C(9)	0.6489 (8)	0.0899 (11)	0.2636 (4)	0.100 (3)
C(10)	0.7631 (9)	0.1179 (17)	0.2064 (5)	0.139 (4)
C(11)	0.3566 (7)	0.5678 (8)	0.2544 (4)	0.069 (2)
C(12)	0.2273 (8)	0.6723 (9)	0.2258 (5)	0.093 (2)
C(13)	0.2408 (11)	0.8251 (11)	0.2621 (6)	0.112 (3)
C(14)	0.1911 (14)	0.8353 (13)	0.3456 (7)	0.140 (4)
C(15)	0.3286 (7)	0.3914 (10)	0.1367 (4)	0.079 (2)
C(16)	0.4686 (7)	0.4367 (12)	0.0938 (4)	0.090 (2)
C(17)	0.4419 (10)	0.4174 (15)	0.0052 (4)	0.117 (3)
C(18)	0.5751 (12)	0.4107 (18)	−0.0449 (5)	0.148 (4)
Cl(1)	0.2045 (2)	0.1146 (3)	0.73726 (9)	0.0874 (6)

Table 2. *Selected geometric parameters (Å, °)*

S(1)—C(1)	1.694 (5)	S(2)—C2	1.688 (5)
C(1)—N(2)	1.308 (9)	C2—N(3)	1.312 (9)
C(1)—N(1)	1.340 (9)	C2—N(4)	1.347 (10)
N(2)—C(1)—N(1)	117.1 (4)	N(3)—C2—N(4)	116.4 (4)
N(2)—C(1)—S(1)	122.3 (5)	N(3)—C2—S(2)	123.0 (6)
N(1)—C(1)—S(1)	120.6 (5)	N(4)—C2—S(2)	120.6 (6)
$N(1^{i})\cdots S(1)$	3.500 (9)	$N(1^{i})\cdots Cl(1)$	3.321 (9)
$N(2^{ii})\cdots S(1)$	3.531 (9)	$N(2^{i})\cdots Cl(1)$	3.261 (9)
$N(3^{iii})\cdots S(2)$	3.483 (9)	$N(3^{iv})\cdots Cl(1)$	3.205 (9)
$N(4^{iv})\cdots S(2)$	3.514 (9)	$N(4^{iv})\cdots Cl(1)$	3.302 (9)
$N(1^{i})\cdots S(1)\cdots N(2^{ii})$	141.2 (5)	$Cl(1^{v})\cdots N(2^{ii})\cdots S(1)$	123.1 (5)
$N(2^{ii})\cdots Cl(1^{v})\cdots N(3^{iii})$	145.5 (5)	$Cl(1^{v})\cdots N(3^{iii})\cdots S(2)$	126.1 (5)
$N(3^{iii})\cdots S(2)\cdots N(4^{iv})$	141.1 (5)	$N(3^{iv})\cdots Cl(1)\cdots N(4^{iv})$	40.6 (5)
$N(4^{iv})\cdots Cl(1)\cdots N(1^{i})$	132.8 (5)	$N(4^{iv})\cdots Cl(1)\cdots N(1^{i})$	132.8 (5)
$Cl(1)\cdots N(1^{i})\cdots S(1)$	132.1 (5)	$N(1^{i})\cdots Cl(1)\cdots N(2^{i})$	40.1 (5)
$Cl(1)\cdots N(4^{iv})\cdots S(2)$	131.9 (5)		

Symmetry codes: (i) $1-x, y-\frac{1}{2}, 1-z$; (ii) $1-x, \frac{1}{2}+y, 1-z$; (iii) $-x, \frac{1}{2}+y, 2-z$; (iv) $-x, y-\frac{1}{2}, 2-z$; (v) $x, 1+y, z$.

Data collection: Siemens diffractometer software. Cell refinement: Siemens diffractometer software. Data reduction: *SHELXTL/PC* (Sheldrick, 1990*a*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*b*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

This work is supported by a Hong Kong Research Grants Council Earmarked Grant (No. CUHK 456/95P).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(2,5-Dichlorophenylhydrazono)glutaric Acid and 2-(3,5-Dichlorophenylhydrazono)-glutaric Acid

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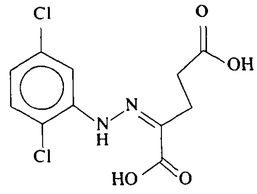
(*Received 18 March 1996; accepted 17 June 1996*)

