Acta Cryst. (1994). C**50**, 1842–1846

Three Substituted 3-Aminopropenoates

Amalija Sinur, Ljubo Golič and Branko Stanovnik

Department of Chemistry and Chemical Technology, University of Ljubljana, Aškerčeva 5, POB 537, 61001 Ljubljana, Slovenia

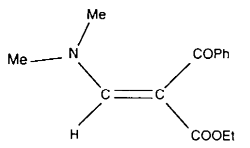
(*Received* 7 *October* 1993; *accepted* 29 *November* 1993)

Abstract

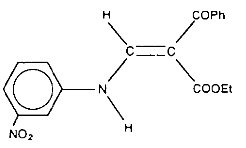
The compounds ethyl (*E*)-2-benzoyl-3-(dimethylamino)-propenoate, $C_{14}H_{17}NO_3$ (I), ethyl (*Z*)-2-benzoyl-3-[(3-nitrophenyl)amino]propenoate, $C_{18}H_{16}N_2O_5$ (II), and methyl (*Z*)-3-[(4,6-dimethyl-2-pyrimidinyl)amino]-2-[(methoxy)(methylthio)methyleneamino]propenoate, $C_{13}H_{18}N_4O_3S$ (III), are propenoates with different substituents at the C(2)=C(3) double bond. C(2) bears benzoyl and ethoxycarbonyl groups in (I) and (II) and (methoxy)(methylthio)methyleneamino and methoxycarbonyl groups in (III), whereas C(3) is substituted with dimethylamino in (I), 3-nitrophenylamino in (II) and 4,6-dimethyl-2-pyrimidinylamino in (III). The configuration with respect to the C(2)=C(3) double bond is *E* in (I) and *Z* in (II) and (III). Bulky substituents such as the benzoyl and (methoxy)(methylthio)methyleneamino groups are turned out of the best plane through the rest of molecule.

Comment

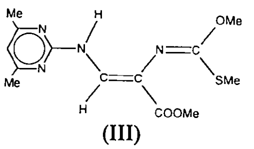
The preparation of compounds (I), (II) and (III) is described in the literature (Breaux & Zwikelmaier, 1981; Svete, Kralj, Stanovnik & Tišler, 1994; Smodiš, Stanovnik & Tišler, 1994). Least-squares planes data, including equations of planes and deviations of atoms from these planes are presented in Table 3. Views of the molecules with atomic numbering are shown in Figs. 1, 2 and 3.



(I)



(II)



(III)

In compound (I), the C(2)=C(3) bond [1.385 (2) Å] is longer than the unweighted mean double-bond distance of 1.340 (13) Å calculated for 211 C=C—C=O (C, H substituted, conjugated) moieties (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). This is probably the result of the conjugation of this double bond with the N-atom lone pair and the carbonyl group of the ethoxycarbonyl moiety. The C(1)—C(2) bond [1.457 (2) Å] is shorter than corresponding unconjugated and even the conjugated C_{sp^2}—C_{sp^2} lengths [1.484 (17) and 1.464 (18) Å, respectively] for C=C—C(=O)(—C*) moieties (Allen *et al.*, 1987). The C(3)—N distance [1.324 (2) Å] is shorter than the mean C_{sp^2}—N(3) length [1.355 (14) Å] in C=C—N—(C#)₂ moieties with N_{sp^2} planar (Allen *et al.*, 1987). The C(2)—C(6) bond length [1.481 (2) Å] corresponds to that of the above unconjugated C_{sp^2}—C_{sp^2} bond. The above observations and the fact that the benzoyl group is turned out of the plane through the rest of the molecule (plane 2 in Table 3) are in agreement with the supposed conjugation. The benzene ring is planar to within 0.009 (3) Å [C(74)]. The torsion angles C(3)=C(2)—C(6)=O(3) −43.0 (2), C(1)—C(2)—C(6)=O(3) 125.7 (1), O(3)=C(6)—C(71)—C(72) 156.4 (1) and O(3)=C(6)—C(71)—C(76) −18.6 (2)° show that the C(6)=O(3) bond is turned out of planes 1 and 2 (defined in Table 3). The angle between these planes is 108.52 (4)°.

