

To understand this phenomenon further structure determinations of crystalline adducts of related pairs (e.g. digitoxigenin and digirezigenin 1:1) are now in progress.

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References

- ARGAY, GY., KÁLMÁN, A., RIBÁR, B., VLADIMIROV, S. & ŽIVANOV-STAKIĆ, D. (1987). *Acta Cryst.* **C43**, 922–926.
- BLOSS, F. D. (1971). *Crystallography and Crystal Chemistry*, pp. 249–250. New York: Holt, Rinehart & Winston.
- CROWFOOT, D. (1935). *Chem. Ind.* pp. 568–569.
- DECLERCO, J.-P., GERMAIN, G. & KING, G. S. D. (1977). *Abstr. 4th Eur. Crystallogr. Meet.*, Oxford, pp. 279–280.
- DUNITZ, J. D. (1979). *X-ray Analysis and the Structure of Organic Molecules*, pp. 106–111. Ithaca: Cornell Univ. Press.
- ENRAF-Nonius (1982). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
- GO, K. & BHANDARY, K. K. (1989). *Acta Cryst.* **B45**, 306–312.
- HÖHNE, E. & PFEIFFER, D. (1983). *Stud. Biophys.* **97**, 81–86.
- KÁLMÁN, A., ARGAY, GY., FÜLÖP, V., RIBÁR, B. & LAZAR, D. (1987). *Acta Cryst.* **A43**, S66–S67.
- KÁLMÁN, A., ARGAY, GY., RIBÁR, B., VLADIMIROV, S. & ŽIVANOV-STAKIĆ, D. (1984). *Croat. Chem. Acta*, **57**, 519–528.
- KÁLMÁN, A., FÜLÖP, V., ARGAY, GY., RIBÁR, B., LAZAR, D., ŽIVANOV-STAKIĆ, D. & VLADIMIROV, S. (1988). *Acta Cryst.* **C44**, 1634–1638.
- KARLE, I. L. & KARLE, J. (1969). *Acta Cryst.* **B25**, 434–442.
- KITAIGORODSKY, A. I. (1961). *Organic Chemical Crystallography*, pp. 5–17. New York: Consultants Bureau.
- MESSERSCHMIDT, A., HÖHNE, E. & MEGGES, R. (1981). *Cryst. Struct. Commun.* **10**, 149–156.
- PRASAD, L. & GABE, E. J. (1983). *Acta Cryst.* **C39**, 273–275.
- RIBÁR, B., ARGAY, GY., KÁLMÁN, A., VLADIMIROV, S. & ŽIVANOV-STAKIĆ, D. (1983). *J. Chem. Res. (M)*, pp. 1001–1042.
- ROHRER, D. C., FULLERTON, D. S., KITATSUJI, E., NAMBARA, T. & YOSHII, E. (1982). *Acta Cryst.* **B38**, 1865–1868.
- SCHARFENBERG-PFEIFFER, D., HÖHNE, E. & WUNDERWALD, M. (1987). *Cryst. Res. Technol.* **22**, 1403–1408.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1991). **B47**, 77–86

Pseudoinversion Centers in Space Group $P\bar{1}$ and a Redetermination of the Crystal Structure of 3,4-Dimethoxycinnamic Acid. A Study of Non-Crystallographic Symmetry

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Abstract

A survey of the 11691 $P\bar{1}$ crystal structures in the Cambridge Structural Database shows that 1166 have $Z = 4$. Of these, a mere 20 have local pseudocenters of symmetry relating the atoms in the two halves of the asymmetric unit. The coordinates of these local pseudocenters often include the special values of 0 , $\frac{1}{4}$ and $\frac{1}{2}$, but can also be perfectly general. As an example of the latter, the crystal structure of 3,4-dimethoxycinnamic acid ($C_{11}H_{12}O_4$, $M_r = 208.21$) was re-examined at both room temperature and at 173 K. The poor refinement reported in the original

study of this compound was ascribed to the low data/parameter ratio and the presence of the pseudocenter. The present study, however, clearly demonstrates that the refinement is unaffected by the presence of the pseudocenter when the data/parameter ratio is reasonable (> 5). This could be true in this specific case because the center is located at a general position: 0.217, 0.433, 0.319. At room temperature, $a = 8.449$ (1), $b = 15.034$ (2), $c = 8.449$ (1) Å, $\alpha = 99.47$ (1), $\beta = 94.57$ (1), $\gamma = 101.53$ (1)°, $V = 1029$ Å³, 1893 reflections, 367 variables, $R = 0.041$, $wR = 0.038$. At 173 K, $a = 8.412$ (2), $b = 14.959$ (2), $c = 8.341$ (2) Å, $\alpha = 100.28$ (2), $\beta = 94.80$ (2), $\gamma = 101.83$ (2)°, $V = 1003$ Å³, 2543 reflections, $R = 0.041$, $wR = 0.040$.

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Table 1. Summary of Cambridge database survey

Space group	Total Number	Z	Number with Z	%
$P\bar{1}$	11691	4	1166	10.0
$P2_1$	4205	4	686	16.3
Pc	255	4	55	21.6
$P2_1/c$	24859	8	1393	5.6
$P2_12_1$	7220	8	390	5.4
$Pbca$	2822	16	98	3.5

Introduction

The presence of multiple molecules in the asymmetric unit, in other words symmetry-independent molecules, is a familiar phenomenon among organic crystal structures but little has been done to correlate or even compare data on such compounds. With the rising interest in organic crystal structure prediction (Desiraju, 1987, 1989; Scheffer, 1987), it would be useful to further understand this phenomenon in terms of the principles of packing or even to ascertain if there is a relationship between its occurrence and attributes such as chemical functionality, molecular weight or crystal system. Realistically, it is impossible, at least at the present time, to make predictions about an effect as subtle as the presence of two or more symmetry-independent molecules in a crystal. However, the basic tool for the systematic examination of crystal structures, the Cambridge Structural Database, is readily available (Allen, Kennard & Taylor, 1983). Hence we decided to examine a small subset of structures which crystallize in the space group $P\bar{1}$ with Z, the number of molecules in the unit cell, equal to 4 and further where the symmetry-independent molecules are related by additional pseudoinversion symmetry.

The general principles to rationalize the crystal structures of simple organic compounds are now well established and the close-packing principle provides a satisfactory explanation for the prevalence of certain space groups and crystal systems (Kitai-gorodskii, 1973). It is not necessary to invoke the presence of symmetry-independent molecules to understand why space groups like $P2_1/c$, $P2_1$, $P\bar{1}$, $P2_12_1$, and $Pbca$ are favored for organic crystal structures. Indeed, although structures with symmetry-independent molecules are more common in the lower-symmetry space groups and crystal systems (Padmaja, Ramakumar & Viswamitra, 1990), they do not occur very frequently. The statistics given in Table 1, from the 1988 version 3.1 of the Cambridge Database (69 691 entries), are pertinent.

As can be seen in the table, the occurrence of multiple molecules in the asymmetric unit seems to be more common in the space groups $P\bar{1}$, $P2_1$ and Pc . We initially considered the $P2_1$ and Pc structures. However, an examination of the 55 Pc structures showed that at least 25 of them may be described as a reduction in symmetry from $P2_1/c$ packing with the

true center in $P2_1/c$ being replaced by a pseudo-center. A similar situation is obtained in around 60 of the 686 $P2_1$ structures where $Z = 4$. These structures are merely variations of $P2_1/c$ packing with the pseudocenter being 'global', in other words, generating approximate molecular positions throughout the crystal. In the large majority of the $P2_1$ structures, however, there do not seem to be obvious orientational relationships between symmetry-independent molecules. In addition, some of the space-group assignments, especially of the Pc structures, are ambiguous because of order-disorder problems. For all these reasons, the structures in these space groups were not examined further.