Abstract

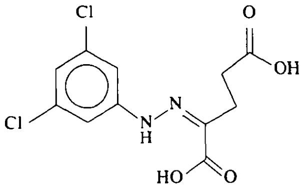
The title compounds, $C_{11}H_{10}Cl_2N_2O_4$, are constitutional isomers which exhibit similar molecular geometry. The configuration about the N=C bond is *E* in both molecules. The same packing pattern was found in both structures. Hydrogen-bonded dimers, formed around the inversion centres *via* O—H···O interactions of carboxyl groups, are connected into infinite chains. The intramolecular N—H···Cl hydrogen bond observed only in the 2,5-dichlorophenyl derivative contributes to the planarity of the molecule.

Comment

During the preparation of dihalogenated indole-3-acetic acids (Baldi, Slovin & Cohen, 1984), the title intermediates 2-(2,5-dichlorophenylhydrazono)glutaric acid, (1), and 2-(3,5-dichlorophenylhydrazono)glutaric acid, (2), were obtained. It is important that the two compounds should be identified unambiguously prior to bioactivity testing to avoid misinterpretation of bioassays. Hence, the crystal structures of the title compounds were solved and the results are reported here.

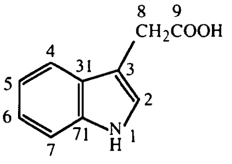


(1)



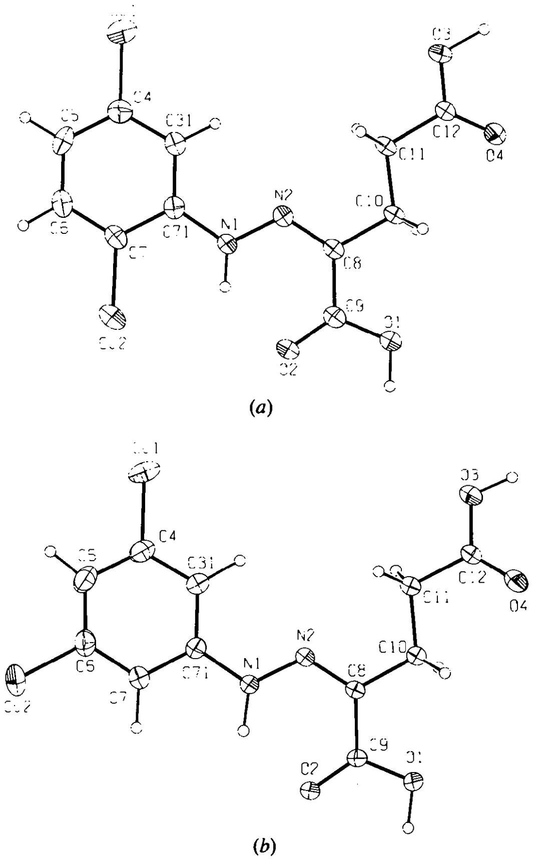
(2)

As part of a systematic study of the structure–activity correlations of the phytohormone indole-3-acetic acid, (3) (auxin), the series of monohalogenated and dihalogenated derivatives has been examined (Antolić, Kojić-Prodić, Tomić, Nigović, Magnus & Cohen, 1996; Nigović, Kojić-Prodić, Antolić, Tomić, Puntarec & Cohen, 1996). Our study includes structural characterization by X-ray analysis, *ab initio* calculations for evaluation of energetically stable conformations (Ramek, Tomić & Kojić-Prodić, 1996) and bioassays. 4-Chloroindole-3-acetic acid is a naturally occurring auxin but some halogenated derivatives also exhibit auxin activity (Rescher, Walther, Schiebl & Klaembt, 1996; Böttger, Engvild & Soll, 1978; Katekar & Geissler, 1982, 1983).



(3)

