31}Cl_2F_2P_2Rh$	Burrell, Clark, Jeffrey, Rickard & Roper (1990)	(*s*)
SIKVIR	*C*2/*c*	8	$C_{20}H_{16}NOBr$	Crisma *et al.* (1990)	
SILLUU	*C*2/*c*	4	$C_{13}H_{34}NS_2P_4Co.2(BF_4)$	Kita, Okuyama, Kashiwabara & Fujita (1990)	(*d*)
SIRWIZ	*Fdd*2	8	$C_{27}H_{22}N_2$	Bond & Scott (1991)	
SIVHIO	*C*2/*c*	4	$C_4H_{16}N_6O_4Co.NO_2$	Bernal, Cetrullo & Berhane (1990)	
SOMJEJ	*C*2/*c*	4	$C_{37}H_{40}O_2Ti_2$	Nadasdi & Stephan (1991)	
SOTPAS	*C*2/*c*	4	$C_{20}H_{24}O_{12}S_4Rh_6.C_6H_{14}$	Rossi, Kallinen, Pursiainen, Pakkanen & Pakkanen (1991)	(*t*)
TARBOD	*C*2/*c*	4	$C_{73}H_{108}O_{12}$	Millini, Del Piero & Bracci (1992)	
VAWNAI	*C*2/*c*	4	$C_{13}H_{10}N_2Br_2$	Quast, Röschert, Peters, Peters & von Schnering (1989)	
VETFIJ	*C*2/*c*	4	$C_{31}H_{29}N_5Cl_2S_2Co.3.12(CH_3OH)$	Gheysen, Potts, Hurrell & Abrũna (1990)	(*u*)
VICZUC	*Fdd*2	16	$C_{16}H_{23}PS_4NiW$	Howard *et al.* (1990)	(*v*)
VOGHII	*C*2/*c*	4	$C_{42}H_{34}N_2O_6Cl_2P_2Rh_2.2(C_2H_4Cl_2)$	Mague (1990)	
VUNSAY	*C*2/*c*	4	$C_{21}H_{50}O_8Cl_4Al_4$	Turova, Yanovskii, Kessler, Kozlova & Struchkov (1991)	
WAKMEA	*R*3*c*	6	$C_6H_9O_6As$	Kamenar, Bruvo & Butumović (1993)	
WESDUT	*Fdd*2	16	$C_{17}H_{15}NFe$	Bosque, López, Sales, Solans & Font-Bardia (1994)	
YACGAK	*Fdd*2	16	$C_6H_4N_2O_2$	Dastidar, Guru Row & Venkatesan (1991)	
YAJZUE	*C*2/*c*	4	$C_{15}H_{38}NO_8S_2P_4Co.2BF_4$	Kita, Okuno, Kashiwabara & Fujita (1992)	
YEYNAR	*C*2/*c*	4	$2(C_{14}H_8S_{12}).C_{12}H_4N_4$	Aqad *et al.* (1994)	
YIDYAL	*C*2/*c*	4	$C_{17}H_{24}O$	Takahashi, Kotora, Kasai, Suzuki & Nakajima (1994)	