From a packing viewpoint it seemed to be of greater interest to examine structures which, rather than being lower-symmetry derivatives of common space groups, contain higher or 'extra' symmetry. Such a situation arises in $P\bar{1}$ when $Z = 4$ if the symmetry-independent molecules are further related by a pseudocenter of symmetry. Such a pseudocenter is 'local' rather than 'global' in that it relates only pairs of symmetry-independent molecules. This curious phenomenon was observed earlier (Desiraju, Kamala, Kumari & Sarma, 1984) by one of us in a structural study of 3,4-dimethoxycinnamic acid (CEMJOT). The room-temperature analysis of this crystal was quite problematic and prompted a re-examination of the structure at low temperature to establish if the problems associated with the analysis were related to the non-crystallographic symmetry relationships in the crystal.

Experimental

Crystals of 3,4-dimethoxycinnamic acid were grown from a 1:1 mixture of acetone and methanol by slow evaporation. The crystals grew as elongated plates and, as noted in the original work (Desiraju, Kamala, Kumari & Sarma, 1984), typically had many visible striations. It was however possible to find small crystals which appeared relatively free of defects as well as large crystals which seemed to have clear sections. Since the crystals were found to shatter rather than cleave, it was the small, intact crystals which were used in this investigation.

All of the crystallographic studies were carried out on a Syntex P3 diffractometer equipped with a molybdenum tube ($\lambda = 0.71069 \text{ \AA}$) and low-temperature apparatus. Data were collected at both 173 K (labelled LT) and at room temperature (labelled RT). ω scans showed half-widths of somewhat less than 0.30° , which represent very reasonable values for the Syntex diffractometer. Both data sets were collected by the ω -scan technique with a scan range of 1.2° and scan speeds which ranged from 2.0

Table 2. Comparison of the crystallographic information used in various studies of 3,4-dimethoxycinnamic acid

	GRD	RT	LT
Crystal size (mm)	0.1 × 0.03 × 0.4	0.30 × 0.15 × 0.47	0.30 × 0.15 × 0.47
<i>a</i> (Å)	8.448 (3)	8.449 (1)	8.412 (2)
<i>b</i> (Å)	15.072 (8)	15.034 (2)	14.959 (2)
<i>c</i> (Å)	8.437 (6)	8.440 (2)	8.341 (2)
α (°)	99.44 (5)	99.47 (1)	100.28 (2)
β (°)	94.71 (5)	94.57 (1)	94.80 (2)
γ (°)	101.59 (4)	101.53 (1)	101.83 (2)
<i>V</i> (Å ³)	1031	1029	1003
<i>T</i> (K)	298	293	173
<i>Z</i>	4	4	4
<i>D_c</i> (g cm ⁻³)	1.34	1.344	1.379
μ (cm ⁻¹)	2.925	0.96	0.98
Data collected	2925	5022	4916
2 θ range (°)	3–45	4–55	4–55
Transmission factors			0.97–0.98
Unique data [<i>I</i> > 3 σ (<i>I</i>)]	725	1893	2543
Parameters	271	367	367
Data/parameter ratio	2.67	5.16	6.92
<i>R</i>	0.107	0.041	0.041
<i>wR</i>	0.136	0.038	0.040
Maximum residual density (e Å ⁻³)	0.44	0.18	0.36
Located near		C(5)	C(21)

to 10.0° min⁻¹. Check reflections were monitored every 100 reflections; corrections for intensity variations were applied, as were Lorentz and polarization corrections. An empirical absorption correction, based on ψ -scan data, was applied to the low-temperature data set.

Both structures were refined by full-matrix, least-squares techniques starting with the atomic coordinates previously published. The H atoms were located and included in all of the refinements. The acid hydrogens, H(1) and H(21), were refined with full occupancy although the C—O bond lengths are indicative of partial disordering of the C—O and C=O moieties. Weights for the refinement were assigned as $[\sigma^2(I) + 0.0009I]^{-1/2}$. The computer programs used for these analyses were written by one of us (JCC).

A summary of the crystallographic information is given in Table 2; for comparative purposes, the previously published (GRD) results are also included. Tables 3 and 4 list the atomic coordinates which resulted from the refinements of the RT and LT data sets respectively.* Selected bond parameters for both studies are compared with those in the original work in Tables 5 and 6. Fig. 1 compares the thermal parameters for the RT and LT studies; the reduction in temperature from 296 to 173 K causes an average decrease in B_{eq} of 45%. Fig. 2 is a packing diagram showing the locations of the pseudoinversion centers.

* Lists of anisotropic thermal parameters and structure amplitudes for RT and LT have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53472 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Fractional coordinates and isotropic thermal parameters for the RT study

	<i>x</i>	<i>y</i>	<i>z</i>	B_{iso}/B_{eq} (Å ²)
O(1)	0.1763 (3)	0.5272 (2)	0.2435 (3)	4.8 (1)*
O(2)	0.0101 (3)	0.4179 (2)	0.3349 (3)	5.6 (1)*
O(3)	-0.2551 (3)	0.8433 (2)	-0.0971 (3)	4.8 (1)*
O(4)	-0.5532 (3)	0.7891 (1)	-0.0512 (3)	4.6 (1)*
O(21)	0.2543 (3)	0.3462 (2)	0.4244 (3)	5.0 (1)*
O(22)	0.4198 (3)	0.4509 (1)	0.3211 (3)	4.6 (1)*
O(23)	0.6944 (3)	0.0219 (2)	0.7374 (3)	5.2 (1)*
O(24)	0.9881 (2)	0.0748 (2)	0.6804 (3)	4.5 (1)*
C(1)	-0.2163 (4)	0.6433 (2)	0.0954 (4)	3.4 (1)*
C(2)	-0.1731 (4)	0.7190 (2)	0.0218 (4)	3.7 (1)*
C(3)	-0.2876 (4)	0.7669 (2)	-0.0272 (4)	3.5 (1)*
C(4)	-0.4499 (4)	0.7370 (3)	-0.0009 (4)	3.5 (1)*
C(5)	-0.4939 (5)	0.6616 (3)	0.0713 (5)	4.2 (1)*
C(6)	-0.3775 (5)	0.6146 (2)	0.1186 (4)	4.2 (1)*
C(7)	-0.0900 (4)	0.5978 (2)	0.1471 (4)	3.9 (1)*
C(8)	-0.1028 (4)	0.5275 (2)	0.2248 (5)	4.3 (1)*
C(9)	0.0355 (4)	0.4882 (2)	0.2705 (4)	3.9 (1)*
C(10)	-0.0926 (4)	0.8743 (3)	-0.1288 (6)	5.1 (1)*
C(11)	-0.7223 (5)	0.7550 (4)	-0.0481 (7)	5.8 (2)*
C(21)	0.6468 (4)	0.2201 (2)	0.5436 (4)	3.3 (1)*
C(22)	0.6061 (4)	0.1438 (2)	0.6194 (4)	3.7 (1)*
C(23)	0.7215 (4)	0.0967 (2)	0.6630 (4)	3.5 (1)*
C(24)	0.8825 (4)	0.1255 (2)	0.6310 (4)	3.5 (1)*
C(25)	0.9239 (4)	0.2005 (2)	0.5564 (4)	4.2 (1)*
C(26)	0.8050 (4)	0.2478 (2)	0.5133 (4)	4.0 (1)*
C(27)	0.5179 (4)	0.2669 (2)	0.4969 (4)	3.8 (1)*
C(28)	0.5326 (4)	0.3407 (2)	0.4307 (4)	3.7 (1)*
C(29)	0.3939 (4)	0.3813 (2)	0.3902 (4)	3.6 (1)*
C(30)	0.5359 (5)	-0.0082 (3)	0.7807 (5)	4.6 (1)*
C(31)	1.1513 (5)	0.0969 (4)	0.6393 (6)	5.2 (1)*
H(1)	0.2655 (65)	0.4897 (36)	0.2788 (61)	14.8 (18)
H(2)	-0.0643 (31)	0.7406 (17)	0.0032 (28)	3.1 (7)
H(5)	-0.6047 (37)	0.6392 (21)	0.0938 (36)	5.4 (9)
H(6)	-0.4081 (36)	0.5634 (20)	0.1711 (36)	4.6 (8)
H(7)	0.0170 (39)	0.6245 (21)	0.1126 (37)	5.3 (8)
H(8)	-0.2015 (32)	0.4980 (19)	0.2565 (31)	3.3 (7)
H(10)	-0.0965 (36)	0.9281 (21)	-0.1787 (36)	4.6 (8)
H(10')	-0.0597 (44)	0.8269 (25)	-0.2034 (43)	7.7 (12)
H(10'')	-0.0117 (40)	0.8934 (21)	-0.0283 (39)	6.2 (9)
H(11)	-0.7681 (33)	0.7974 (19)	-0.0909 (32)	3.6 (7)
H(11')	-0.7535 (44)	0.7518 (27)	0.0645 (48)	8.5 (12)
H(11'')	-0.7599 (50)	0.6928 (32)	-0.1194 (53)	11.4 (16)
H(21)	0.1451 (55)	0.3787 (31)	0.3817 (54)	12.7 (16)
H(22)	0.4938 (33)	0.1186 (19)	0.6427 (32)	3.9 (7)
H(25)	1.0399 (38)	0.2206 (21)	0.5360 (35)	5.5 (9)
H(26)	0.8428 (34)	0.3001 (19)	0.4655 (34)	4.2 (8)
H(27)	0.4122 (39)	0.2381 (22)	0.5226 (38)	6.3 (9)
H(28)	0.6415 (35)	0.3690 (20)	0.4080 (34)	4.9 (8)
H(30)	0.4484 (37)	-0.0209 (20)	0.6930 (36)	5.3 (8)
H(30')	0.5544 (35)	-0.0610 (21)	0.8398 (34)	5.4 (8)
H(30'')	0.5023 (32)	0.0416 (19)	0.8506 (32)	4.5 (7)
H(31)	1.1992 (43)	0.0474 (27)	0.6911 (43)	8.2 (11)
H(31')	1.2003 (40)	0.1593 (25)	0.6842 (42)	6.8 (10)
H(31'')	1.1488 (44)	0.0894 (26)	0.5237 (46)	8.6 (13)