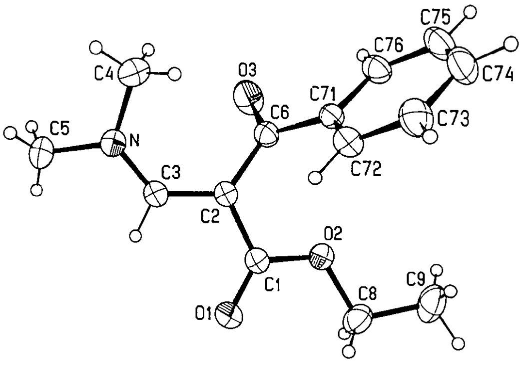


Fig. 1. *ORTEP* (Johnson, 1965) view of (I) showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 50% probability level and the H atoms as small circles of arbitrary radii.

The lengthening of the C(2)=C(3) double bond [1.374 (2) Å] in (II) can also be explained by the delocalization of the electron density towards the C(1)—C(2) and C(3)—N(1) bonds. This is consistent with the shortening of these bonds [1.465 (2) and 1.342 (2) Å, respectively]; the reported distance for a pure C_{sp^2}—N_{sp^2} single bond is 1.470 (5) Å (Camerman, Jensen & Balaban, 1969; Camerman, 1970). The atoms C(6), C(71)–C(76) (plane 3) are coplanar to within 0.012 (3) Å

Acta Crystallographica Section C
ISSN 0108-2701 ©1994

[C(75)], the best plane through them making an angle of 70.37 (4)° with the plane through the rest of the molecule (plane 4). The whole benzoyl group is twisted around the C(6)—C(71) bond; the torsion angles around this bond are C(2)—C(6)—C(71)—C(72) −141.9 (1), C(2)—C(6)—C(71)—C(76) 38.3 (2), O(3)=C(6)—C(71)—C(72) 39.3 (2) and O(3)=C(6)—C(71)—C(76) −140.5 (1)°.

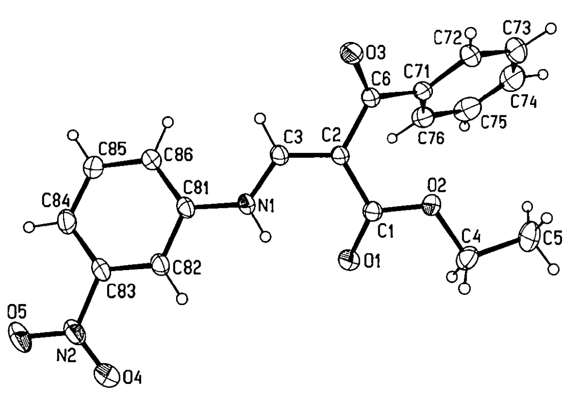


Fig. 2. *ORTEP* (Johnson, 1965) view of (II) showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 50% probability level and the H atoms as small circles of arbitrary radii.

In compound (III), the C(2)=C(3) [1.347 (2) Å], C(1)—C(2) [1.474 (2) Å] C(3)—N(1) [1.374 (2) Å] and C(2)—N(4) [1.417 (2) Å] bond lengths are close to those of the corresponding unconjugated bonds, so the canonical structures which consider electron delocalization are less important. The (methoxy)(methylthio)methyleneamino group deviates from planarity; the largest deviation is 0.122 (3) Å for C(11). The angle between this plane and the plane through the rest of molecule (plane 6) is 108.45 (3)°. The bond lengths do not indicate any electron delocalization in this part of molecule.

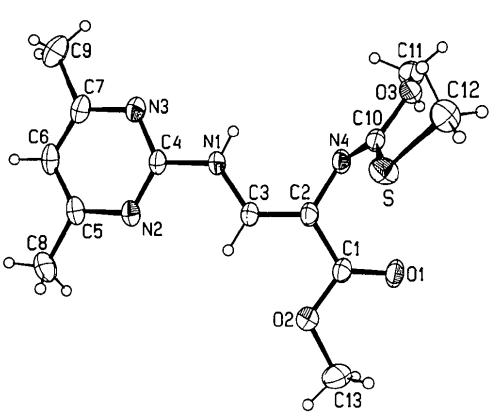


Fig. 3 *ORTEP* (Johnson, 1965) view of (III) showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 50% probability level and the H atoms as small circles of arbitrary radii.

