We thank Professor J. R. Scheffer and Ms L. Walsh for their collaborative studies on the solid-state photochemistry, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

References

Appel, W. K., Jiang, Z. Q., Scheffer, J. R. & Walsh, L. (1983). *J. Am. Chem. Soc.* **105**, 5354–5363.

Ariel, S., Askari, S., Scheffer, J. R., Trotter, J. & Walsh, L. (1984). *J. Am. Chem. Soc.* **106**, 5726–5728.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Greenhough, T. J. & Trotter, J. (1980). *Acta Cryst.* B**36**, 1831–1835.

Phillips, S. E. V. & Trotter, J. (1976). *Acta Cryst.* B**32**, 3088–3091.

Secco, A. S. & Trotter, J. (1983). C**39**, 1665–1667.

Sheldrick, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Trotter, J. (1983). *Acta Cryst.* B**39**, 373–381.

Acta Cryst. (1985). C**41**, 298–300

Diphthalimide Phthalate,* $C_{24}H_{12}N_2O_8$

By J. W. Bats

Institut für Kristallographie und Mineralogie der Universität, Senckenberganlage 30, *D*-6000 *Frankfurt/Main* 1, *Federal Republic of Germany*

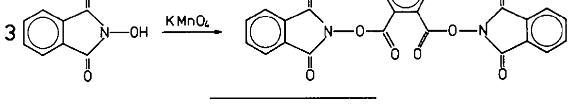
and H.-J. Teuber

Institut für Organische Chemie, Niederurseler Hang, D-6000 *Frankfurt/Main* 50, *Federal Republic of Germany*

(*Received* 27 *June* 1984; *accepted* 1 *October* 1984)

Abstract. $M_r = 456{\cdot}37$, triclinic, $P\bar{1}$, $a = 8{\cdot}025$ (2), $b = 12{\cdot}910$ (1), $c = 20{\cdot}589$ (2) Å, $\alpha = 73{\cdot}90$ (1), $\beta = 88{\cdot}72$ (1), $\gamma = 77{\cdot}87$ (1)°, $V = 2002{\cdot}1$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}514$ (1) g cm^{-3}, Mo $K\alpha$ radiation, graphite monochromator, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}1$ cm^{-1}, $F(000) = 936$, $T = 296$ K, $R = 0{\cdot}046$ for 3938 independent reflections. The two independent molecules differ only in the relative orientation of one of the side groups. One carboxyl group is coplanar with the central phenyl group, the other forms an angle of 101·2 (4)° (I) and 111·2 (4)° (II). The phthalimide groups are almost perpendicular to the carboxyl groups. The packing shows stacks of parallel phthalimide groups along **a**.

Introduction. The title compound (m.p. 497–498 K) was obtained by the oxidation of *N*-hydroxyphthalimide by $KMnO_4$ in acetone. The reaction occurs by dehydrogenation and oxidative elimination of nitrogen from each third molecule of the starting material. The product corresponds in some ways to the well known class of activated esters. The formation of such a compound in the present case is quite unusual.



As the nature of the reaction product was not clear from a chemical analysis, a crystal structure determination was undertaken.

Experimental. Colorless transparent lath-shaped crystals from acetone, sample dimensions 0·075 × 0·25 × 0·62 mm, cell constants from setting angles of 25 reflections with $7 < \theta < 13°$, CAD-4 diffractometer, hemisphere up to $2\theta = 42°$, $0 \le h \le 8$, $-12 \le k \le 13$, $-20 \le l \le 20$, $(\sin\theta/\lambda)_{\max} = 0{\cdot}50$ Å^{-1}, ω scan, total number of reflections 4811, independent reflections 4382; three standards every 5000 s remained stable; empirical absorption correction based on ψ scans (North, Phillips & Mathews, 1968), correction range 1·00–1·035; internal agreement of equivalent reflections $R_{\rm int} = 0{\cdot}02$, 3938 reflections with $I > 0{\cdot}5\sigma(I)$ used, $w(I) = 1/[\sigma^2(I) + (0{\cdot}03I)^2]$, structure determination by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), H atoms from difference synthesis and refined with fixed isotropic thermal parameters, refinement on F, extinction parameter $g = 37$ (4) $\times 10^{-8}$, the two independent molecules refined in alternating cycles, peaks in final difference synthesis less than 0·22 e Å^{-3}, $(\Delta/\sigma)_{\max} = 0{\cdot}06$, $R = 0{\cdot}046$, $wR = 0{\cdot}040$, $S = 1{\cdot}52$, scattering factors from *International Tables for X-ray Crystallography* (1974), calculations performed with the *SDP* program system (Enraf–Nonius, 1982).