Structure of 3,4-dimethoxycinnamic acid

In the original X-ray study (GRD) of 3,4-dimethoxycinnamic acid, CEMJOT (Desiraju, Kamala, Kumari & Sarma, 1984), severe refinement problems were encountered. The two symmetry-independent molecules had to be refined individually in separate cycles to keep the refinement orderly, and even then, two of the atoms had to be fixed. Not surprisingly, the resulting bond distances were less than satisfactory. The poor refinement appeared to have two root causes. First, because the crystal was very small and the X-ray flux relatively low, the number of observed reflections was very small (see Table 2). Second, the molecules were seen to pack as dimers across pseudocenters of symmetry and it was suggested that correlations between the two molecules hindered the

Table 4. Fractional coordinates and isotropic thermal parameters for the LT study

	x	y	z	$B_{iso}/B_{eq}^*(\text{\AA}^2)$
O(1)	0.1780 (2)	0.5292 (1)	0.2431 (2)	2.6 (1)*
O(2)	0.0120 (2)	0.4189 (1)	0.3369 (2)	3.0 (1)*
O(3)	-0.2544 (2)	0.8444 (1)	-0.0998 (2)	2.6 (1)*
O(4)	-0.5537 (2)	0.7903 (1)	-0.0541 (2)	2.5 (1)*
O(21)	0.2556 (2)	0.3460 (1)	0.4263 (2)	2.8 (1)*
O(22)	0.4199 (2)	0.4519 (1)	0.3205 (2)	2.5 (1)*
O(23)	0.6965 (2)	0.0218 (1)	0.7391 (2)	2.7 (1)*
O(24)	0.9924 (2)	0.0751 (1)	0.6807 (2)	2.5 (1)*
C(1)	-0.2173 (3)	0.6438 (2)	0.0927 (3)	2.0 (1)*
C(2)	-0.1722 (3)	0.7201 (2)	0.0174 (3)	2.0 (1)*
C(3)	-0.2873 (3)	0.7679 (2)	-0.0301 (3)	2.0 (1)*
C(4)	-0.4509 (3)	0.7377 (2)	-0.0044 (3)	2.0 (1)*
C(5)	-0.4962 (3)	0.6625 (2)	0.0686 (3)	2.3 (1)*
C(6)	-0.3806 (3)	0.6154 (2)	0.1172 (3)	2.3 (1)*
C(7)	-0.0894 (3)	0.5983 (2)	0.1453 (3)	2.1 (1)*
C(8)	-0.1040 (3)	0.5281 (2)	0.2254 (3)	2.3 (1)*
C(9)	0.0353 (3)	0.4892 (2)	0.2713 (3)	2.1 (1)*
C(10)	-0.0900 (3)	0.8755 (2)	-0.1309 (3)	2.7 (1)*
C(11)	-0.7249 (3)	0.7561 (2)	-0.0522 (4)	3.1 (1)*
C(21)	0.6481 (3)	0.2205 (2)	0.5431 (3)	1.9 (1)*
C(22)	0.6075 (3)	0.1441 (2)	0.6197 (3)	2.0 (1)*
C(23)	0.7243 (3)	0.0964 (2)	0.6640 (3)	2.0 (1)*
C(24)	0.8862 (3)	0.1254 (2)	0.6318 (3)	2.0 (1)*
C(25)	0.9271 (3)	0.2010 (2)	0.5555 (3)	2.2 (1)*
C(26)	0.8083 (3)	0.2480 (2)	0.5117 (3)	2.2 (1)*
C(27)	0.5191 (3)	0.2673 (2)	0.4974 (3)	2.1 (1)*
C(28)	0.5353 (3)	0.3421 (2)	0.4309 (3)	2.1 (1)*
C(29)	0.3965 (3)	0.3831 (2)	0.3900 (3)	2.0 (1)*
C(30)	0.5352 (3)	-0.0077 (2)	0.7813 (3)	2.3 (1)*
C(31)	1.1554 (3)	0.0979 (2)	0.6393 (3)	2.7 (1)*
H(1)	0.2676 (49)	0.5013 (27)	0.2775 (46)	9.2 (12)
H(2)	-0.0610 (27)	0.7402 (15)	-0.0008 (25)	1.9 (5)
H(5)	-0.6094 (28)	0.6360 (17)	0.0889 (28)	2.9 (6)
H(6)	-0.4108 (33)	0.5616 (18)	0.1620 (32)	3.9 (6)
H(7)	0.0198 (32)	0.6250 (18)	0.1141 (31)	3.7 (6)
H(8)	-0.2056 (27)	0.4989 (16)	0.2561 (26)	2.0 (5)
H(10)	-0.0917 (28)	0.9305 (16)	-0.1754 (28)	2.1 (5)
H(10')	-0.0522 (30)	0.8243 (18)	-0.2035 (30)	3.6 (6)
H(10'')	-0.0107 (30)	0.8935 (17)	-0.0305 (30)	3.4 (6)
H(11)	-0.7791 (29)	0.7994 (17)	-0.0980 (29)	3.0 (5)
H(11')	-0.7513 (32)	0.7544 (19)	0.0578 (35)	4.1 (7)
H(11'')	-0.7637 (28)	0.6917 (18)	-0.1159 (29)	3.3 (6)
H(21)	0.1629 (40)	0.3780 (23)	0.3919 (39)	6.9 (9)
H(22)	0.4958 (30)	0.1225 (16)	0.6473 (29)	2.9 (5)
H(25)	1.0412 (31)	0.2238 (17)	0.5392 (29)	3.2 (6)
H(26)	0.8418 (24)	0.2965 (14)	0.4613 (24)	1.0 (4)
H(27)	0.4155 (30)	0.2379 (17)	0.5184 (29)	3.0 (6)
H(28)	0.6419 (28)	0.3728 (16)	0.4083 (27)	2.6 (5)
H(30)	0.4506 (27)	-0.0277 (15)	0.6805 (27)	2.5 (5)
H(30')	0.5447 (28)	-0.0598 (16)	0.8340 (28)	2.7 (5)
H(30'')	0.5067 (26)	0.0423 (16)	0.8561 (26)	2.4 (5)
H(31)	1.2093 (30)	0.0539 (18)	0.6840 (30)	3.4 (6)
H(31')	1.2123 (30)	0.1620 (19)	0.6903 (31)	3.6 (6)
H(31'')	1.1529 (29)	0.0856 (17)	0.5201 (31)	3.4 (6)

refinement. The present study was initiated to resolve this problem structure and clarify its origins.