The configuration about the N(4)=C(10) double bond is *Z*. The pyrimidine ring is planar to within 0.016 (2) Å [C(6)]. The angles at N(2) and N(3) [both 115.3 (1)°] are smaller than 120°, as is usual for heterocyclic rings. The remaining bond lengths and angles are within the normal ranges for this type of molecule. There are no short intermolecular contacts in (I), (II) or (III).

Experimental

Compounds (I), (II) and (III) were prepared as described by Breaux & Zwikelmaier (1981), Svete *et al.* (1994) and Smodiš *et al.* (1994). Densities D_m were measured by flotation in chlorobenzene–tetrachloromethane mixture.

Compound (I)

Crystal data

$C_{14}H_{17}NO_3$
$M_r = 247.3$
Monoclinic
$P2_1/c$
$a = 12.1732\ (9)$ Å
$b = 6.8173\ (6)$ Å
$c = 16.737\ (1)$ Å
$\beta = 93.457\ (6)°$
$V = 1386.4\ (2)$ Å^3
$Z = 4$
$D_x = 1.185$ Mg m^{-3}
$D_m = 1.18\ (2)$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 100 reflections
$\theta = 10.095–17.903°$
$\mu = 0.0778$ mm^{-1}
$T = 293$ K
Irregular
0.92 × 0.76 × 0.72 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
13360 measured reflections
3309 independent reflections
2401 observed reflections [$I > 4.0\sigma(I)$]
$R_{\rm int} = 0.037$
$\theta_{\rm max} = 27.94°$

$h = -16 \rightarrow 16$
$k = -8 \rightarrow 8$
$l = -22 \rightarrow 22$
3 standard reflections monitored every 600 reflections (orientation) and every 333 min (intensity)
intensity variation: −1.09%

Refinement

Refinement on F
$R = 0.047$
$wR = 0.049$
$S = 0.957$
2749 reflections
215 parameters
$w = 6w_fw_s$ (see below)
$(\Delta/\sigma)_{\rm max} = 0.109$
$\Delta\rho_{\rm max} = 0.217$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.263$ e Å^{-3}

Extinction correction: Zachariasen (Larson, 1970)
Extinction coefficient: 23751 (4311)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Compound (II)

Crystal data

$C_{18}H_{16}N_2O_5$
$M_r = 340.3$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Triclinic
$P\overline{1}$
$a = 8.290\ (1)$ Å
$b = 8.067\ (1)$ Å
$c = 12.500\ (2)$ Å
$\alpha = 90.38\ (1)°$
$\beta = 94.50\ (2)°$
$\gamma = 90.35\ (2)°$
$V = 833.3\ (2)$ Å^3
$Z = 2$
$D_x = 1.356$ Mg m^{-3}
$D_m = 1.35\ (2)$ Mg m^{-3}
Cell parameters from 75 reflections
$\theta = 8.101–14.154°$
$\mu = 0.0939$ mm^{-1}
$T = 293$ K
Prism
$1.0 \times 0.72 \times 0.36$ mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
10074 measured reflections
4814 independent reflections
3862 observed reflections $[I > 2.5\sigma(I)]$
$R_{\rm int} = 0.012$
$\theta_{\rm max} = 29.91°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 17$
3 standard reflections monitored every 600 reflections (orientation) and every 333 min (intensity) intensity variation: none

Refinement

Refinement on F
$R = 0.059$
$wR = 0.062$
$S = 0.997$
4297 reflections
290 parameters
$w = 6w_fw_s$ (see below)
$(\Delta/\sigma)_{\rm max} = 0.030$
$\Delta\rho_{\rm max} = 0.522$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.353$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Compound (III)

Crystal data

$C_{13}H_{18}N_4O_3S$
$M_r = 310.4$
Triclinic
$P\overline{1}$
$a = 8.3383\ (7)$ Å
$b = 9.4365\ (7)$ Å
$c = 10.5925\ (8)$ Å
$\alpha = 71.904\ (7)°$
$\beta = 88.558\ (7)°$
$\gamma = 81.614\ (8)°$
$V = 783.6\ (2)$ Å^3
$Z = 2$
$D_x = 1.315$ Mg m^{-3}
$D_m = 1.31\ (2)$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 75 reflections
$\theta = 9.965–15.790°$
$\mu = 0.2111$ mm^{-1}
$T = 293$ K
Irregular
$0.6 \times 0.52 \times 0.32$ mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
9131 measured reflections
4540 independent reflections
3269 observed reflections $[I > 2.5\sigma(I)]$
$R_{\rm int} = 0.011$
$\theta_{\rm max} = 29.92°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 500 reflections (orientation) and every 333 min (intensity) intensity variation: −0.23%