---

* Bis(1,3-dioxo-2*H*-isoindol-2-yl) phthalate.

0108-2701/85/020298-03$01.50

Table 1. *Positional parameters and equivalent values of anisotropic thermal parameters*

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq} (Å^2)		x	y	z	B_{eq} (Å^2)
O(1)	0·2460 (2)	0·1933 (1)	0·0940 (1)	6·01 (5)	C(15)	−0·2075 (3)	−0·1033 (2)	0·2517 (1)	4·03 (6)
O(2)	0·4819 (2)	0·2448 (1)	0·04903 (9)	5·38 (4)	C(16)	−0·0316 (3)	−0·1431 (2)	0·2320 (1)	5·13 (7)
O(3)	0·1034 (2)	0·0008 (2)	0·07607 (8)	5·95 (5)	C(17)	0·2550 (3)	0·5967 (2)	0·0994 (1)	5·25 (7)
O(4)	0·1892 (2)	−0·0445 (1)	0·18446 (7)	5·08 (4)	C(18)	0·1621 (3)	0·6964 (2)	0·0609 (2)	6·12 (8)
O(5)	0·4553 (2)	0·3511 (1)	0·15370 (9)	6·36 (5)	C(19)	0·1118 (3)	0·7099 (2)	−0·0045 (2)	6·59 (8)
O(6)	0·2948 (3)	0·3965 (2)	−0·0649 (1)	8·55 (6)	C(20)	0·1528 (4)	0·6251 (2)	−0·0349 (1)	6·13 (8)
O(7)	−0·0793 (2)	0·1421 (1)	0·17460 (9)	6·44 (5)	C(21)	−0·4043 (3)	0·0688 (2)	0·2438 (1)	4·93 (7)
O(8)	0·0513 (3)	−0·2336 (2)	0·2389 (1)	8·59 (7)	C(22)	−0·5230 (3)	0·0083 (2)	0·2743 (1)	5·49 (7)
O(9)	0·2932 (2)	0·7013 (1)	0·40053 (9)	6·33 (5)	C(23)	−0·4851 (3)	−0·1044 (2)	0·2923 (1)	5·51 (7)
O(10)	0·0486 (2)	0·7883 (1)	0·43438 (9)	5·55 (4)	C(24)	−0·3262 (3)	−0·1637 (2)	0·2817 (1)	5·35 (7)
O(11)	0·4172 (2)	0·4684 (2)	0·41002 (9)	6·56 (5)	C(25)	0·1482 (3)	0·7029 (2)	0·4135 (1)	4·06 (6)
O(12)	0·3039 (2)	0·5050 (1)	0·30460 (8)	4·89 (4)	C(26)	0·0452 (3)	0·6223 (2)	0·4107 (1)	3·41 (5)
O(13)	0·0807 (2)	0·9921 (1)	0·34244 (8)	5·43 (4)	C(27)	−0·1237 (3)	0·6373 (2)	0·4299 (1)	4·32 (6)
O(14)	0·1804 (3)	0·7798 (1)	0·56098 (9)	6·79 (5)	C(28)	−0·2228 (3)	0·5630 (2)	0·4275 (1)	4·38 (6)
O(15)	0·5282 (3)	0·6419 (1)	0·2431 (1)	6·92 (6)	C(29)	−0·1555 (3)	0·4727 (2)	0·4068 (1)	4·28 (6)
O(16)	0·4815 (2)	0·2835 (1)	0·32473 (8)	5·67 (5)	C(30)	0·0125 (3)	0·4556 (2)	0·3872 (1)	4·41 (6)
N(1)	0·3969 (3)	0·3516 (1)	0·0447 (1)	4·88 (5)	C(31)	0·1131 (3)	0·5295 (2)	0·3893 (1)	3·56 (5)
N(2)	0·0192 (2)	−0·0460 (2)	0·20041 (9)	4·59 (5)	C(32)	0·2964 (3)	0·5002 (2)	0·3723 (1)	4·30 (6)
N(3)	0·1384 (3)	0·8622 (1)	0·4464 (1)	4·72 (5)	C(33)	0·1336 (3)	0·9646 (2)	0·4002 (1)	3·91 (6)
N(4)	0·4693 (2)	0·4696 (2)	0·2861 (1)	5·06 (5)	C(34)	0·2081 (3)	1·0257 (2)	0·4393 (1)	3·27 (5)