Since there was an expectation that the structure would always be a problem with data taken at room temperature, the LT structure was examined first. Surprisingly, the refinement of the structure converged rapidly and smoothly. The H atoms were clearly seen in the difference map and ultimately included in the refinement. Unexpectedly, the refinement of the room-temperature structure also converged in a straightforward fashion. A review of the correlation coefficients revealed none greater than 0.56 for the RT case and 0.52 for LT, both of which were related to components of the thermal-motion tensor of individual atoms. In fact, the only problem with the structures was the partial disorder of the carbonyl and hydroxyl O atoms of the acid

Table 5. Comparative bond lengths (\AA) for the three studies of the structure of 3,4-dimethoxycinnamic acid

	GRD	RT	LT
C(1)—C(2)	1.39 (3)	1.385 (4)	1.400 (3)
C(21)—C(22)	1.42 (3)	1.401 (4)	1.403 (3)
C(1)—C(6)	1.40 (2)	1.383 (4)	1.395 (3)
C(21)—C(26)	1.48 (3)	1.376 (4)	1.387 (3)
C(1)—C(7)	1.45 (2)	1.453 (4)	1.464 (3)
C(21)—C(27)	1.50 (2)	1.472 (4)	1.466 (3)
C(2)—C(3)	1.34 (3)	1.392 (4)	1.388 (3)
C(22)—C(23)	1.38 (2)	1.376 (4)	1.389 (3)
C(3)—C(4)	1.41 (2)	1.403 (4)	1.405 (3)
C(23)—C(24)	1.49 (2)	1.404 (4)	1.405 (3)
C(3)—O(3)	1.40 (2)	1.365 (3)	1.364 (3)
C(23)—O(23)	1.30 (1)	1.365 (3)	1.363 (2)
C(4)—C(5)	1.34 (3)	1.375 (5)	1.375 (3)
C(24)—C(25)	1.33 (3)	1.380 (4)	1.392 (3)
C(4)—O(4)	1.37 (3)	1.370 (3)	1.369 (3)
C(24)—O(24)	1.43 (2)	1.367 (3)	1.362 (2)
C(5)—C(6)	1.43 (3)	1.392 (5)	1.386 (3)
C(25)—C(26)	1.37 (3)	1.400 (4)	1.394 (3)
C(7)—C(8)	1.31 (2)	1.324 (4)	1.332 (3)
C(27)—C(28)	1.30 (2)	1.311 (4)	1.323 (3)
C(8)—C(9)	1.54 (3)	1.463 (4)	1.464 (3)
C(28)—C(29)	1.49 (3)	1.468 (4)	1.469 (3)
C(9)—O(1)	1.22 (2)	1.272 (4)	1.287 (3)
C(29)—O(21)	1.27 (2)	1.268 (3)	1.287 (3)
C(9)—O(2)	1.27 (3)	1.256 (4)	1.257 (3)
C(29)—O(22)	1.27 (3)	1.269 (3)	1.258 (3)
C(10)—O(3)	1.46 (2)	1.420 (4)	1.427 (3)
C(30)—O(23)	1.41 (2)	1.422 (4)	1.432 (3)
C(11)—O(4)	1.53 (2)	1.422 (4)	1.428 (3)
C(31)—O(24)	1.53 (2)	1.436 (4)	1.430 (3)
O(1)—H(1)		1.08 (5)	0.98 (4)
O(21)—H(21)		1.19 (5)	1.04 (3)
O(2)—H(21)		1.44 (5)	1.59 (3)
O(22)—H(1)		1.57 (5)	1.65 (4)
C—H range		0.92–1.05	0.91–1.01

groups. Clearly, the problems associated with the original structure stem solely from the lack of observed reflections and not from the presence of pseudocenters of symmetry.

The pseudocenter of symmetry in the LT structure, calculated as the centroid of the non-H atoms of the dimer, is located at a general position: 0.217, 0.433, 0.319. The slight observed deviation from an inversion center occurs because the two molecules are not exactly coplanar; although each individual molecule is reasonably planar (largest deviations are 0.086 and 0.053 \AA , respectively), the dihedral angle has a non-zero value of 4.2° which causes significant deviations at the methoxy ends of the dimer.

A review of the packing in the LT structure reveals several C—H...O interactions which, although weak, are noteworthy. First, as seen in Fig. 2, the molecules pack in planar sheets with the carboxyl O atoms interacting with H atoms from neighboring benzene rings. Not seen in Fig. 2 are the head-to-tail interactions between the dimers wherein the methoxy O atoms interact with neighboring methoxy —CH₃ groups. The shorter of these contacts vary from 2.52 to 2.71 \AA . It is interesting to note that two of the

Table 6. Comparative bond angles ($^{\circ}$) for the three studies of the structure of 3,4-dimethoxycinnamic acid

	GRD	RT	LT
C(2)—C(1)—C(6)	119 (1)	118.7 (3)	119.0 (2)
C(22)—C(21)—C(26)	119 (1)	119.0 (3)	118.6 (2)
C(2)—C(1)—C(7)	117 (1)	118.7 (3)	118.1 (2)
C(22)—C(21)—C(27)	122 (1)	118.7 (3)	118.7 (2)
C(6)—C(1)—C(7)	122 (1)	122.6 (3)	122.8 (2)
C(26)—C(21)—C(27)	118 (1)	122.3 (3)	122.6 (2)
C(1)—C(2)—C(3)	121 (1)	121.5 (3)	120.6 (2)
C(21)—C(22)—C(23)	121 (1)	121.0 (3)	121.2 (2)
C(2)—C(3)—C(4)	121 (1)	118.7 (3)	119.1 (2)
C(22)—C(23)—C(24)	115 (1)	119.6 (3)	119.4 (2)
C(2)—C(3)—O(3)	129 (1)	125.4 (3)	125.0 (2)
C(22)—C(23)—O(23)	124 (1)	125.5 (3)	125.4 (2)
C(4)—C(3)—O(3)	108 (1)	116.0 (3)	115.9 (2)
C(24)—C(23)—O(23)	120 (1)	115.0 (3)	120.2 (2)
C(3)—C(4)—C(5)	115 (1)	120.2 (3)	120.5 (2)
C(23)—C(24)—C(25)	125 (1)	120.0 (3)	119.6 (2)
C(3)—C(4)—O(4)	117 (1)	114.5 (3)	114.3 (2)
C(23)—C(24)—O(24)	102 (1)	115.0 (3)	115.3 (2)
C(5)—C(4)—O(4)	124 (3)	125.3 (3)	125.2 (2)
C(25)—C(24)—O(24)	130 (1)	125.0 (3)	125.1 (2)
C(4)—C(5)—C(6)	125 (1)	120.1 (4)	120.2 (2)
C(24)—C(25)—C(26)	118 (1)	119.7 (4)	120.2 (2)
C(1)—C(6)—C(5)	115 (1)	120.8 (4)	120.5 (2)
C(21)—C(26)—C(25)	120 (1)	120.8 (4)	120.9 (2)
C(1)—C(7)—C(8)	127 (1)	128.7 (4)	127.9 (2)
C(21)—C(27)—C(28)	129 (1)	127.8 (3)	127.3 (2)
C(7)—C(8)—C(9)	120 (1)	123.1 (4)	122.2 (2)
C(27)—C(28)—C(29)	121 (1)	122.6 (3)	122.6 (2)
C(8)—C(9)—O(1)	119 (1)	118.4 (3)	118.1 (2)
C(28)—C(29)—O(21)	117 (1)	119.5 (3)	117.9 (2)
C(8)—C(9)—O(2)	114 (1)	118.6 (3)	119.3 (2)
C(28)—C(29)—O(22)	116 (2)	117.8 (3)	119.0 (2)
O(1)—C(9)—O(2)	126 (1)	123.0 (3)	122.6 (2)
O(21)—C(29)—O(22)	125 (1)	122.7 (3)	123.0 (2)
C(3)—O(3)—C(10)	116 (1)	117.5 (3)	117.2 (2)
C(23)—O(23)—C(30)	116 (1)	118.0 (2)	117.4 (2)
C(4)—O(4)—C(11)	114 (1)	117.0 (3)	117.0 (2)
C(24)—O(24)—C(31)	109 (1)	117.2 (3)	117.0 (2)
C(9)—O(1)—H(1)		110 (3)	115 (2)
C(29)—O(21)—H(21)		117 (2)	114 (2)
O(1)—H(1)···O(22)		169 (5)	176 (4)
O(21)—H(21)···O(2)		178 (4)	175 (3)
C(9)—O(2)···H(21)		117 (2)	120 (1)
C(29)—O(22)···H(1)		115 (2)	120 (1)