Refinement

Refinement on F
$R = 0.042$
$wR = 0.044$
$S = 1.019$
3852 reflections
262 parameters
$w = 6w_fw_s$ (see below)
$(\Delta/\sigma)_{\rm max} = 0.069$
$\Delta\rho_{\rm max} = 0.445$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.420$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
(I)				
C(1)	0.89113 (10)	0.76556 (19)	0.58348 (7)	0.0465 (3)
C(2)	0.79152 (9)	0.64801 (18)	0.57153 (7)	0.0432 (3)
C(3)	0.80064 (10)	0.48590 (20)	0.52253 (7)	0.0464 (3)
C(4)	0.63476 (14)	0.30564 (25)	0.55497 (12)	0.0648 (5)
C(5)	0.75014 (17)	0.1932 (3)	0.44789 (13)	0.0694 (6)
C(6)	0.68566 (10)	0.72182 (19)	0.59883 (7)	0.0447 (3)
C(8)	0.96434 (14)	1.06745 (24)	0.63394 (11)	0.0605 (5)
C(9)	0.93317 (21)	1.2139 (3)	0.69433 (16)	0.0793 (7)
C(71)	0.67533 (9)	0.77690 (17)	0.68456 (7)	0.0412 (3)
C(72)	0.74794 (11)	0.70507 (21)	0.74475 (8)	0.0505 (4)
C(73)	0.73108 (16)	0.7437 (3)	0.82486 (9)	0.0660 (5)
C(74)	0.64187 (18)	0.8539 (3)	0.84487 (11)	0.0723 (6)
C(75)	0.57029 (16)	0.92824 (25)	0.78538 (13)	0.0721 (6)
C(76)	0.58637 (12)	0.89028 (21)	0.70527 (10)	0.0549 (4)
O(1)	0.98205 (10)	0.71953 (21)	0.56435 (10)	0.0757 (4)
O(2)	0.87123 (8)	0.93967 (15)	0.61768 (7)	0.0542 (3)
O(3)	0.60430 (10)	0.73249 (25)	0.55275 (8)	0.0750 (4)
N	0.73117 (10)	0.33832 (18)	0.50949 (7)	0.0521 (3)
(II)				
C(1)	0.80186 (14)	0.04640 (15)	0.56889 (10)	0.0347 (3)
C(2)	0.71554 (14)	0.19930 (15)	0.53906 (10)	0.0337 (3)
C(3)	0.61310 (14)	0.20405 (16)	0.44724 (10)	0.0352 (3)
C(4)	0.97932 (18)	−0.08592 (19)	0.69932 (13)	0.0455 (4)
C(5)	1.07589 (23)	−0.0327 (3)	0.80030 (17)	0.0617 (6)
C(6)	0.72893 (15)	0.35117 (15)	0.60644 (10)	0.0359 (3)
C(71)	0.88982 (15)	0.39774 (15)	0.66158 (11)	0.0367 (3)
C(72)	0.89434 (20)	0.46485 (18)	0.76485 (12)	0.0475 (4)
C(73)	1.04240 (25)	0.50698 (23)	0.81858 (17)	0.0646 (6)
C(74)	1.18345 (24)	0.48597 (25)	0.76925 (21)	0.0701 (7)
C(75)	1.17994 (20)	0.42268 (22)	0.66614 (20)	0.0618 (6)
C(76)	1.03267 (17)	0.37590 (18)	0.61187 (14)	0.0456 (4)
C(81)	0.50277 (13)	0.07547 (16)	0.27762 (10)	0.0342 (3)
C(82)	0.48119 (15)	−0.07800 (16)	0.22617 (10)	0.0364 (3)
C(83)	0.39573 (15)	−0.08370 (17)	0.12704 (10)	0.0388 (3)
C(84)	0.33259 (19)	0.05534 (21)	0.07541 (12)	0.0470 (4)
C(85)	0.35891 (21)	0.20713 (20)	0.12686 (13)	0.0499 (4)
C(86)	0.44197 (18)	0.21841 (18)	0.22756 (12)	0.0431 (4)
O(1)	0.79799 (15)	−0.07905 (13)	0.51364 (10)	0.0514 (3)
O(2)	0.87976 (12)	0.05433 (12)	0.66592 (8)	0.03975 (25)
O(3)	0.61099 (13)	0.43932 (15)	0.61574 (10)	0.0538 (3)
O(4)	0.44966 (20)	−0.36336 (17)	0.10981 (13)	0.0699 (5)
O(5)	0.2706 (3)	−0.25892 (22)	−0.00041 (15)	0.0966 (7)
N(1)	0.59058 (13)	0.07640 (14)	0.37819 (9)	0.0384 (3)
N(2)	0.37136 (18)	−0.24720 (18)	0.07502 (11)	0.0521 (4)