C(1)	0·3862 (3)	0·1667 (2)	0·0765 (1)	3·83 (6)	C(35)	0·2357 (3)	0·9617 (2)	0·5058 (1)	3·53 (5)
C(2)	0·4816 (3)	0·0550 (2)	0·0814 (1)	3·14 (5)	C(36)	0·1845 (3)	0·8561 (2)	0·5125 (1)	4·50 (6)
C(3)	0·6523 (3)	0·0339 (2)	0·0651 (1)	3·74 (6)	C(37)	0·5720 (3)	0·5435 (2)	0·2565 (1)	4·64 (6)
C(4)	0·7405 (3)	−0·0721 (2)	0·0730 (1)	4·03 (6)	C(38)	0·7333 (3)	0·4707 (2)	0·2475 (1)	4·08 (6)
C(5)	0·6640 (3)	−0·1595 (2)	0·0980 (1)	4·29 (6)	C(39)	0·7207 (3)	0·3630 (2)	0·2732 (1)	3·89 (6)
C(6)	0·4925 (3)	−0·1398 (2)	0·1142 (1)	4·22 (6)	C(40)	0·5475 (3)	0·3592 (2)	0·2988 (1)	4·22 (6)
C(7)	0·4017 (3)	−0·0339 (2)	0·1053 (1)	3·28 (5)	C(41)	0·2495 (3)	1·1274 (2)	0·4186 (1)	4·07 (6)
C(8)	0·2147 (3)	−0·0204 (2)	0·1172 (1)	3·79 (6)	C(42)	0·3201 (3)	1·1631 (2)	0·4672 (1)	4·40 (6)
C(9)	0·3924 (3)	0·3980 (2)	0·0983 (1)	4·45 (6)	C(43)	0·3459 (3)	1·0997 (2)	0·5335 (1)	4·45 (6)
C(10)	0·2957 (3)	0·5115 (2)	0·0700 (1)	4·02 (6)	C(44)	0·3040 (3)	0·9975 (2)	0·5542 (1)	4·31 (6)
C(11)	0·2457 (3)	0·5253 (2)	0·0040 (1)	4·46 (6)	C(45)	0·8833 (3)	0·4986 (2)	0·2205 (1)	5·59 (7)
C(12)	0·3084 (3)	0·4216 (2)	−0·0138 (1)	5·33 (7)	C(46)	1·0181 (3)	0·4123 (2)	0·2206 (1)	6·08 (8)
C(13)	−0·0989 (3)	0·0506 (2)	0·1993 (1)	4·37 (6)	C(47)	1·0065 (3)	0·3046 (2)	0·2460 (1)	5·73 (7)
C(14)	−0·2469 (3)	0·0101 (2)	0·2329 (1)	3·90 (6)	C(48)	0·8561 (3)	0·2774 (2)	0·2736 (1)	5·00 (7)

Discussion. The positional parameters are reported in Table 1,* the bond lengths and most relevant angles in Table 2, and the geometries of the two independent molecules are shown in Fig. 1.

The bond lengths and angles in the two independent molecules are very similar. Small, but significant differences among related groups are only found in the carboxyl groups and can be related to the relative orientation of these groups with respect to the benzene rings. The dimensions of the four phthalimide groups are in good agreement with those in crystalline phthalimide (Matzat, 1972), except for the C—N—C angle which is on the average 2·6 (4)° larger in the title compound and may result from the substitution at N.

The average N—O bond length of 1·390 (2) Å corresponds to a single bond. Each molecule contains five planar parts: the central phenyl ring, two carboxyl groups and two phthalimide groups (see deposition footnote).