shortest C—H···O contacts [O(2)···H(26)', 2.52; O(22)···H(6)'', 2.57 Å] involve those carboxyl oxygens with the greater carbonyl character while contacts involving the other two oxygens with the lesser carbonyl character are much longer: O(1)···H(5)'', 2.71, O(21)···H(25)', 2.65 Å. This suggests that the partial ordering of the acid groups reflects a sensitivity to their environment, a carbonyl oxygen being a much better hydrogen-bond acceptor than a hydroxyl oxygen.

Database studies

As shown in Table 1 above, of the 11 691 structures in $P1$, 1166 have $Z = 4$. Identification of those structures with pseudoinversion symmetry was accomplished with a program which calculates all angles

between mean planes of all chemical residues in the structures (minimum number of residues = 2) and which retrieves a structure if any one interplanar angle is less than a specified value. When this tolerance value was set at 5° , 68 hits were retrieved. These 68 structures were manually inspected and it was observed that in many cases the small interplanar angles are a matter of coincidence and occur between chemically distinct residues (salts, complexes). Some planar compounds were also found where the small interplanar angles are between parallel, stacked, symmetry-independent and chemically similar but not pseudoinversion-related molecules. For example, such molecules could be rotated about the

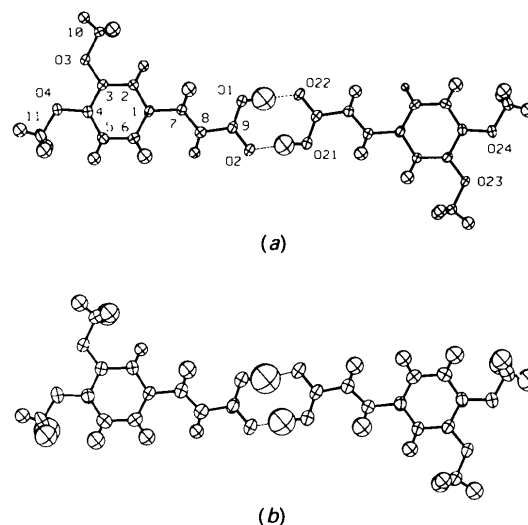


Fig. 1. Thermal parameters obtained (a) at 173 K and (b) at 296 K.

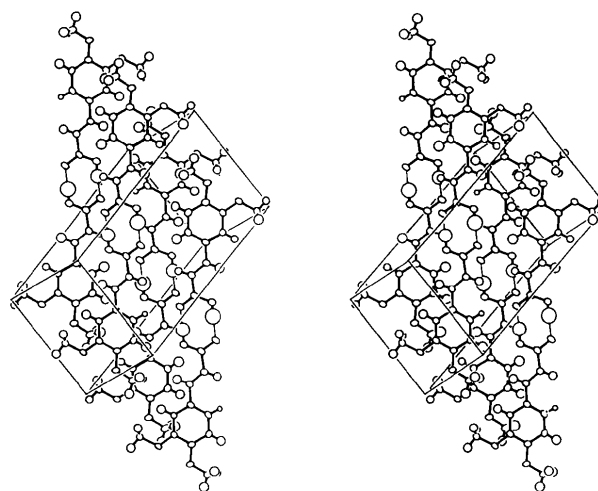


Fig. 2. Packing diagram showing the O—H···O and C—H···O mediated layer structure of 3,4-dimethoxycinnamic acid.

Table 7. List of compounds in this study and their bibliographic references

AZFAPD10, (hexafluoroacetylacetonato-*O,O'*)[phenylazophenyl-C(1),N(2)]palladium, $C_{17}H_{10}F_6N_2O_5Pd$, α form, yellow. ETTER, M. C. & SIDLE, A. R. (1983). *J. Am. Chem. Soc.* **105**, 641. Class 71, Coord 56, $R = 0.045$, Sigf 2, Intf 3, $P\bar{1}$.

BAHFAR, *p*-hydroxy- α -1-[(1-methyl-2-phenoxyethyl)amino]ethylbenzyl alcohol hydrochloride (isoxsuprine hydrochloride), $C_{18}H_{24}NO_3Cl$. LEGER, J.-M., CARPY, A. & COLLETER, J.-C. (1981). *Acta Cryst.* **B37**, 1927. Class 17, Coord 94, $R = 0.048$, Sigf 2, Intf 3, $P\bar{1}$.

CEMJOT, 3,4-dimethoxycinnamic acid, $C_{11}H_{12}O_4$. DESIRAJU, G. R., KAMALA, R., KUMARI, B. H. & SARMA, J. A. R. P. (1984). *J. Chem. Soc. Perkin Trans. 2*, p. 81. Class 17, Coord 54, $R = 0.107$, Sigf 3, Intf 3, $P\bar{1}$.

CISPID, dimethylpyrazolo[1,2-*a*]pyrazolo[1,2-*b*]benzotriazole-3,4-dicarboxylate, $C_{15}H_{13}N_5O_4$. BOVIO, B. (1984). *J. Cryst. Spectrosc.* **14**, 369. Class 36, Coord 70, $R = 0.034$, Sigf 1, Intf 3, $P\bar{1}$.

CPRDCA, cyclopropane-1,1-dicarboxylic acid, $C_3H_4O_4$. MEESTER, M. A. M., SCHENK, H. & MACGILLAVRY, C. H. (1971). *Acta Cryst.* **B27**, 630. Class 20, Coord 30, $R = 0.043$, Sigf 1, Intf 3, $P\bar{1}$.

CTZHPC, chloro(2,7,12-trimethyl-3,7,11,17-tetraazabicyclo[11.3.1]heptadeca-1(17),2,11,13,15-pentaene)copper(II) nitrate dihydrate $[C_{18}H_{24}C_4N_4Cu][NO_3]_2 \cdot 2H_2O$. CAIRA, M. R., NASSIMBENI, L. R. & WOOLLEY, P. R. (1975). *Acta Cryst.* **B31**, 1334. Class 83, Coord 56, $R = 0.108$, Sigf 4, Intf 3, $P\bar{1}$.