(III)

S	0.58045 (5)	0.29674 (5)	0.48459 (4)	0.05460 (16)
O(1)	0.51682 (15)	0.50181 (13)	0.15432 (13)	0.0569 (4)
O(2)	0.77469 (15)	0.46254 (13)	0.09288 (14)	0.0583 (4)
O(3)	0.34026 (12)	0.16263 (12)	0.45193 (11)	0.0452 (4)
N(1)	0.82096 (16)	0.01051 (14)	0.27280 (14)	0.0470 (4)
N(2)	1.06423 (14)	−0.02181 (15)	0.16832 (13)	0.0451 (4)
N(3)	0.93867 (16)	−0.23225 (15)	0.29666 (15)	0.0511 (5)
N(4)	0.52197 (14)	0.19147 (14)	0.28424 (13)	0.0427 (4)
C(1)	0.64018 (18)	0.41690 (16)	0.15512 (15)	0.0436 (5)
C(2)	0.66017 (17)	0.25336 (16)	0.22362 (15)	0.0415 (4)
C(3)	0.79865 (17)	0.16413 (16)	0.21435 (15)	0.0425 (5)
C(4)	0.94890 (16)	−0.08561 (16)	0.24432 (15)	0.0421 (4)
C(5)	1.18296 (17)	−0.11785 (20)	0.13798 (16)	0.0488 (5)
C(6)	1.18181 (20)	−0.27089 (21)	0.18263 (19)	0.0561 (6)
C(7)	1.05835 (21)	−0.32594 (19)	0.26416 (19)	0.0543 (6)
C(8)	1.31625 (24)	−0.0485 (3)	0.05565 (24)	0.0663 (8)
C(9)	1.0491 (4)	−0.49181 (24)	0.3198 (4)	0.0820 (11)
C(10)	0.47560 (16)	0.20988 (14)	0.39349 (14)	0.0385 (4)
C(11)	0.23686 (22)	0.10957 (22)	0.37445 (21)	0.0539 (6)
C(12)	0.45797 (25)	0.28580 (25)	0.62774 (19)	0.0601 (7)
C(13)	0.7637 (3)	0.62123 (23)	0.02401 (25)	0.0707 (8)

Table 2. *Bond distances* (Å) *and angles* (°)

(I)

C(1)—C(2)	1.457 (2)	C(6)—O(3)	1.220 (2)
C(1)—O(1)	1.212 (2)	C(8)—C(9)	1.486 (3)
C(1)—O(2)	1.346 (2)	C(8)—O(2)	1.442 (2)
C(2)—C(3)	1.385 (2)	C(71)—C(72)	1.389 (2)
C(2)—C(6)	1.481 (2)	C(71)—C(76)	1.391 (2)
C(3)—N	1.324 (2)	C(72)—C(73)	1.394 (2)
C(4)—N	1.454 (2)	C(73)—C(74)	1.379 (3)
C(5)—N	1.457 (2)	C(74)—C(75)	1.379 (3)
C(6)—C(71)	1.496 (2)	C(75)—C(76)	1.391 (3)

