

## References

- ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–22.
- BUCHI, G., KITaura, Y., YUAN, S. S., WRIGHT, H. E., CLARDY, J., DEMAIN, A. L., GLINSUKIN, T., HUNT, N. & WOGAN, G. N. (1973). *J. Am. Chem. Soc.* **95**, 5423–5425.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- DE TITTA, G. T., EDMONDS, J. E., LANGS, D. A. & HAUPTMAN, H. A. (1975). *Acta Cryst.* **A31**, 472–479.
- ENGEL, D. W. (1972). *Acta Cryst.* **B28**, 1496–1509.
- HAUPTMAN, H. A. (1972). *Crystal Structure Determination*. New York: Plenum.
- HAUPTMAN, H. A. (1975). *Acta Cryst.* **A31**, 680–687.
- KABUTO, C., SILVERTON, J. V., AKIYAMA, T., SANKAWA, V., HUTCHISON, R. D., STEYN, P. S. & VLEGGAR, R. (1976). *J. Chem. Soc. Chem. Commun.* pp. 728–729.
- MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1971). *MULTAN, A Computer Program for the Automatic Solution of Crystal Structures*. Univs. of York, England, and Louvain, Belgium.
- PETERSON, S. W. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 70–76.
- SILVERTON, J. V. & AKIYAMA, T. (1977). Unpublished results.
- SILVERTON, J. V., AKIYAMA, T., KABUTO, C., SEKITA, S., YOSHIHARA, K. & NATORI, S. (1976). *Tetrahedron Lett.* pp. 1349–1350.
- SILVERTON, J. V. & KABUTO, C. (1975). Unpublished programs.
- SPRINGER, J. P., CLARDY, J., WELL, J. M., COLE, R. J., KIRKSEY, J. W., MACFARLANE, R. D. & TORGERSON, D. F. (1976). *Tetrahedron Lett.* pp. 1355–1358.
- STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). The XRAY system—version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- TSUKUDA, Y. & KOYAMA, H. (1972). *J. Chem. Soc. Perkin Trans. 2*, pp. 739–744.

*Acta Cryst.* (1978). **B34**, 593–597

## The Crystal and Molecular Structures of Two Derivatives of a Spiro-oxazolone: a *cis* vs *trans* X-ray Study

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The typical *cis*–*trans* ambiguity for two spiro-oxazolone derivatives has been solved. The compounds ( $C_{17}H_{13}O_2N$ ) crystallize in space groups  $P2_1/c$  and  $P\bar{1}$ , respectively, with  $Z = 4$  and 2, in unit cells of dimensions:  $a = 5.988$  (2),  $b = 10.703$  (4),  $c = 21.338$  (8) Å,  $\beta = 94.12$  (2)° and  $a = 11.099$  (2),  $b = 11.182$  (2),  $c = 5.482$  (1) Å,  $\alpha = 82.39$  (2),  $\beta = 95.43$  (2),  $\gamma = 94.26$  (2)°. Direct methods followed by Fourier and then least-squares analysis, gave final  $R$  factors of 0.045 and 0.042, respectively, for the 970 and 2036 observed reflections. Half-normal probability analysis has been used to compare the two compounds.

### Introduction

At the Centro Nacional de Química Orgánica (CSIC Madrid) a search is being carried out for a suitable synthetic route of 1-aminocyclopropane carboxylic acids, compounds that are believed to be of interest for interfering with the biochemistry of naturally occurring amino acids. In particular 1-amino-2-arylcyclopropane carboxylic acids could be useful as substrates as well as inhibitors of dopadecarboxylase, a key enzyme in the synthesis of biogenic amines. One of the pathways studied was the known oxazolone synthesis of amino acids. It was found that the *cis* and *trans* isomers of 2-phenyl-4-benzylideneoxazolin-5-one, on

Table 1. Crystallographic data for *cis*- and *trans*-DOAS

	<i>cis</i> -DOAS	<i>trans</i> -DOAS
Formula	$C_{17}H_{13}O_2N$	$C_{17}H_{13}O_2N$
Space group	$P2_1/c$	$P\bar{1}$
$Z$	4	2
$\mu$	0.89 cm <sup>-1</sup>	0.89 cm <sup>-1</sup>
Size	0.1 × 0.2 × 0.3 mm	0.6 × 0.2 × 0.3 mm
$a$	5.988 (2) Å	11.099 (2) Å
$b$	10.703 (4)	11.182 (2)
$c$	21.338 (8)	5.4816 (5)
$\alpha$	—	82.39 (2)°
$\beta$	94.12 (1)°	95.43 (2)
$\gamma$	—	94.26 (2)
$V$	1364.0 (8) Å <sup>3</sup>	670.0 (5) Å <sup>3</sup>