To avoid short O···O repulsions, the phthalimide groups have to be approximately perpendicular to the plane of the nearest carboxyl groups. The observed dihedral angles of 95·7 (4), 104·6 (4), 93·8 (4) and 99·7 (4)° agree with values found in other phthalimides

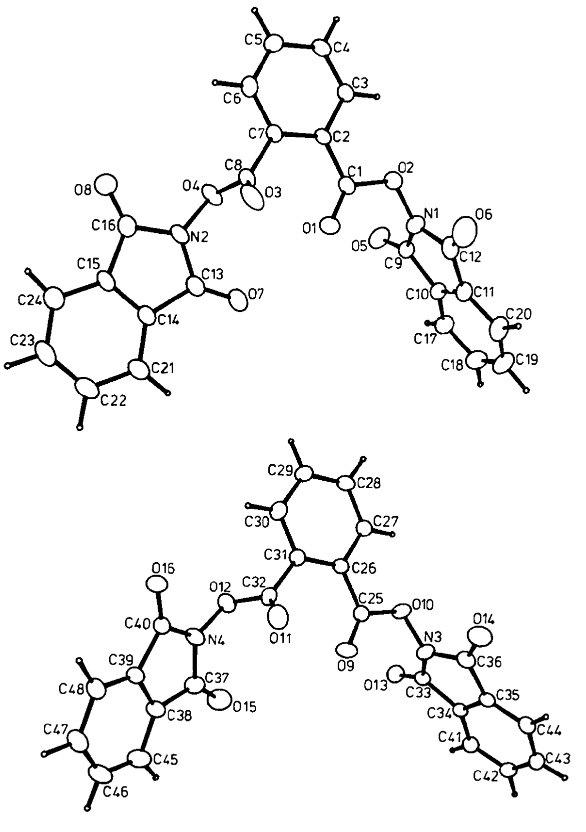


Fig. 1. Perspective view of the geometries of the two independent molecules.

---

* Lists of anisotropic thermal parameters, hydrogen-atom coordinates, bond angles, least-squares planes, and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39794 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

substituted at N with planar groups (Moor, Grieb, Niggli, Hoesch & Dreiding, 1979; Barlow, Davidson, Lewis & Russell, 1979). For the same reason the two carboxyl groups cannot be coplanar.

Table 2. *Bond distances* (Å) *and bond angles* (°) *excluding phenyl rings*

O(1)—C(1)	1·184 (2)	O(9)—C(25)	1·186 (2)
O(2)—N(1)	1·383 (2)	O(10)—N(3)	1·386 (2)
O(2)—C(1)	1·384 (2)	O(10)—C(25)	1·384 (2)
O(3)—C(8)	1·180 (2)	O(11)—C(32)	1·184 (2)
O(4)—N(2)	1·399 (2)	O(12)—N(4)	1·391 (2)
O(4)—C(8)	1·354 (2)	O(12)—C(32)	1·378 (2)
O(5)—C(9)	1·199 (2)	O(13)—C(33)	1·202 (2)
O(6)—C(12)	1·197 (3)	O(14)—C(36)	1·198 (2)
O(7)—C(13)	1·192 (2)	O(15)—C(37)	1·198 (2)
O(8)—C(16)	1·189 (3)	O(16)—C(40)	1·198 (2)
N(1)—C(9)	1·391 (3)	N(3)—C(33)	1·392 (3)
N(1)—C(12)	1·391 (3)	N(3)—C(36)	1·393 (3)
N(2)—C(13)	1·394 (3)	N(4)—C(37)	1·395 (3)
N(2)—C(16)	1·383 (3)	N(4)—C(40)	1·385 (3)
C(1)—C(2)	1·461 (3)	C(25)—C(26)	1·472 (3)
C(2)—C(3)	1·391 (3)	C(26)—C(27)	1·392 (3)
C(2)—C(7)	1·397 (2)	C(26)—C(31)	1·391 (2)
C(3)—C(4)	1·370 (3)	C(27)—C(28)	1·380 (3)
C(4)—C(5)	1·367 (3)	C(28)—C(29)	1·354 (3)
C(5)—C(6)	1·396 (3)	C(29)—C(30)	1·389 (3)
C(6)—C(7)	1·372 (3)	C(30)—C(31)	1·382 (3)
C(7)—C(8)	1·497 (3)	C(31)—C(32)	1·499 (3)
C(9)—C(10)	1·469 (3)	C(33)—C(34)	1·483 (3)
C(10)—C(11)	1·379 (3)	C(34)—C(35)	1·384 (2)
C(10)—C(17)	1·375 (3)	C(34)—C(41)	1·372 (3)
C(11)—C(12)	1·471 (3)	C(35)—C(36)	1·476 (3)
C(11)—C(20)	1·381 (3)	C(35)—C(44)	1·375 (3)
C(13)—C(14)	1·478 (3)	C(37)—C(38)	1·473 (3)
C(14)—C(15)	1·375 (3)	C(38)—C(39)	1·369 (3)
C(14)—C(21)	1·379 (3)	C(38)—C(45)	1·388 (3)
C(15)—C(16)	1·485 (3)	C(39)—C(40)	1·481 (3)
C(15)—C(24)	1·385 (3)	C(39)—C(48)	1·376 (3)
C(17)—C(18)	1·378 (3)	C(41)—C(42)	1·387 (3)
C(18)—C(19)	1·367 (4)	C(42)—C(43)	1·379 (3)
C(19)—C(20)	1·385 (4)	C(43)—C(44)	1·380 (3)
C(21)—C(22)	1·388 (3)	C(45)—C(46)	1·380 (3)
C(22)—C(23)	1·367 (3)	C(46)—C(47)	1·366 (3)
C(23)—C(24)	1·389 (3)	C(47)—C(48)	1·393 (3)