C(2)—C(1)—O(1)	125.9 (1)	C(6)—C(71)—C(76)	119.3 (1)
C(2)—C(1)—O(2)	111.9 (1)	C(72)—C(71)—C(76)	119.1 (1)
O(1)—C(1)—O(2)	122.1 (1)	C(71)—C(72)—C(73)	120.5 (1)
C(1)—C(2)—C(3)	115.1 (1)	C(72)—C(73)—C(74)	120.0 (1)
C(1)—C(2)—C(6)	120.2 (1)	C(73)—C(74)—C(75)	119.8 (2)
C(3)—C(2)—C(6)	123.8 (1)	C(74)—C(75)—C(76)	120.5 (2)
C(2)—C(3)—N	129.1 (2)	C(71)—C(76)—C(75)	120.0 (1)
C(2)—C(6)—C(71)	120.4 (1)	C(1)—O(2)—C(8)	117.0 (1)
C(2)—C(6)—O(3)	120.9 (1)	C(3)—N—C(4)	123.9 (1)
C(71)—C(6)—O(3)	118.6 (1)	C(3)—N—C(5)	120.4 (1)
C(9)—C(8)—O(2)	107.7 (1)	C(4)—N—C(5)	115.7 (1)
C(6)—C(71)—C(72)	121.4 (1)		

(II)

C(1)—C(2)	1.465 (2)	C(73)—C(74)	1.375 (3)
C(1)—O(1)	1.220 (2)	C(74)—C(75)	1.381 (3)
C(1)—O(2)	1.330 (2)	C(75)—C(76)	1.398 (2)
C(2)—C(3)	1.374 (2)	C(81)—C(82)	1.395 (2)
C(2)—C(6)	1.481 (2)	C(81)—C(86)	1.394 (2)
C(3)—N(1)	1.342 (2)	C(81)—N(1)	1.402 (2)
C(4)—C(5)	1.499 (3)	C(82)—C(83)	1.378 (2)
C(4)—O(2)	1.449 (2)	C(83)—C(84)	1.383 (2)
C(6)—C(71)	1.497 (2)	C(83)—N(2)	1.473 (2)
C(6)—O(3)	1.225 (2)	C(84)—C(85)	1.387 (2)
C(71)—C(72)	1.394 (2)	C(85)—C(86)	1.389 (2)
C(71)—C(76)	1.391 (2)	O(4)—N(2)	1.207 (2)
C(72)—C(73)	1.391 (3)	O(5)—N(2)	1.212 (2)

C(2)—C(1)—O(1)	124.3 (1)	C(74)—C(75)—C(76)	120.1 (2)
C(2)—C(1)—O(2)	113.1 (1)	C(71)—C(76)—C(75)	119.4 (2)
O(1)—C(1)—O(2)	122.5 (1)	C(82)—C(81)—C(86)	120.0 (1)
C(1)—C(2)—C(3)	120.1 (1)	C(82)—C(81)—N(1)	116.7 (1)
C(1)—C(2)—C(6)	122.6 (1)	C(86)—C(81)—N(1)	123.3 (1)
C(3)—C(2)—C(6)	117.3 (1)	C(81)—C(82)—C(83)	118.3 (1)
C(2)—C(3)—N(1)	123.7 (1)	C(82)—C(83)—C(84)	123.3 (1)
C(5)—C(4)—O(2)	106.2 (1)	C(82)—C(83)—N(2)	117.5 (1)
C(2)—C(6)—C(71)	119.1 (1)	C(84)—C(83)—N(2)	119.2 (1)
C(2)—C(6)—O(3)	120.7 (1)	C(83)—C(84)—C(85)	117.5 (1)
C(71)—C(6)—O(3)	120.2 (1)	C(84)—C(85)—C(86)	121.2 (1)
C(6)—C(71)—C(72)	118.5 (1)	C(81)—C(86)—C(85)	119.7 (1)
C(6)—C(71)—C(76)	121.5 (1)	C(1)—O(2)—C(4)	117.2 (1)
C(72)—C(71)—C(76)	120.1 (1)	C(3)—N(1)—C(81)	127.6 (1)
C(71)—C(72)—C(73)	119.7 (2)	C(83)—N(2)—O(4)	119.1 (1)
C(72)—C(73)—C(74)	120.2 (2)	C(83)—N(2)—O(5)	117.8 (1)
C(73)—C(74)—C(75)	120.5 (2)	O(4)—N(2)—O(5)	123.0 (2)

(III)