(*a*) The coordinates reported for space group *Cc* lead to short intermolecular contacts, including a C···C distance of 1.84 Å. These contacts can be removed if it is presumed that the origin in *Cc* was shifted by 0.25 in *y*, from its conventional location on a *c*-glide plane to an *n*-glide; the origin has been shifted back in the revised *C*2/*c* description. Included in the *Cc* description were two additional 'unsatisfactory' atoms, presumed to be water but separated by only 2.3 Å; these have been deleted. (*b*) In the original *Cc* description the N—O distance is very short (1.0 Å) and both atoms appear, in an *ORTEP* plot (Johnson, 1965), to have large displacement parameters; the Rh—N—O angle is 130°. In the revised *C*2/*c* structure this grouping lies on a C_2 axis and is presumably disordered. (*c*) An unusual bond length remains: C9—C9 (across a C_2 axis), at 1.72 Å. (*d*) Some H atoms are missing or are in unsatisfactory positions. (*e*) The C and N atoms of the solvate pyridine group could not be distinguished and were assumed to be disordered. In space group *C*2/*c* this group lies on a center of inversion. (*f*) The coordinated ethyl group was reported as disordered and has been discarded. In *C*2/*c* it lies on a C_2 axis. (*g*) Coordinates of the *tert*-butyl group, which is disordered, are not available. (*h*) In the revised *C*2/*c* structure, one tetraethylammonium ion lies on a center of inversion and must be disordered; it has been deleted. In the original *Cc* refinement, all three tetraethylammonium ions were constrained. (*i*) One ether grouping is apparently disordered. (*j*) In the *Cc* refinement the molecule was constrained to have C_2 symmetry (see text). (*k*) Coordinates of the triethylammonium ions and the dimethylformamide solvent molecule are not available. (*l*) In *C*2/*c* the toluene molecule of solvation is probably disordered and has been omitted; its geometry was constrained in the earlier *Cc* refinement. (*m*) I have refined the structure in *C*2/*c*, based on the original structure factors (SUP 71333), to an *R* of 0.073 compared with 0.074 for the earlier description; see text. (*n*) In *C*2/*c* the Cl and CO ligands are related by a C_2 axis and must be disordered. (*o*) The CO and Cl ligands are reported as disordered and have been removed. (*p*) The bond lengths and angles involving the coordinated formate group cannot be reproduced from the published coordinates. (*q*) The BF_4 group appears to be disordered and has been removed. (*r*) The original authors noted the trigonal symmetry and attempted, without success, to describe the structure in *R*3*m* (rather than *R*3*c*). (*s*) The difluoromethyl group is apparently disordered; the F atoms have been removed. (*t*) Coordinates of the hexane solvate are not available. (*u*) The methanol solvate molecules are probably disordered and have been removed. (*v*) The coordinates of five phenyl C atoms of one molecule, and of all H atoms of the second molecules, are missing. These unpaired atoms have been deleted.

example, $P2_1$ into $P2_1/m$ or $Pna2_1$ into *Pnam*. [*C*2/*c* is a very popular space group (Brock & Dunitz, 1994).] Finally, the addition of a C_2 or a C_3 axis to *Cc* can lead immediately to space group *Fdd*2 or *R*3*c*, with lattice types that perhaps are not easily recognized by standard cell-reduction routines (particularly some early versions).

In those instances in which experimental information was available in the original articles (deplorably, that was often not the case), it appears as though the most common reason for choosing *Cc* over *C*2/*c* was the $Z = 4$ situation: the asymmetric unit contained but a single formula unit and the authors failed to realize that the molecule might contain a C_2 axis. In at least two cases *C*2/*c* was explicitly rejected because the molecule could not have an inversion center. (In one of these cases – FAWBAG – the authors noted molecular C_2 symmetry and, indeed, constrained the molecule to lie on a C_2 axis parallel to **b**, apparently without realizing that this automatically generates *C*2/*c*.) In several other instances, however, the authors recognized the space-group ambiguity and attempted refinement in *C*2/*c* with results they considered unsatisfactory. In the case of CPOECU10 the authors report that refinement in *C*2/*c* led to an *R* of 0.438, compared with 0.071 for *Cc*; yet the distortions from *C*2/*c* are far too small to be responsible for this large a change. (The authors noted that the structure is pseudoface-centered and it is possible that they selected an incorrect origin in their attempts to describe it in *C*2/*c*.) In other cases the *R* values reported for *C*2/*c* were only slightly higher than those reported for *Cc*; while the difference might be formally significant according to the '*R* test' (Hamilton, 1964), it is well known that this test is not applicable in such situations. Not only must the test be carried out on weighted rather than unweighted residuals, but it cannot be considered valid unless an unbiased goodness-of-fit is close enough to 1.0 to ensure that the additional parameters are not obscuring systematic errors in the intensity data. In all these cases I believe that the unusual bond lengths and angles that resulted from refinement in *Cc* are adequate indicators that the choice of space group was inappropriate.