DAPPOZ, tricarbonyl-(μ -diphenylphosphino)-(7⁻⁵-cyclopentadienyl)triphenylphosphineplatinumtungsten, $C_{38}H_{40}O_5P_2PtW$. POWELL, J., SAWYER, J. F. & SMITH, S. J. (1985). *J. Chem. Soc. Chem. Commun.* p. 1312. Class 86, Coord 90, $R = 0.049$, Sigf 3, Intf 3, $P\bar{1}$.

DEPKIS, 1,4-diazathianthrene, $C_{10}H_8N_2S_2$, 163 K. LARSON, S. B., SIMONSEN, S. H., LAM, W. W., MARTIN, G. E., LINDSAY, C. M. & SMITH, K. (1985). *Acta Cryst.* **C41**, 1784. Class 39, Coord 40, $R = 0.039$, Sigf 1, Intf 3, $P\bar{1}$.

DEPZIH, octadecamethylcyclononaphosphazene, $C_{18}H_{24}N_8P_8$. OAKLEY, R. T., RETTIG, S. J., PADDUCK, N. L. & TROTTER, J. (1985). *J. Am. Chem. Soc.* **107**, 6923. Class 64, Coord 180, $R = 0.040$, Sigf 0, Intf 3, $P\bar{1}$.

DEXCOY, 1,2,3,4,4a,9,9a,10-octahydro-*syn*-17-methyl-9,10(1',2')-benzeno-1,4-methanoanthracene, $C_{22}H_{22}$. LINDNER, H. J., LENOIR, D. & BUTLER, D. N. (1985). *Chem. Ber.* **118**, 363. Class 31, Coord 87, $R = 0.061$, Sigf 1, Intf 3, $P\bar{1}$.

DOTWAK, 2,2-ethylenedioxy-1-hydroxytricyclo[10.6.0.0^{13,18}]octadeca-13,15,17-triene, $C_{20}H_{28}O_2$. COURTOIS, A., BENABICHA, F., GREGOIRE, B. & CARRE, M. C. (1986). *Acta Cryst.* **C42**, 868. Class 38, Coord 100, $R = 0.060$, Sigf 2, Intf 3, $P\bar{1}$.

FEDLEF, (pentamethylcyclopenta-2,4-diene-1,1-dithiocarboxylato-*S,S'*)bis(triphenylphosphine)copper, $C_{47}H_{54}CuP_2S_2$. OTTO, H. & WERNER, H. (1987). *Chem. Ber.* **120**, 97. Class 86, Coord 194, $R = 0.138$, Sigf 4, Intf 3, $P\bar{1}$.

FERTHF, tris(ferrocenyl)(2-tetrahydrofuran)ylmethane, $C_{33}H_{34}Fe_3O$. HANIC, F., SEVCIK, J. & MCGRANDY, E. L. (1970). *Chem. Zvesti*, **24**, 81. Class 73, Coord 78, $R = 0.063$, Sigf 3, Intf 3, $P\bar{1}$.

HALOPE, *N*-(2-[4-(5-chloro-2-oxo-1-benzimidazolonyl)piperidino]ethyl)-*p*-fluorobenzamide (halopemide), $C_{27}H_{27}ClF_2N_5O_2$, antitumor, antipsychotic. VAN OPDENBOSCH, N., WEYLAND, M., EVRARD, G. & DURANT, F. (1980). *Acta Cryst.* **B36**, 965. Class 35, Coord 58, $R = 0.090$, Sigf 0, Intf 3, $P\bar{1}$.

IMMAZO10, 2-imino-3-methyl-5-methoxycarbonylmethylene-1,3-thiazolidin-4-one methanol solvate, $C_7H_{11}N_2O_5S \cdot 0.5CH_3O$. ADMAN, E., JENSEN, L. H. & WARRENER, R. N. (1975). *Acta Cryst.* **B31**, 1915. Class 41, Coord 42, $R = 0.055$, Sigf 0, Intf 3, $P\bar{1}$.

MIBDIO, 3-methoxyimino-1,2-benzodithiole-1,1-dioxide, $C_8H_8NO_2S_2$. FERRARI, M. B., CAPACCHI, L. C. & FAVA, G. G. (1973). *Cryst. Struct. Commun.* **2**, 185. Class 39, Coord 28, $R = 0.104$, Sigf 2, Intf 2, $P\bar{1}$.

MPPHAZ10, 6-methyl-2,2,4,4-tetraphenyldiphospha-1,3,5-triazine, $C_{28}H_{24}N_3P_2$. AHMED, F. R. & POLLARD, D. R. (1971). *Acta Cryst.* **B27**, 163. Class 64, Coord 98, $R = 0.067$, Sigf 3, Intf 3, $P\bar{1}$.

NAPTPR, 2,7-(1,8-naphthyridino)-1,4,7,10,13,16-hexaoxahexadeca-1,16-diyli, $C_{18}H_{24}N_2O_6$. NEWKOME, G. R., GARIBIS, S. J., MAJESTIC, V. K., FRONCZEK, F. R. & CHIARI, G. (1981). *J. Org. Chem.* **46**, 833. Class 40, Coord 100, $R = 0.055$, Sigf 3, Intf 3, $P\bar{1}$.

ODECYA, 4-octadecyenoic acid, $C_{18}H_{32}O_2$. MO, F. (1979). *Acta Cryst.* **B35**, 2135. Class 1, Coord 102, $R = 0.071$, Sigf 3, Intf 1, $P\bar{1}$.

PACYPT, *cis*-bis(phenylacetylido)bis(triphenylphosphine)platinum(II), $C_{52}H_{40}P_2Pt$. BONAMICO, M., DESSY, G., FARES, V., RUSSO, M. V. & SCARAMUZZA, L. (1977). *Cryst. Struct. Commun.* **6**, 39. Class 71, Coord 110, $R = 0.110$, Sigf 4, Intf 1, $P\bar{1}$.

PHNQUN10, 7-hydroxy-2,8-dimethoxy-1,4-phenanthrenequinone (cypripedin), $C_{16}H_{12}O_5$. BARTSCH, H.-H., SCHMALLE, H. W., JARCHOW, O. H., HAUSEN, B. M. & SCHULZ, K.-H. (1986). *Acta Cryst.* **C42**, 322. Class 59, Coord 66, $R = 0.041$, Sigf 2, Intf 3, $P\bar{1}$.

stacking axis. Removal of these two types of structures resulted in around 20 entries where symmetry-independent molecules are related by a pseudocenter or a pseudotranslation. In $P\bar{1}$ for $Z = 4$, a pseudo-inversion located at x_c, y_c, z_c is equivalent to a pseudotranslation, $x_i, y_i, z_i = 2x_c, 2y_c, 2z_c$, and so either of these representations is equally appropriate.