S—C(10)	1.764 (2)	N(3)—C(4)	1.336 (2)
S—C(12)	1.791 (2)	N(3)—C(7)	1.346 (2)
O(1)—C(1)	1.207 (2)	N(4)—C(2)	1.417 (2)
O(2)—C(1)	1.344 (2)	N(4)—C(10)	1.266 (2)
O(2)—C(13)	1.438 (2)	C(1)—C(2)	1.474 (2)
O(3)—C(10)	1.340 (2)	C(2)—C(3)	1.347 (2)
O(3)—C(11)	1.444 (3)	C(5)—C(6)	1.374 (3)
N(1)—C(3)	1.374 (2)	C(5)—C(8)	1.494 (3)
N(1)—C(4)	1.387 (2)	C(6)—C(7)	1.381 (2)
N(2)—C(4)	1.328 (2)	C(7)—C(9)	1.504 (3)
N(2)—C(5)	1.345 (2)		

C(10)—S—C(12)	103.1 (1)	N(1)—C(4)—N(2)	116.8 (1)
C(1)—O(2)—C(13)	115.7 (2)	N(1)—C(4)—N(3)	115.0 (1)
C(10)—O(3)—C(11)	116.2 (1)	N(2)—C(4)—N(3)	128.2 (1)
C(3)—N(1)—C(4)	123.5 (1)	N(2)—C(5)—C(6)	121.4 (1)
C(4)—N(2)—C(5)	115.3 (1)	N(2)—C(5)—C(8)	116.1 (2)
C(4)—N(3)—C(7)	115.3 (1)	C(6)—C(5)—C(8)	122.5 (2)
C(2)—N(4)—C(10)	119.2 (1)	C(5)—C(6)—C(7)	118.6 (2)
O(1)—C(1)—O(2)	123.1 (1)	N(3)—C(7)—C(6)	121.1 (2)
O(1)—C(1)—C(2)	124.1 (1)	N(3)—C(7)—C(9)	116.6 (2)
O(2)—C(1)—C(2)	112.8 (1)	C(6)—C(7)—C(9)	122.3 (2)
N(4)—C(2)—C(1)	117.5 (1)	S—C(10)—O(3)	113.3 (1)
N(4)—C(2)—C(3)	121.2 (1)	S—C(10)—N(4)	124.4 (1)
C(1)—C(2)—C(3)	120.9 (1)	O(3)—C(10)—N(4)	122.3 (1)
N(1)—C(3)—C(2)	123.0 (1)		

Table 3. *Several planes of interest* (*Ax* + *By* + *Cz* = *D where x, y and z are fractional coordinates*) *with the deviations* (Å) *of the atoms forming these planes*

(I)

Plane 1

A =	6.855 (7)	C(71)	0.038 (1)	C(75)	−0.019 (2)
B =	5.610 (3)	C(72)	0.022 (2)	C(76)	0.026 (2)
C =	−1.845 (8)	C(73)	−0.025 (2)	C(6)	−0.042 (2)
D =	7.686 (7)	C(74)	−0.055 (3)		

Plane 2

A =	3.420 (8)	C(1)	0.069 (1)	C(8)	−0.040 (2)
B =	−3.423 (2)	C(2)	−0.030 (1)	C(9)	0.160 (3)
C =	13.379 (6)	C(3)	−0.099 (1)	N	−0.006 (1)
D =	8.164 (3)	C(4)	0.385 (2)	O(1)	0.281 (2)
		C(5)	−0.268 (2)	O(2)	−0.138 (1)

(II)

Plane 3

A =	−0.525 (5)	C(71)	−0.000 (1)	C(75)	0.012 (3)
B =	7.453 (2)	C(72)	0.009 (2)	C(76)	−0.003 (2)
C =	−4.736 (9)	C(73)	−0.010 (3)	C(6)	−0.002 (2)
D =	−0.636 (6)	C(74)	−0.007 (3)		

Plane 4

A =	7.0052 (7)	C(1)	0.029 (1)	C(86)	0.167 (2)
B =	1.753 (2)	C(2)	−0.101 (1)	O(1)	0.164 (1)
C =	−6.908 (1)	C(3)	−0.176 (1)	N(2)	−0.090 (2)
D =	1.7396 (7)	N(1)	−0.081 (1)	O(4)	0.015 (2)
		C(81)	−0.003 (1)	O(5)	−0.295 (2)
		C(82)	−0.068 (1)	O(2)	−0.082 (1)
		C(83)	0.008 (1)	C(4)	0.139 (2)
		C(84)	0.166 (2)	C(5)	0.212 (3)
		C(85)	0.261 (2)		