In a few instances, as noted in Table 1, the additional molecular symmetry required by space group *C*2/*c* can only be realized if disorder is assumed. In all cases this disorder was either already present in the original *Cc* description or was strongly suggested by unusual bond or conformational angles or by unrealistic displacement ellipsoids. Moreover, the disorder was not unusual, involving *tert*-butyl or isopropyl groups, BF_4^- ions, the interchange of Cl and C=O, or the like.

In those cases in which the increase in space-group symmetry entails a change in lattice type rather than the addition of an inversion center, the revised space group tends to be either *Fdd*2 or *R*3*c*, depending on whether a C_2 or C_3 axis is added; in one case the change is to $I\bar{4}c2$. In these cases there are no significant changes in the interatomic distances, as expected (Schomaker & Marsh, 1979); the symmetrizing of the coordinates of related atoms required no shift of over 0.01 Å or so. Thus, there can be no doubt that the change to higher symmetry is appropriate. However, when an inversion center is added and the space group becomes *C*2/*c* (there are no cases in Table 1 where both a center is added and the lattice type is changed) the symmetrizing is much less exact: coordinates of related atoms must usually be shifted by 0.1 Å or more and hence the interatomic distances are changed, well beyond reasonable e.s.d.'s. So how can we be certain that the change to *C*2/*c* is appropriate? The answer is: We cannot. In one case – HALJUZ – I recovered the original intensity data and carried out successful refinement in *C*2/*c* with entirely satisfactory results: a slightly lower *R* and greatly improved interatomic distances. However, to do the same with the other 74 structures is beyond my endurance (even if all the original intensity data were available, which is surely not the case). Even when better results are obtained in *C*2/*c*, one can never be sure that there are not small deviations from centrosymmetry, for such deviations are not detectable by standard diffraction methods (*e.g.* Schomaker & Marsh, 1979). What can be said is that the centrosymmetric *C*2/*c* descriptions lead to far more reasonable descriptions of the structures and that, lacking conclusive proof to the contrary, there is no valid reason to reject the more reasonable – and simpler – centrosymmetric descriptions.

So I believe that the estimate of 10% error in the assignment of space group *Cc* (Baur & Kessner, 1992) is accurate. This seems, to me, an intolerable rate of error. Perhaps the situation is improving, as cell reduction software has become more sophisticated and the access to symmetry-checking programs such as *MISSYM* (Le Page, 1988) is wider. However, it is still important that crystallographers remain ever alert to the many pitfalls that pocket the road to a correct structure solution and that they travel that road with thought, care and respect rather than with breakneck speed. If they do so, they may successfully avoid the perils of *Cc* – and many other potholes as well.

References

Alcock, N. W. & Countryman, R. M. (1979). *J. Chem. Soc. Dalton Trans.* pp. 851–853.

Allen, D. W., Nowell, I. W. & Taylor, B. F. (1985). *J. Chem. Soc. Dalton Trans.* pp. 2505–2508.

Anzenhofer, K. & ten Rouwelaar, L. N. A. (1967). *Recl. Trav. Chim. Pays-Bas*, **86**, 801–806.

Aqad, E., Becker, J. Y., Bernstein, J., Ellern, A., Khodorkovsky, V. & Shapiro, L. (1994). *J. Chem. Soc. Chem. Commun.* pp. 2775–2776.

Arif, A. M., Heaton, D. E. & Jones, R. A. (1986). *J. Chem. Soc. Chem. Commun.* pp. 1506–1507.

Baur, W. H. & Kassner, D. (1992). *Acta Cryst.* B**48**, 356–357.

Bello, P. (1973). *Gazz. Chim. Ital.* **103**, 537–541.