N(1)—O(2)—C(1)	113·4 (2)	N(3)—O(10)—C(25)	114·0 (2)
N(2)—O(4)—C(8)	113·3 (2)	N(4)—O(12)—C(32)	112·2 (2)
O(2)—N(1)—C(9)	122·9 (2)	O(10)—N(3)—C(33)	121·7 (2)
O(2)—N(1)—C(12)	122·7 (2)	O(10)—N(3)—C(36)	120·1 (2)
C(9)—N(1)—C(12)	114·4 (2)	C(33)—N(3)—C(36)	114·4 (2)
O(4)—N(2)—C(13)	121·3 (2)	O(12)—N(4)—C(37)	122·0 (2)
O(4)—N(2)—C(16)	121·7 (2)	O(12)—N(4)—C(40)	122·9 (2)
C(13)—N(2)—C(16)	115·5 (2)	C(37)—N(4)—C(40)	115·0 (2)
O(1)—C(1)—O(2)	120·7 (2)	O(9)—C(25)—O(10)	121·8 (2)
O(1)—C(1)—C(2)	127·6 (2)	O(9)—C(25)—C(26)	128·9 (2)
O(2)—C(1)—C(2)	111·7 (2)	O(10)—C(25)—C(26)	109·4 (2)
C(1)—C(2)—C(3)	121·8 (2)	C(25)—C(26)—C(27)	120·3 (2)
C(1)—C(2)—C(7)	119·4 (2)	C(25)—C(26)—C(31)	121·4 (2)
C(2)—C(7)—C(8)	123·3 (2)	C(26)—C(31)—C(32)	123·1 (2)
C(6)—C(7)—C(8)	116·4 (2)	C(30)—C(31)—C(32)	117·0 (2)
O(3)—C(8)—O(4)	123·2 (2)	O(11)—C(32)—O(12)	123·5 (2)
O(3)—C(8)—C(7)	127·0 (2)	O(11)—C(32)—C(31)	127·6 (2)
O(4)—C(8)—C(7)	109·6 (2)	O(12)—C(32)—C(31)	108·7 (2)
O(5)—C(9)—N(1)	125·2 (2)	O(13)—C(33)—N(3)	125·7 (2)
O(5)—C(9)—C(10)	130·9 (2)	O(13)—C(33)—C(34)	130·7 (2)
N(1)—C(9)—C(10)	103·8 (2)	N(3)—C(33)—C(34)	103·6 (2)
C(9)—C(10)—C(11)	109·0 (2)	C(33)—C(34)—C(35)	108·6 (2)
C(9)—C(10)—C(17)	129·9 (2)	C(33)—C(34)—C(41)	130·1 (2)
C(10)—C(11)—C(12)	109·1 (2)	C(34)—C(35)—C(36)	109·1 (2)
C(12)—C(11)—C(20)	129·7 (2)	C(36)—C(35)—C(44)	129·2 (2)
O(6)—C(12)—N(1)	124·5 (2)	O(14)—C(36)—N(3)	125·2 (2)
O(6)—C(12)—C(11)	131·9 (2)	O(14)—C(36)—C(35)	131·1 (2)
N(1)—C(12)—C(11)	103·6 (2)	N(3)—C(36)—C(35)	103·7 (2)
O(7)—C(13)—N(2)	125·7 (2)	O(15)—C(37)—N(4)	124·7 (2)
O(7)—C(13)—C(14)	130·8 (2)	O(15)—C(37)—C(38)	132·1 (2)
N(2)—C(13)—C(14)	103·4 (2)	N(4)—C(37)—C(38)	103·2 (2)
C(13)—C(14)—C(15)	108·5 (2)	C(37)—C(38)—C(39)	109·4 (2)
C(13)—C(14)—C(21)	129·5 (2)	C(37)—C(38)—C(45)	129·1 (2)
C(14)—C(15)—C(16)	109·9 (2)	C(38)—C(39)—C(40)	109·2 (2)
C(16)—C(15)—C(24)	129·0 (2)	C(40)—C(39)—C(48)	129·3 (2)
O(8)—C(16)—N(2)	125·7 (2)	O(16)—C(40)—N(4)	125·0 (2)
O(8)—C(16)—C(15)	131·7 (2)	O(16)—C(40)—C(39)	131·8 (2)
N(2)—C(16)—C(15)	102·7 (2)	N(4)—C(40)—C(39)	103·2 (2)