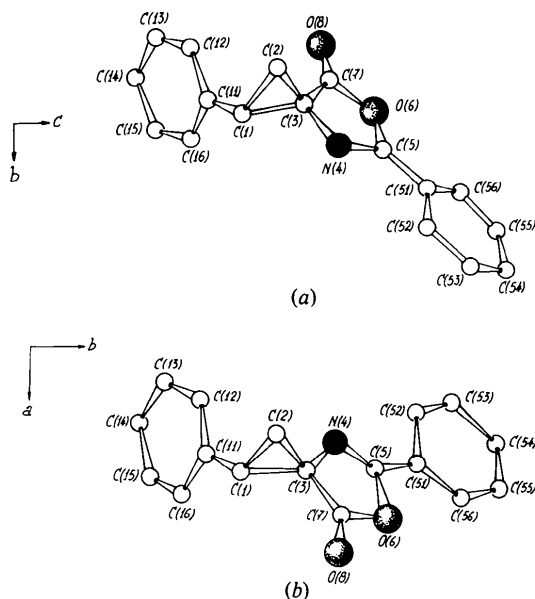


Fig. 1. The two molecular formulae: (a) *cis*-DOAS, (b) *trans*-DOAS, with the atomic numbering.

treatment with diazomethane, give two different compounds (Bernabe, Fernández-Alvarez & Penadés-Ullate, 1972). Spectroscopically they were suggested to be the two spiro compounds illustrated in Fig. 1, as corresponding to the *cis* and *trans* derivatives, but no conclusive evidence could be stated, and so this X-ray work was undertaken for *cis*-DOAS and *trans*-DOAS, namely *cis*- and *trans*-1,5-diphenyl-6-oxa-4-aza-spiro[2.4]hept-4-en-7-one. This paper confirms the supposed configurations.

### Experimental

Table 1 shows the crystallographic data for both compounds. The lattice parameters were taken from the least-squares fit of the diffractometer settings of 35 reflections up to  $25^\circ$  in  $\theta$  for both crystals. Density was measured by flotation in aqueous KI solution. Intensity data were collected in both cases on a Philips PW 1100 automatic diffractometer, in the  $\omega$ - $2\theta$  scan mode, with Mo  $K\alpha$  radiation monochromatized by graphite. No

Table 2. Fractional atomic coordinates for both compounds ( $\times 10^4$ ), with the least-squares *e.s.d.*'s in parentheses