Bernal, I., Cetrullo, J. & Berhane, S. (1990). *Struct. Chem.* **1**, 361–366.

Bombieri, G., Benetollo, F., Polo, A., de Cola, L., Hawkins, W. T. & Vallarino, L. M. (1989). *Polyhedron*, **8**, 2157–2167.

Bond, D. R. & Scott, J. L. (1991). *J. Chem. Soc. Perkin Trans. 2*, pp. 47–51.

Bosque, R., López, C., Sales, J., Solans, X. & Font-Bardia, M. (1994). *J. Chem. Soc. Dalton Trans.* pp. 735–745.

Brock, C. P. & Dunitz, J. D. (1994). *Chem. Mater.* **6**, 1118–1127.

Bullivant, D. P., Dove, M. F. A. & Haley, M. J. (1980). *J. Chem. Soc. Dalton Trans.* pp. 109–114.

Burrell, A. K., Clark, G. R., Jeffery, J. G., Rickard, C. E. F. & Roper, W. R. (1990). *J. Organomet. Chem.* **388**, 391–408.

Cambridge Structural Database (1992). Version 4.6. Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, England.

Carballo, R. & Castiñeiras, A. (1993). *J. Cryst. Spectrosc.* **23**, 891–893.

Cecconi, F., Ghilardi, C. A., Midollini, S., Moneti, S. & Orlandini, A. (1986). *Angew. Chem. Int. Ed. Engl.* **25**, 833–834.

Colborn, R. E., Garbauskas, M. F. & Hejna, C. I. (1988). *Inorg. Chem.* **27**, 3661–3663.

Cooper, M. K., Duckworth, P. A., Hambley, T. W., Organ, G. J., Henrick, K., McPartlin, M. & Parekh, A. (1989). *J. Chem. Soc. Dalton Trans.* pp. 1067–1073.

Cosmo, R., Hambley, T. W. & Sternhell, S. (1987). *J. Org. Chem.* **52**, 3119–3123.

Cotton, F. A., Eglin, J. L. & Wiesinger, K. J. (1992). *Inorg. Chim. Acta*, **195**, 11–23.

Crisma, M., Valle, G., Bonora, G. M., De Menego, E., Toniolo, C., Lelj, F., Barone, V. & Fraternali, F. (1990). *Biopolymers*, **30**, 1–11.

Dastidar, P., Guru Row, T. N. & Venkatesan, K. (1991). *J. Mater. Chem.* **1**, 1057–1059.

Debaerdemaeker, T., Roth, H. & Brune, H.-A. (1991). *J. Organomet. Chem.* **412**, 243–249.

Dobson, A., Moore, D. S., Robinson, S. D., Galas, A. M. R. & Hursthouse, M. B. (1985). *J. Chem. Soc. Dalton Trans.* pp. 611–616.

Dvorkin, A. A., Simonov, Yu. A., Luk'yanenko, N. G., Kirichenko, T. I., Shcherbakov, S. V., Malinovskii, T. I. & Bogatskii, A. V. (1985). *Kristallografiya*, **30**, 915–918.

Emsley, J., Jones, D. J. & Kuroda, R. (1982). *J. Chem. Soc. Dalton Trans.* pp. 1179–1184.

Ermer, O. & Dunitz, J. (1970). *Acta Cryst.* A**26**, 163.

Escande, P., Tichit, D., Ducourant, B., Fourcade, R. & Mascherpa, G. (1978). *Ann. Chim.* (*Paris*), **3**, 117–124.

Fenske, D., Grissinger, A., Hey-Hawkins, E. M. & Magull, J. (1991). *Z. Anorg. Allg. Chem.* **595**, 57–66.

Filippova, T. V., Polynova, T. N., Il'inskii, A. L., Porai-Koshits. M. A. & Ezerskaya, N. A. (1981). *Zh. Neorg. Khim.* **26**, 1418–1419.