(III)

Plane 5

A =	−3.204 (4)	S	0.019 (1)	C(11)	0.122 (3)
B =	6.779 (3)	C(10)	−0.010 (2)	N(4)	−0.015 (2)
C =	−2.458 (6)	O(3)	−0.040 (1)	C(12)	−0.014 (3)
D =	−1.059 (3)				

Plane 6

$A =$	4.024 (3)		C(1)	−0.055 (2)		C(6)	−0.244 (2)
$B =$	3.818 (2)		C(2)	0.043 (2)		C(7)	−0.186 (2)
$C =$	9.373 (2)		C(3)	0.173 (2)		N(2)	0.100 (2)
$D =$	5.676 (2)		N(1)	0.224 (2)		N(3)	−0.006 (2)
			C(4)	0.105 (2)		C(8)	−0.044 (3)
			C(5)	−0.073 (2)		C(9)	−0.335 (4)
			O(1)	−0.234 (2)		O(2)	0.077 (2)

Data were collected with an ω–2θ scan width of $(0.8 + 0.3\tan\theta)^\circ$ for (I) and $(0.9 + 0.3\tan\theta)^\circ$ for (II) and (III). Background counts were measured for one quarter of a scan on both sides. As the crystals were of reasonable sizes and had low absorption coefficients, absorption was ignored. The structures were solved by direct methods using *MULTAN*88 (Debaerdemaeker, Germain, Main, Refaat, Tate & Woolfson, 1988). Full-matrix anisotropic refinement was used for the non-H atoms while the H atoms were refined with isotropic displacement parameters for (II) and (III); only the positional parameters were refined for (I) . Calculated weights were used, $w = 6w_fw_s$. For (I), $w_f = |F_o|/2.2$ for $F_o < 2.2$, $w_f = 4.2/F_o$ for $|F_o| > 4.2$, $w_f = 1.0$ for $2.2 \leq F_o \leq 4.2$ and $w_s = (\sin\theta/0.54)^3$ for $\sin\theta < 0.54$, $w_s = 1.0$ for $\sin\theta \geq 0.54$. For (II), $w_f = |F_o|/1.34$ for $|F_o| < 1.34$, $w_f = (4.22/|F_o|)^{1.55}$ for $|F_o| > 4.22$, $w_f = 1.0$ for $1.34 \leq F_o \leq 4.22$ and $w_s = \sin\theta/0.53$ for $\sin\theta < 0.53$, $w_s = 0.64/\sin\theta$ for $\sin\theta > 0.64$, $w_s = 1.0$ for $0.53 \leq \sin\theta \leq 0.64$. For (III), $w_f = (|F_o|/2.59)^{1.2}$ for $|F_o| < 2.59$, $w_f = (7.77/|F_o|)^{1.4}$ for $|F_o| > 7.77$, $w_f = 1.0$ for $2.59 \leq |F_o| \leq 7.77$ and $w_s = (\sin\theta/0.4)^{1.9}$ for $\sin\theta < 0.4$, $w_s = 1.0$ for $\sin\theta \geq 0.4$. The *Xtal* system (Hall & Stewart, 1990) was used for the correlation and reduction of data, structure refinement and interpretation. Calculations were performed on VAX 8550 computers at the University Computer Centre, Ljubljana. *ORTEP* (Johnson, 1965) was used to produce molecular graphics.

The financial support of the Slovene Ministry for Science and Technology is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1064). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Breaux, E. J. & Zwikelmaier, K. E. (1981). *J. Heterocycl. Chem.* **18**, 183–184.

Camerman, A. (1970). *Can. J. Chem.* **48**, 179–181.

Camerman, A., Jensen, L. H. & Balaban, A. T. (1969). *Acta Cryst.* B**25**, 2623–2630.

Debaerdemaeker, T., Germain, G., Main, P., Refaat, L. S., Tate, C. & Woolfson, M. M. (1988). *MULTAN*88. *A System of Computer Programs for the Automatic Solutions of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal*3.0 *Reference Manual.* Univs. of Western Australia, Australia, and Maryland, USA.

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

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

Smodiš, J., Stanovnik, B. & Tišler, M. (1994). In preparation.

Svete, J., Kralj, L., Stanovnik, B. & Tišler, M. (1994). *Org. Prep. Proc. Int.* In the press.