Table 7 lists the refcodes and bibliographic information for 18 of the resulting structures. Fig. 3 gives the structural formulae of these compounds. In

Table 8. Coordinates of the pseudo-inversion centers for the compounds in this study

Refcode	x	y	z
AZFAPD10	0.74	0.75	0.25
BAHFAR	0.75	0.74	0.25
CEMJOT	0.22	0.43	0.32
CISPID	0.48	0.74	0.51
CPRDCA	0.25	0.44	0.68
CTZHPC	0.50	0.25	0.50
DAPPOZ	0.26	0.50	0.25
DEPKIS	0.57	0.32	0.57
DEPZIH	0.25	0.00	0.75
DEXCOY	0.81	0.27	0.24
DOTWAK	0.00	0.26	0.24
FEDLEF	0.68	0.21	0.42
FERTHF	0.36	0.67	0.75
HALOPE	0.25	0.50	0.26
IMMAZO10	0.49	0.55	0.24
MIBDIO	0.86	0.25	0.24
MPPHAZ10	0.26	0.25	0.50
NAPTPR	0.74	0.00	0.75
ODECYA	0.25	0.50	0.50
PACYPT	0.77	-0.01	0.25
PHNQUN10	0.30	0.28	0.52

all cases, the unit-cell dimensions were checked and the cells reduced so as to confirm the space-group assignments. No errors or additional crystallographic symmetry relationships were found. Fig. 4 shows stereoviews of the structures from which it may be noted that the deviations from the pseudocenter (or pseudotranslation) are very minor. Purely geometrical considerations suggest that the most likely coordinates of the pseudocenters should include the 'special' values 0, $\frac{1}{4}$, $\frac{1}{2}$ and $\frac{3}{4}$. Table 8 shows that this indeed is often the case. It may be shown that even for those structures in which the pseudocenter lies at a 'general' position (CEMJOT, DEPKIS), a redefinition of the unit cell will enable at least one of the pseudocenter coordinates to be 'special'.

A rough classification of these entries is possible in terms of the deviation of the molecular structure from pseudosymmetry. Fig. 4 shows that in the structures CEMJOT, CTZHPC, DOTWAK, PHNQUN and MIBDIO the pseudocenter is nearly perfect. More commonly, the pseudocenter relates only a part of the symmetry-independent molecules while there are variations in the other part which disturb the pseudocenter. These variations could be conformational in nature as in AZFAPD (trifluoromethyl group), BAHFAR, BUFNEW (phenyl ring), CISPID (methyl group), FEDLEF (cyclopentadienyl ring), FERTHF (tetrahydrofuran ring), HALOPE (benzoyl group), MPPHAZ (part of a macrocycle) and ODECYA (carboxylate group). In CPRDCA, the molecules would be related by a pseudocenter if the hydrogen-bonded catemer of carboxyl groups were completely disordered. However, the structure is ordered and to that extent, deviates from a pseudocenter. In the structure of DAPPOZ, the diphenylphosphine and triphenylphosphine groups form a hydrocarbon envelope. While the individual phenyl rings deviate considerably from a pseudo-inversion relationship, their combined

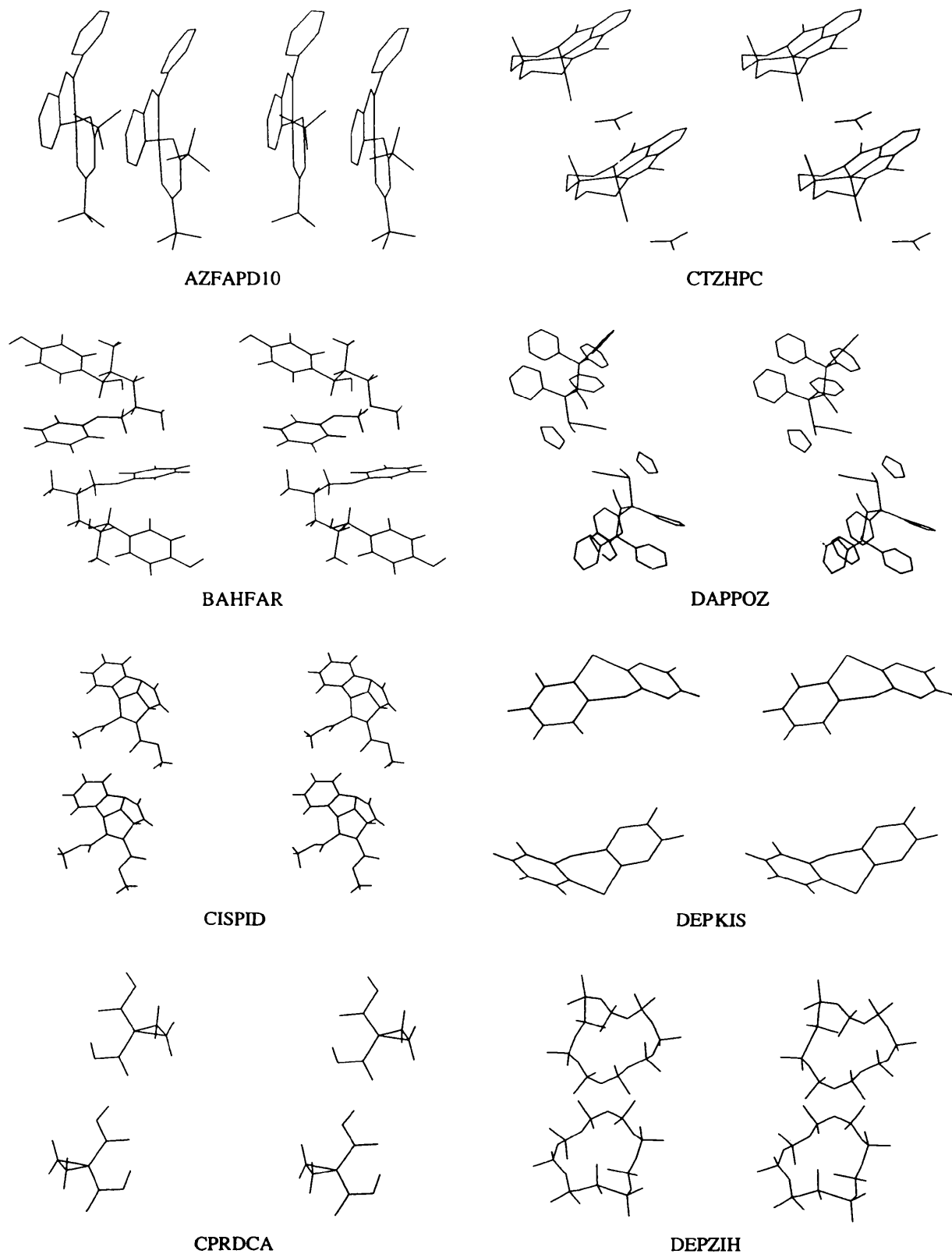


Fig. 4. Stereodrawings of the asymmetric units of the structures in this database study. Note that a pseudotranslation is equivalent to a pseudoinversion in $P\bar{1}$, $Z = 4$.