Fletton, R. A., Humber, D. C., Roberts, S. M., Owston, P. G. & Henrick, K. (1983). *J. Chem. Soc. Chem. Commun.* pp. 968–970.

Gérard, C., Lucken, E. A. C. & Bernardinelli, G. (1986). *J. Chem. Soc. Perkin Trans. 2*, pp. 879–884.

Gheysen, K. A., Potts, K. T., Hurrell, H. C. & Abrũna, H. D. (1990). *Inorg. Chem.* **29**, 1589–1592.

Hamilton, W. C. (1964). *Statistics in Physical Science*, pp. 157–162. New York: The Ronald Press Company.

Harrison, P. G., King, T. J. & Healy, M. A. (1979). *J. Organomet. Chem.* **182**, 17–36.

Hou, Z., Wakatsuki, Y. & Yamazaki, H. (1990). *J. Organomet. Chem.* **399**, 103–114.

Howard, K. E., Lockemeyer, J. R., Massa, M. A., Rauchfuss, T. B., Wilson, S. R. & Yang, X. (1990). *Inorg. Chem.* **29**, 4385–4390.

Hunt, M. M., Kemmitt, R. D. W., Russell, D. R. & Tucker, P. A. (1979). *J. Chem. Soc. Dalton Trans.* pp. 287–294.

Irngartinger, H., Reimann, W., Garner, P. & Dowd, P. (1988). *J. Org. Chem.* **53**, 3046–3050.

Ishida, T., Hatta, K.-I., Yamashita, S., Doi, M. & Inoue, M. (1986). *Chem. Pharm. Bull.* **34**, 3553–3562.

Ji-Zhu, J., Zhong-Sheng, J., Wen-Qi, C. & Yin, Z. (1993). *Jiegou Huaxue* (*J. Struct. Chem.*), **12**, 241–244.

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Jones, W. D. & Chin, R. M. (1994). *J. Am. Chem. Soc.* **116**, 198–203.

Kabe, Y., Kawase, T., Okada, J., Yamashita, O., Goto, M. & Masamune, S. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 794–796.

Kamenar, B., Bruvo, M. & Butumović, J. (1993). *Z. Anorg. Allg. Chem.* **619**, 943–946.

Kita, M., Okuno, M., Kashiwabara, K. & Fujita, J. (1992). *Bull. Chem. Soc. Jpn*, **65**, 3042–3048.

Kita, M., Okuyama, A., Kashiwabara, K. & Fujita, J. (1990). *Bull. Chem. Soc. Jpn*, **63**, 1994–2001.

Konovalikhin, S. V., Shilov, G. V., D'yachenko, O. A., Lyubovskaya, R. N., Aldoshina, M. Z. & Lyubovskii, R. B. (1992). *Izv. Akad. Nauk. SSSR Ser. Khim.* pp. 903–909.

Krakowiak, K. E., Bradshaw, J. S., Dalley, N. K., Zhu, C., Yi, G., Curtis, J. C., Li, D. & Izatt, R. M. (1992). *J. Org. Chem.* **57**, 3166–3173.

Kulishov, V. I., Bokii, N. G., Struchkov, Yu. T., Nefedov, O. M., Kolesnikov, S. P. & Perl'mutter, B. L. (1970). *Zh. Strukt. Khim.* **11**, 71–74.

Lan-Sung, Z., Hua-Hui, Y. & Qian-Er, Z. (1991). *Jiegou Huaxue* (*J. Struct. Chem.*), **10**, 97–99.

Le Page, Y. (1988). *J. Appl. Cryst.* **21**, 983–984.

Lee, S., Nakanishi, K., Chiang, M. Y., Frankel, R. B. & Spartalian, K. (1988). *J. Chem. Soc. Chem. Commun.* pp. 785–786.

Lu, T., Yoo, H. K., Zhang, H., Bott, S., Atwood, J. L., Echegoyen, L. & Gokel, G. W. (1990). *J. Org. Chem.* **55**, 2269–2270.