	<i>cis</i> -DOAS			<i>trans</i> -DOAS		
	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	4572 (7)	-225 (5)	2619 (2)	2840 (2)	4852 (2)	13276 (3)
C(2)	6003 (8)	-1165 (5)	2952 (3)	2037 (2)	5496 (2)	14586 (3)
C(3)	4577 (7)	-366 (5)	3341 (2)	2759 (2)	6236 (2)	12649 (3)
N(4)	5445 (6)	637 (4)	3733 (2)	2199 (1)	7763 (1)	10356 (3)
C(5)	4118 (8)	735 (5)	4172 (3)	2846 (1)	7734 (1)	9780 (3)
O(6)	2346 (5)	-111 (3)	4150 (2)	3854 (1)	7970 (1)	11396 (2)
C(7)	2619 (8)	-859 (5)	3629 (3)	3824 (2)	7031 (2)	13317 (3)
O(8)	1399 (6)	-1715 (3)	3438 (2)	4570 (1)	6967 (1)	15044 (3)
C(11)	2532 (8)	-518 (5)	2206 (2)	2406 (2)	4046 (1)	11380 (3)
C(12)	2128 (9)	-1687 (5)	1967 (3)	1188 (2)	3774 (2)	10729 (4)
C(13)	222 (10)	-1930 (5)	1573 (3)	840 (2)	3027 (2)	8942 (4)
C(14)	-1264 (9)	-989 (6)	1424 (3)	1696 (2)	2533 (2)	7782 (4)
C(15)	-869 (9)	190 (5)	1649 (3)	2912 (2)	2785 (2)	7429 (4)
C(16)	1019 (8)	418 (4)	2044 (2)	3263 (2)	3534 (2)	10215 (4)
C(51)	4162 (8)	1639 (5)	4675 (2)	2556 (2)	8628 (1)	7590 (3)
C(52)	5919 (8)	2496 (5)	4735 (2)	1585 (2)	8524 (2)	6069 (4)
C(53)	5944 (10)	3402 (6)	5189 (3)	1393 (2)	9345 (2)	3976 (4)
C(54)	4234 (11)	3498 (6)	5586 (3)	2271 (2)	10264 (2)	3389 (4)
C(55)	2513 (12)	2651 (7)	5532 (3)	3310 (2)	10371 (2)	4886 (4)
C(56)	2463 (10)	1720 (6)	5084 (3)	3515 (2)	9563 (2)	6999 (4)
H(1)	5325 (59)	600 (34)	2525 (17)	3642 (17)	4664 (17)	14193 (33)
H(2A)	5461 (60)	-2018 (34)	2946 (17)	1144 (18)	5429 (18)	14094 (36)
H(2B)	7520 (62)	-987 (37)	3020 (18)	2266 (19)	5620 (19)	16243 (38)
H(12)	3036 (67)	-2342 (39)	2059 (18)	557 (20)	4126 (21)	11517 (41)
H(13)	-29 (68)	-2776 (39)	1416 (19)	-3 (19)	2859 (19)	8541 (38)
H(14)	-2499 (78)	-1222 (44)	1154 (22)	1452 (19)	2019 (20)	6496 (40)
H(15)	-1818 (71)	898 (40)	1509 (20)	3509 (20)	2408 (21)	7599 (42)
H(16)	1298 (60)	1277 (33)	2207 (17)	4118 (19)	3709 (19)	10669 (39)
H(52)	7038 (64)	2429 (37)	4432 (18)	994 (19)	7901 (19)	6498 (39)
H(53)	7161 (89)	4020 (49)	5180 (24)	631 (23)	9262 (23)	2977 (48)
H(54)	4322 (77)	4203 (45)	5892 (22)	2081 (22)	10824 (23)	2007 (46)
H(55)	1390 (78)	2700 (43)	5808 (21)	3923 (23)	11036 (24)	4530 (46)
H(56)	1243 (78)	1145 (46)	5064 (23)	4248 (21)	9628 (21)	8103 (43)

decomposition was detected from the standard reflections monitored every 90 min. Although the data for both compounds were collected with the same conditions up to  $27^\circ$  in  $\theta$ , the numbers of observed reflections were 970 and 2036, respectively, with a  $2\sigma(\text{counting})$  criterion. Lorentz and polarization correction factors were applied, but none for absorption. The normalized  $E$ -factor distribution clearly suggested a centrosymmetric structure for the *trans* compound. Through all the calculations the scattering factors for C, O and N were taken from *International Tables for X-ray Crystallography* (1962), while those for H were from Stewart, Davidson & Simpson (1965). Unless otherwise stated, the calculations were performed with the XRAY 70 system of crystallographic programs (Stewart, Kundell & Baldwin, 1970).

The H atoms found from a difference map were included in the refinement with isotropic temperature factors. The refinement had then to be carried out dividing the parameters into two blocks. The weighting schemes employed to give no trends in  $\langle w\Delta(F_o - F_c)^2 \rangle$  vs  $\sin \theta/\lambda$  or  $|F_o|$  were: *cis*:  $w_{hkl} = 1/1.533\sigma_c^2(F_o)$ ; *trans*:  $w_{hkl} = 0.047/(0.416 - 0.042|F_o|)^2$  if  $|F_o| \leq 3.6$  and  $w_{hkl} = 0.047/(0.140 + 0.028|F_o|)^2$  otherwise. A final difference synthesis gave values for the residual electron density which ranged from  $-0.20$  to  $0.16$  and from  $-0.26$  to  $0.16$  e  $\text{\AA}^{-3}$  for *cis* and *trans*, respectively, with no significant features. The respective final conventional  $R$  factors were 0.045 and 0.045, and  $R_w = [(\sum w\Delta^2/\sum w|F_o|^2)^{1/2}]$  0.055 and 0.054.

Table 2 shows the fractional coordinates for both compounds, related to the numbering expressed in Fig. 1.\*

### Determination of the structure

Both compounds were examined systematically in parallel ways. Multisolution tangent formula analysis (*MULTAN*; Main, Woolfson, Lessinger, Germain & Declercq, 1974) phased the data to give the structures in a Fourier map, confirming the proposed configurations. The positional models were then refined by least-squares procedures in the anisotropic, full-matrix mode.