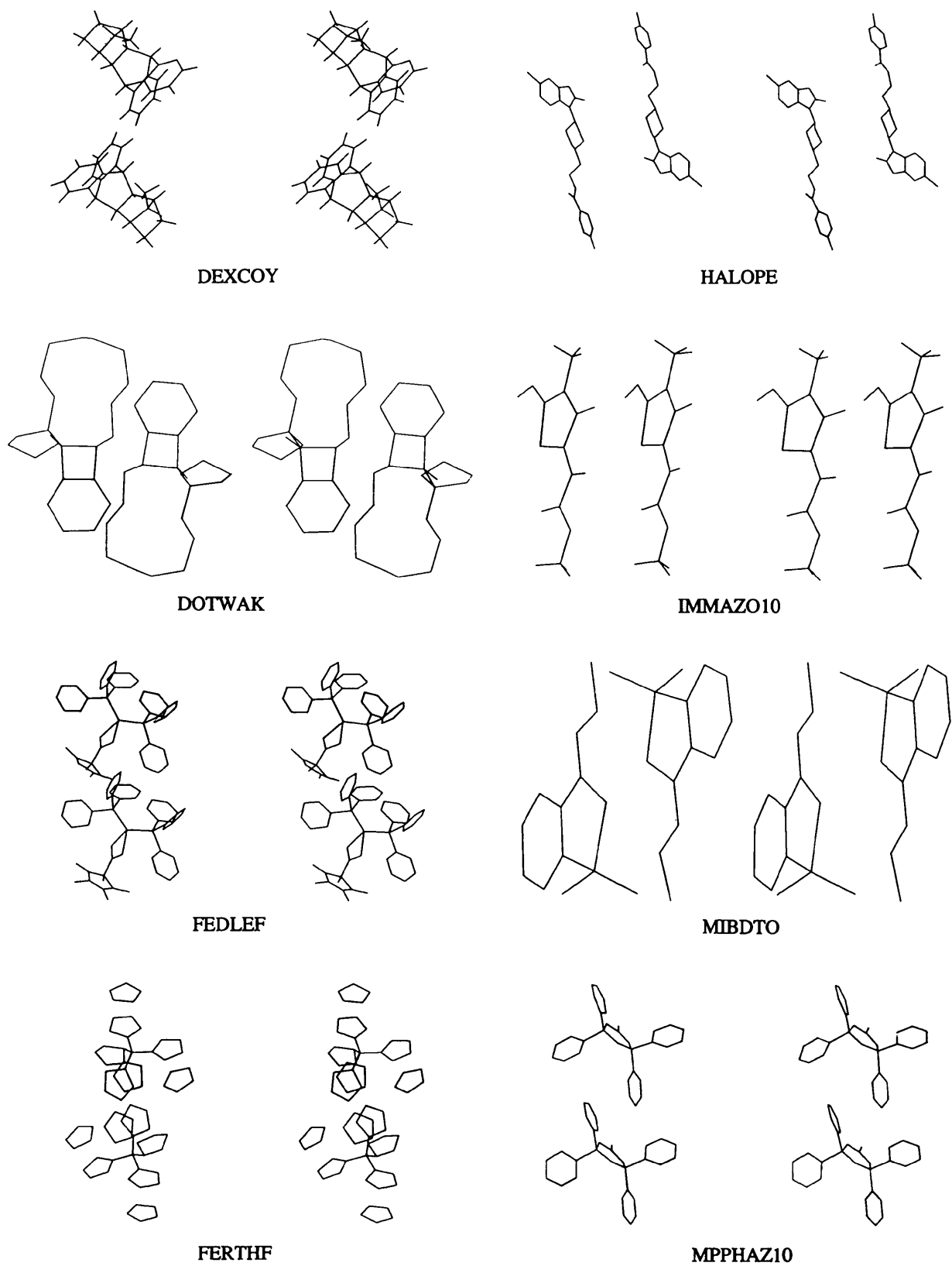


Fig. 4 (cont.) Stereodrawings of the asymmetric units of the structures in this database study.

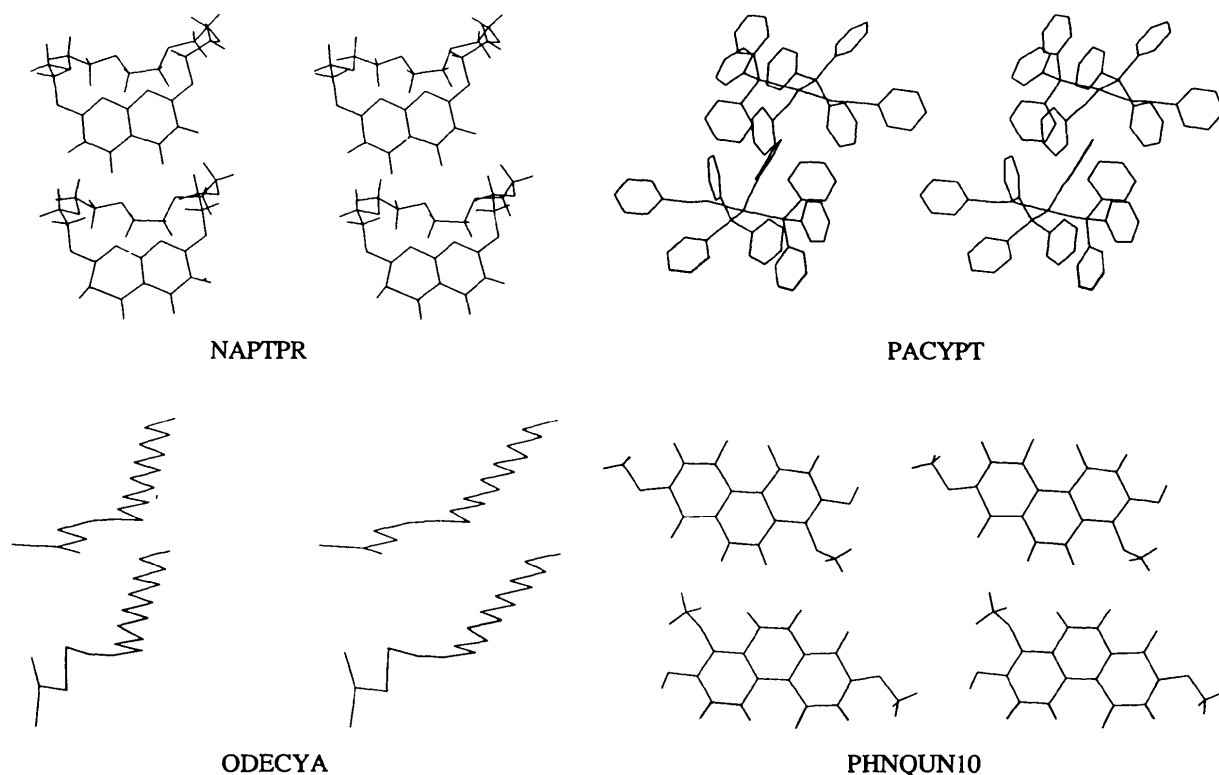


Fig. 4 (cont.) Stereodrawings of the asymmetric units of the structures in this database study.

located in special positions where all three coordinates are either 0 , $\frac{1}{4}$, $\frac{1}{2}$ or $\frac{3}{4}$ (more commonly thought of as pseudotranslations of 0 , $\frac{1}{2}$, 1 , etc.) give rise to superlattices. Approximately half of the structures listed in Table 8 fall into this category and yet only a few authors reported being aware of the superlattice. Complications during the least-squares refinement of superlattices are well known. The refinement of the crystal structure of 3,4-dimethoxycinnamic acid presented here strongly suggests, however, that a pseudocenter located in a 'general' position does not adversely affect the refinement. It should be re-emphasized, however, that at least one coordinate of such a 'general' pseudocenter can, by suitable axial transformation, be converted to a 'special' value of $\frac{1}{4}$, $\frac{1}{2}$ etc. In the CEMJOT case, the pseudocenter at $0.22, 0.43, 0.32$ is equivalent to a pseudocenter at $0.22, 0.43, 0.75$ in a cell which is obtained by the transformation $[100, 01\bar{1}, 00\ 1]$. To confirm our suggestion that 'general' pseudocenters do not cause refinement problems, a review of references contain-

ing such structures was conducted. The results were unenlightening primarily because these structures are few in number, variable in quality, and were published for the most part without comment about refinement difficulties.

References

- ALLEN, F. H., KENNARD, O. & TAYLOR, R. (1983). *Acc. Chem. Res.* **16**, 146–151.
- DESIRAJU, G. R. (1987). Editor. *Organic Solid State Chemistry*. Amsterdam: Elsevier.
- DESIRAJU, G. R. (1989). *Crystal Engineering. The Design of Organic Solids*. Amsterdam: Elsevier.
- DESIRAJU, G. R., KAMALA, R., KUMARI, B. H. & SARMA, J. A. R. P. (1984). *J. Chem. Soc. Perkin Trans. 2*, pp. 181–189.
- KITAIGORODSKII, A. I. (1973). *Molecular Crystals and Molecules*. New York: Academic Press.
- PADMAJA, N., RAMAKUMAR, S., & VISWAMITRA, M. A. (1990). *Acta Cryst. A* **46**, 725–730.
- SCHEFFER, J. R. (1987). *Organic Chemistry in Anisotropic Media*, Tetrahedron Symposium-in-Print, No. 29. *Tetrahedron*, **43**, 1197–1737.