Mague, J. T. (1990). *Polyhedron*, **9**, 2635–2640.

Mahato, S. B., Mandal, N. B., Pal, A. K., Maitra, S. K., Lehmann, C. & Luger, P. (1988). *J. Org. Chem.* **53**, 5554–5558.

Mangion, M. M., Smith, R. & Shore, S. G. (1976). *Cryst. Struct. Commun.* **5**, 493–500.

Matsumoto, H., Minemura, M., Takatsuna, K., Nagai, Y. & Goto, M. (1985). *Chem. Lett.* pp. 1005–1006.

Matsumoto, H., Takatsuna, K., Minemura, M., Nagai, Y. & Goto, M. (1985). *J. Chem. Soc. Chem. Commun.* pp. 1366–1368.

Meersche, M. van, Germain, G., Declercq, J. P. & Wilputte-Steinert, L. (1981). *Cryst. Struct. Commun.* **10**, 47–53.

Millini, R., Del Piero, G. & Bracci, M. (1992). *Acta Cryst.* C**48**, 696–699.

Miskowski, V. M., Houlding, V. H., Che, C.-M. & Wang, Y. (1993). *Inorg. Chem.* **32**, 2518–2524.

Morrison, R. M., Thompson, R. C. & Trotter, J. (1983). *Can. J. Chem.* **61**, 1651–1658.

Mösges, G., Hampel, F., Kaupp, M. & von Ragué Schleyer, P. (1992). *J. Am. Chem. Soc.* **114**, 10880–10889.

Munakata, M., Kitagawa, S., Shimono, H. & Masuda, H. (1989). *Inorg. Chim. Acta*, **158**, 217–220.

Nadasdi, T. T. & Stephan, D. W. (1991). *Can. J. Chem.* **69**, 167–171.

Nasielski, J., Nasielski-Hinkens, R., Heliporn, S., Rypens, C. & Declercq, J. P. (1988). *Bull. Soc. Chim. Belg.* **97**, 983–992.

Neidlein, R., Degener, H.-J., Gieren, A., Weber, G. & Hubner, T. (1985). *Z. Naturforsch. Teil B*, **40**, 1532–1536.

Newton, M. G., Caughman, H. D. & Taylor, R. C. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1031–1037.

Oku, A., Harada, K., Yagi, T. & Shirahase, Y. (1983). *J. Am. Chem. Soc.* **105**, 4400–4407.

Ovchinnikov, Yu. E., Igonin, V. A., Timofeeva, T. V., Lindeman, S. V., Struchkov, Yu. T., Ustinov, M. V. & Bravo-Zhivotovskii, D. A. (1992). *Metalloorg. Khim.* (*Organomet. Chem.*, in Russian), **5**, 1154–1160.

Paseshnitchenko, K. A., Aslanov, L. A., Jatsenko, A. V. & Medvedev, S. V. (1985). *J. Organomet. Chem.* **287**, 187–194.

Pasynskii, A. A., Eremenko, I. L., Gasanov, G. Sh., Struchkov, Yu. T. & Shklover, V. E. (1984). *J. Organomet. Chem.* **276**, 349–362.

Perevalova, E. G., Reshetova, M. D., Ostapchuk, P. N., Slovokhotov, Yu. L., Struchkov, Yu. T., Spiridonov, F. M., Kisin, A. V. & Yukhno, I. G. (1990). *Metalloorg. Khim.* (*Organomet. Chem.*, in Russian), **3**, 100–104.

Precigoux, G., Busetta, B., Courseille, C., Hospital, M. & Miquel, J. F. (1978). *Acta Cryst.* B**34**, 3300–3305.

Qin, L., Guo-Xiong, W., Yan, Z., Zhan-Feng, Y., Cheng, Z. & Zhong-Yuan, Z. (1989). *Huaxue Xuebao* (*Acta Chim. Sin.*), **47**, 1065–1070.