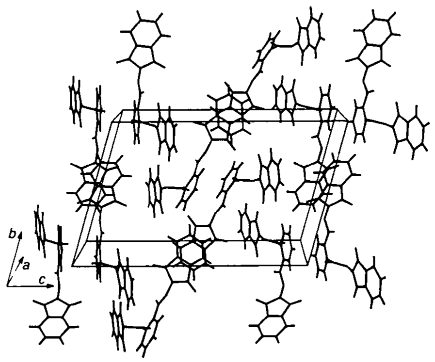


Fig. 2. View along **a*** of the molecular packing.

In the title compound one carboxyl group is approximately coplanar with the central phenyl ring, while the second carboxyl group makes an angle of 101·2 (4)° with the phenyl in molecule I and 111·2 (4)° in molecule II. As a result of this arrangement the C(1)—C(2) and C(25)—C(26) bonds are significantly shorter than the corresponding C(7)—C(8) and C(31)—C(32) bonds, due to overlap of the π systems in the former cases.

The packing of the structure (Fig. 2) shows a number of planar parts to line up between neighboring molecules. Both independent molecules have their central phenyl group parallel to those of their centrosymmetric counterparts. The distance between the planes is 3·512 (6) Å (molecule I) and 3·590 (4) Å (molecule II). Moreover one phthalimide group of each molecule is found to form a stack in the **a** direction. The distances between parallel phthalimide layers range from 3·32 (1) to 3·88 (1) Å. Two of the four N atoms show considerable deviations from planarity. The distances of the N atoms from the planes through their three neighbors are: N(1) 0·001 (3), N(2) 0·098 (3), N(3) 0·158 (3), N(4) 0·024 (3) Å. These differences have to be attributed to packing effects.

References

BARLOW, J. H., DAVIDSON, R. S., LEWIS, A. & RUSSELL, D. R. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 1103–1109.

Enraf–Nonius (1982). *SDP. Structure Determination Package.* Enraf–Nonius, Delft.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MATZAT, E. (1972). *Acta Cryst.* B**28**, 415–418.

MOOR, R., GRIEB, R., NIGGLI, A., HOESCH, L. & DREIDING, A. S. (1979). *Helv. Chim. Acta*, **62**, 1202–1209.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.