### Description and discussion

Table 3 shows the molecular geometry for both compounds. It may be noticed that the least-squares

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33030 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for *cis*- and *trans*-DOAS with the least-squares *e.s.d.*'s in parentheses

	<i>cis</i> -DOAS	<i>trans</i> -DOAS		<i>cis</i> -DOAS	<i>trans</i> -DOAS
C(1)–C(2)	1.471 (7)	1.478 (3)	C(7)–O(6)–C(5)	105.6 (4)	105.4 (1)
C(1)–C(3)	1.548 (7)	1.548 (3)	O(6)–C(5)–N(4)	115.7 (5)	116.6 (1)
C(2)–C(3)	1.501 (7)	1.511 (3)	C(5)–N(4)–C(3)	105.2 (4)	105.2 (1)
C(3)–C(7)	1.461 (7)	1.473 (2)	C(3)–C(7)–O(8)	131.6 (5)	132.3 (2)
C(3)–N(4)	1.435 (6)	1.427 (2)	O(6)–C(7)–O(8)	122.6 (5)	122.5 (2)
N(4)–C(5)	1.276 (7)	1.269 (2)	C(11)–C(1)–C(2)	124.6 (5)	124.3 (2)
C(5)–O(6)	1.393 (6)	1.392 (2)	C(11)–C(1)–C(3)	120.8 (4)	119.7 (1)
O(6)–C(7)	1.388 (7)	1.386 (2)	C(2)–C(3)–N(4)	123.5 (4)	122.1 (2)
C(7)–C(8)	1.197 (6)	1.194 (2)	C(1)–C(3)–C(7)	120.7 (4)	118.6 (2)
C(1)–C(11)	1.488 (6)	1.487 (3)	C(1)–C(3)–N(4)	118.9 (4)	122.2 (2)
C(5)–C(51)	1.443 (7)	1.466 (2)	C(2)–C(3)–C(7)	122.2 (5)	121.9 (2)
C(11)–C(12)	1.367 (8)	1.387 (2)	N(4)–C(5)–C(51)	128.4 (5)	127.3 (2)
C(12)–C(13)	1.393 (8)	1.381 (3)	O(6)–C(5)–C(51)	115.8 (4)	116.1 (1)
C(13)–C(14)	1.366 (8)	1.378 (3)	C(1)–C(11)–C(12)	122.0 (5)	123.2 (2)
C(14)–C(15)	1.365 (8)	1.380 (3)	C(1)–C(11)–C(16)	119.5 (5)	118.4 (2)
C(15)–C(16)	1.382 (7)	1.382 (3)	C(5)–C(51)–C(52)	118.9 (5)	118.7 (2)
C(16)–C(15)	1.377 (7)	1.391 (3)	C(5)–C(51)–C(56)	122.2 (5)	121.6 (2)
C(51)–C(52)	1.395 (7)	1.390 (3)	C(11)–C(12)–C(13)	120.8 (5)	120.6 (2)
C(52)–C(53)	1.370 (8)	1.382 (3)	C(12)–C(13)–C(14)	119.6 (5)	120.6 (2)
C(53)–C(54)	1.379 (9)	1.383 (3)	C(13)–C(14)–C(15)	120.4 (5)	119.5 (2)
C(54)–C(55)	1.371 (10)	1.366 (3)	C(14)–C(15)–C(16)	119.5 (5)	120.0 (2)
C(55)–C(56)	1.379 (9)	1.383 (3)	C(15)–C(16)–C(11)	121.3 (5)	120.9 (2)
C(56)–C(51)	1.390 (8)	1.390 (3)	C(16)–C(11)–C(12)	118.5 (5)	118.4 (2)
			C(51)–C(52)–C(53)	120.0 (5)	119.8 (2)
C(2)–C(1)–C(3)	59.6 (4)	59.9 (1)	C(52)–C(53)–C(54)	121.2 (6)	120.2 (2)
C(1)–C(3)–C(2)	57.7 (3)	57.8 (1)	C(53)–C(54)–C(55)	119.0 (6)	120.2 (2)
C(3)–C(2)–C(1)	62.8 (3)	62.4 (1)	C(54)–C(55)–C(56)	121.0 (6)	120.4 (2)
N(4)–C(3)–C(7)	107.0 (4)	107.5 (1)	C(55)–C(56)–C(51)	120.0 (6)	119.8 (2)
C(3)–C(7)–O(6)	105.8 (4)	105.2 (1)	C(56)–C(51)–C(52)	118.8 (5)	119.6 (2)

e.s.d.'s are larger for *cis*-DOAS than for *trans*-DOAS as a consequence of the deviations in atomic coordinates (average of 0.005 vs 0.003 Å), which we suppose to be due to the different numbers of observed reflections. This taken into account, and considering both results as coming from independent experiments with the same technique, we have calculated the half-normal probability distribution (Abrahams & Keve, 1971), for all intramolecular distances, excluding those involved in the different configuration (Fig. 2). The plot is linear with zero intercept and slope of 1.30, showing the small underestimation in the least-squares e.s.d.'s.