Quast, H., Röschert, H., Peters, E.-M., Peters, K. & von Schnering, H. G. (1989). *Liebigs Ann. Chem.* pp. 503–513.

Rickard, C. E. F., Roper, W. R., Taylor, G. E., Waters, J. M. & Wright, L. J. (1990). *J. Organomet. Chem.* **389**, 375–388.

Ridder, D. J. A. de, Schenk, H. & Döpp, D. (1993). *Acta Cryst.* C**49**, 1970–1971.

Robertson, G. B. & Tucker, P. A. (1981). *Acta Cryst.* B**37**, 814–821.

Robinson, J. A., Flohr, H., Kempe, U. M., Pannhorst, W. & Rétey, J. (1983). *Liebigs Ann. Chem.* pp. 181–203.

Rossi, S., Kallinen, K., Pursiainen, J., Pakkanen, T. T. & Pakkanen, T. A. (1991). *J. Organomet. Chem.* **419**, 219–232.

Ruiter, B. de, Benson, J. E., Jacobson, R. A. & Verkade, J. G. (1990). *Inorg. Chem.* **29**, 1065–1068.

Schomaker, V. & Marsh, R. E. (1979). *Acta Cryst.* B**35**, 1933–1934.

Schreiber, P., Wieghardt, K., Nuber, B. & Weiss, J. (1990). *Z. Anorg. Allg. Chem.* **587**, 174–192.

Schultz, A. G., Macielag, M., Podhorez, D. E., Suhadolnik, J. C. & Kullnig, R. K. (1988). *J. Org. Chem.* **53**, 2456–2464.

Shimoi, M., Saito, Y. & Ogino, H. (1991). *Bull. Chem. Soc. Jpn*, **64**, 2629–2634.

Takahashi, T., Kotora, M., Kasai, K., Suzuki, N. & Nakajima, K. (1994). *Organometallics*, **13**, 4183–4185.

Tobita, H., Wada, H., Ueno, K. & Ogino, H. (1994). *Organometallics*, 2545–2547.

Turova, N. Ya., Yanovskii, A. I., Kessler, V. G., Kozlova, N. I. & Struchkov, Yu. T. (1991). *Zh. Neorg. Khim.* **36**, 2497–2502.

Wade, P. A., Kondracki, P. A. & Carroll, P. J. (1991). *J. Am. Chem. Soc.* **113**, 8807–8811.

Walther, D., Schmidt, A., Klettke, T., Imhof, W. & Görls, H. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 1373–1376.

Willert-Porada, M. A., Burton, D. J. & Baenziger, N. C. (1989). *J. Chem. Soc. Chem. Commun.* pp. 1633–1634.

Woning, J., Daniels, L. M. & Verkade, J. G. (1990). *J. Am. Chem. Soc.* **112**, 4601–4602.

Xintao, W., Bing, W., Yifan, Z. & Jiaxi, L. (1988). *Jiegou Huaxue* (*J. Struct. Chem.*), **7**, 47–52.

Yamamoto, Y. & Yamazaki, H. (1985). *Bull. Chem. Soc. Jpn*, **58**, 1843–1844.

Zeiher, C., Mohyla, J., Lorenz, I.-P. & Hiller, W. (1985). *J. Organomet. Chem.* **286**, 159–170.

Zhanfeng, Y., Guoxiong, W., Ling, Y., Fengshan, W. & Yuguo, F. (1990). *Nanjing Univ. Dax. Xuebao Zir. Kex.* **26**, 256.

Zhigui, Z., Chunxiao, G., Yuguo, F. & Fengshan, W. (1986). *J. Inorg. Chem.* (*Wuji Huaxue Xuebao*), **2**, 26–31.

Zviedre, I., Fundamensky, V. S. & Schwartz, E. (1992). *Latv. Khim.* Zh. pp. 527–532.

Zyss, J., Ledoux, I., Bertault, M. & Toupet, E. (1991). *Chem. Phys.* **150**, 125–135.