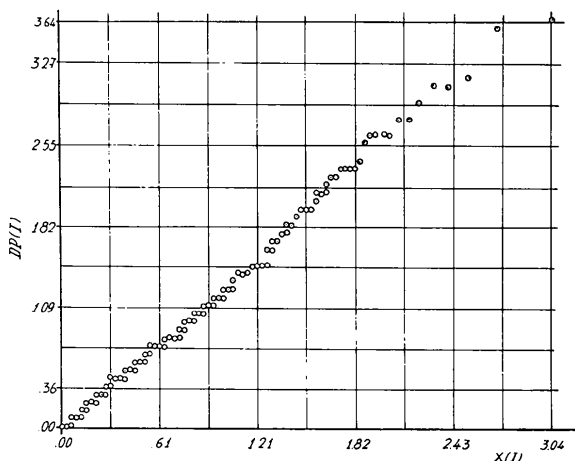


Fig. 2. The half-normal probability plot for the corresponding intramolecular distances.

So it seems there is no significant difference between the two molecules, within the experimental error; moreover, there seems to exist good agreement between some individual bond distances, the C—C bonds being the more susceptible to variations.

The bonds of the cyclopropane rings show a shortening from the normal  $C(sp^3)-C(sp^3)$  bond because of the triangular bending, which is also reflected in the geometry outside the cyclopropane ring, as is recorded in the literature (Riche & Pascard-Billy, 1975; Lemley, Skarstad & Hughes, 1976; Grant & Speakman, 1958; Carlström, 1975; Schenk, 1971; Beintema, 1976). The differences within the ring have to be referred to the difference in degree of substitution at each corner (Meester, Schenk & MacGillavry, 1971), the effect of which may also be noticed in the areas of the triangles, 0.986 and 0.991 Å<sup>2</sup>, which are between the theoretical values of 0.977 Å<sup>2</sup> for cyclopropane and 0.996 Å<sup>2</sup> for cyclopropylidene (Lathan, Radon, Hariharan, Hehre & Pople, 1973).

The bond distances and angles in the five-membered ring are within the range of values found in the literature (Cotrait, Sy & Ptak, 1975; Martinez-Ripoll & Lorenz, 1973, 1974, 1976*a,b*; Riche, 1973).

For the phenyl groups the bond lengths are in the range 1.365–1.395 Å with a mean value of 1.382 (2) Å, and the angles average 120.0 (2)° (Tranqui & Fillion, 1972; Griffith & Robertson, 1972; Iwasaki & Iwasaki, 1972). The C—H distances (not shown) were normal, ranging from 0.90 to 1.00 Å, and angles from 115 to 125°.

Table 4. Some conformational data for *cis*- and *trans*-DOAS

(a) Deviations (Å) of some atoms from the plane through the asterisked atoms

	<i>cis</i>	<i>trans</i>		<i>cis</i>	<i>trans</i>
C(11)*	0.003	0.005	C(51)*	-0.006	-0.004
C(12)*	-0.003	-0.003	C(52)*	-0.002	0.002
C(13)*	-0.003	-0.001	C(53)*	0.009	0.003
C(14)*	0.008	0.004	C(54)*	-0.008	-0.003
C(15)*	-0.008	-0.002	C(55)*	-0.000	-0.002
C(16)*	0.002	-0.003	C(56)*	0.007	0.005
C(1)	-0.021	0.013	C(5)	-0.085	-0.030

	<i>cis</i>	<i>trans</i>		<i>cis</i>	<i>trans</i>	
C(1)*	—	—	C(3)*	-0.002	C(3)*	-0.001
C(2)*	—	—	N(4)*	0.003	N(4)*	0.001
C(3)*	—	—	C(5)*	-0.003	C(5)*	-0.001
H(1)	0.91	0.89	O(6)*	0.002	C(7)*	0.001
H(2 <i>a</i> )	-0.85	-0.86	C(7)	0.039	O(6)	0.023
H(2 <i>b</i> )	0.80	0.79				

(b) Torsion angles (°) with the usual Klyne & Prelog (1960) criterion

	<i>cis</i>	<i>trans</i>		<i>cis</i>	<i>trans</i>
C(3)—N(4)—C(5)—O(6)	0.7 (5)	0.9 (2)	C(7)—C(3)—N(4)—C(5)	-2.1 (5)	0.2 (2)
N(4)—C(5)—O(6)—C(7)	1.1 (4)	-1.7 (2)	C(12)—C(11)—C(1)—C(2)	-17.6 (7)	-3.1 (2)
C(5)—O(6)—C(7)—C(3)	-2.3 (5)	1.6 (2)	C(12)—C(11)—C(1)—C(3)	-89.9 (5)	-75.0 (2)
O(6)—C(7)—C(3)—N(4)	2.7 (4)	-1.1 (2)			

Table 4 describes some conformational parameters. The angles between the cyclopropane and the five-membered ring are 89 and 88° for *cis*- and *trans*-DOAS, respectively, while the angles between the five-membered ring and the nearest phenyl are 7 and 9°. The pentagonal ring is almost planar, the conformational parameters (Altona, Geise & Romers, 1968) being  $\Delta = 8.3$  and 318.2°,  $\varphi_m = 2.7$  and 1.8° for *cis*- and *trans*-DOAS, respectively, with a mean bond angle of 108° for both compounds (Dunitz, 1972).

No significant distances less than 4 Å were found, the packing being due only to van der Waals forces.

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#### References

- ABRAHAMS, S. C. & KEVE, E. T. (1971). *Acta Cryst.* **A27**, 157–165.
- ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–32.
- BEINTEMA, J. (1976). *Acta Cryst.* **B32**, 1631–1634.
- BERNABE, M., FERNÁNDEZ-ALVAREZ, E. & PENADÉS-ULLATE, S. (1972). *An. Quím.* **68**, 501–522.
- CARLSTRÖM, D. (1975). *Acta Cryst.* **B31**, 2185–2188.
- COTRAIT, M., SY, D. & PTAK, M. (1975). *Acta Cryst.* **B31**, 1869–1874.
- DUNITZ, J. D. (1972). *Tetrahedron*, **28**, 5459–5467.
- GRANT, W. K. & SPEAKMAN, J. C. (1958). *J. Chem. Soc.* pp. 3753–3760.
- GRIFFITH, E. A. H. & ROBERTSON, B. E. (1972). *Acta Cryst.* **B28**, 3377–3384.
- International Tables for X-ray Crystallography* (1962). Vol. III, p. 202. Birmingham: Kynoch Press.
- IWASAKI, F. & IWASAKI, H. (1972). *Acta Cryst.* **B28**, 3370–3376.
- KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.
- LATHAN, W. H., RADON, L., HARIHARAN, P. C., HEHRE, W. J. & POPLÉ, J. A. (1973). *Topics in Current Chemistry*. Vol. 40. *Three-Membered Rings*, edited by DAVIDSON *et al.*, pp. 1–46. Berlin: Springer.
- LEMLEY, J. T., SKARSTAD, P. M. & HUGHES, R. E. (1976). *Acta Cryst.* **B32**, 35–40.
- MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MARTÍNEZ-RIPOLL, M. & LORENZ, H. P. (1973). *Acta Cryst.* **B29**, 2260–2263.
- MARTÍNEZ-RIPOLL, M. & LORENZ, H. P. (1974). *Acta Cryst.* **B30**, 793–796.
- MARTÍNEZ-RIPOLL, M. & LORENZ, H. P. (1976a). *Acta Cryst.* **B32**, 2322–2325.
- MARTÍNEZ-RIPOLL, M. & LORENZ, H. P. (1976b). *Acta Cryst.* **B32**, 2325–2328.
- MEESTER, M. A. M., SCHENK, H. & MACGILLAVRY, C. H. (1971). *Acta Cryst.* **B27**, 630–634.
- RICHE, C. (1973). *Acta Cryst.* **B29**, 2147–2153.
- RICHE, C. & PASCARD-BILLY, C. (1975). *Acta Cryst.* **B31**, 2565–2570.
- SCHENK, H. (1971). *Acta Cryst.* **B27**, 185–188.
- STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- TRANQUI, D. & FILLION, H. (1972). *Acta Cryst.* **B28**, 3306–3315.