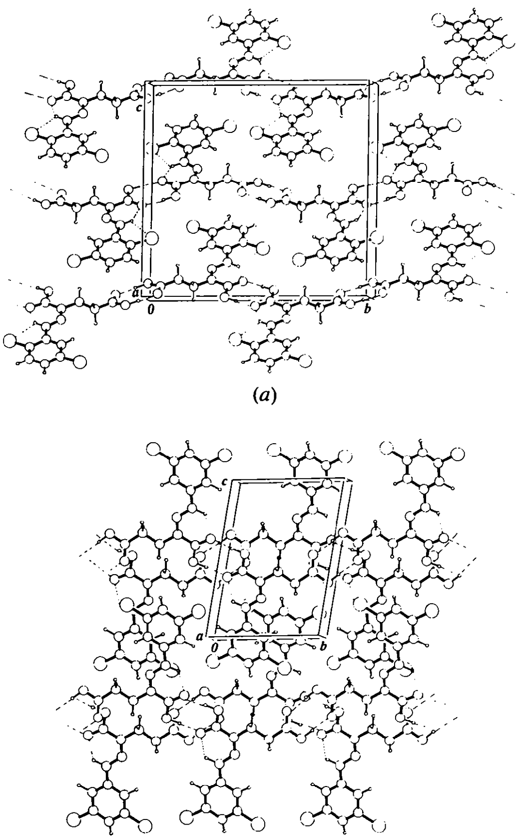
The molecular structures of compounds (1) and (2) are shown in Fig. 1 and their packing diagrams are shown in Fig. 2. In the structure (1), 17 of the 19 non-H atoms in the molecule are almost coplanar, the average deviation from the best least-squares plane being 0.041 (5) Å. However, O3 and O4 of the carboxyl group deviate from this plane by 0.247 (4) and 0.314 (4) Å, respectively. Both carboxyl groups are in *syn*-periplanar conformations; the torsion angle C10—C8—C9—O1 is 0.5 (5)° and C10—C11—C12—O4 is 12.0 (6)°. The coplanarity of the C9,O1,O2 carboxyl group with the aromatic system may result from the formation of bifurcated intramolecular N1—H1···O2 hydrogen bonds (Table 5). An N1—H1···Cl2 intramolecular hydrogen bond was found in compound (1) (Table 5; Fig. 2*a*). Each carboxyl group is incorporated into a centrosymmetric ring-shaped hydrogen-bonded dimer which is part of an infinite chain along **b**. In compound (2), the ten-atom fragment is almost planar [the average deviation is 0.018 (3) Å]. The carboxyl groups are in *syn*-periplanar conformations with torsion angles C10—C8—C9—O1 = 0.8 (5)° and C10—C11—C12—O4 = 19.3 (6)°. The hydrogen-bonding motif is similar to that observed in compound (1); the only difference is the lack of any N—H···Cl-type intramolecular interaction owing to the *meta* substitution of the benzene ring. As a result, bending about the C71—N1 bonds occurs; C7—C71—N1—N2 = 168.9 (3)° in (2) whereas this torsion angle is 177.8 (3)° in (1) and the molecule is more planar.



(*a*)

(*b*)

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the molecular structures of (*a*) compound (1) and (*b*) compound (2) with the labelling of the non-H atoms. The atom-numbering scheme is that of indole-3-acetic acid for ease of comparison. Displacement ellipsoids are shown at the 30% probability level; H atoms are drawn as small circles of arbitrary radii.



(*a*)

(*b*)

Fig. 2. Molecular packing for (*a*) compound (1) and (*b*) compound (2): intramolecular N1—H···O2 (both compounds) and N1—H···Cl2 [compound (1)] interactions are shown as dotted lines. Intermolecular O1—H11···O2 and O3—H3···O4 hydrogen bonds are shown as dashed lines.

Experimental

During synthesis of dihalogenated indole-3-acetic acids (Baldi, Slovin & Cohen, 1984), the title compounds (1) and (2) were obtained as intermediate products. Single crystals suitable for data collection were grown from a mixture of methanol, 2-propanol and water (1:1:1 by volume) at room temperature over a few days.

Compound (1)

Crystal data

$C_{11}H_{10}Cl_2N_2O_4$
$M_r = 305.12$
Monoclinic
$P2_1/n$
$a = 4.783\ (1)$ Å
$b = 16.982\ (6)$ Å
$c = 16.205\ (6)$ Å
$\beta = 96.82\ (4)°$
$V = 1306.9\ (7)$ Å^3
$Z = 4$
$D_x = 1.551$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 11.37–42.39°$
$\mu = 4.68$ mm^{-1}
$T = 295\ (3)$ K
Plate
0.25 × 0.11 × 0.06 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2963 measured reflections
2640 independent reflections
1211 observed reflections [$I > 3\sigma(I)$]
$R_{\text{int}} = 0.0287$

$\theta_{\text{max}} = 74.23°$
$h = -5 \rightarrow 0$
$k = -21 \rightarrow 10$
$l = -20 \rightarrow 20$
3 standard reflections monitored every 77 reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R(F) = 0.0549$
$wR(F^2) = 0.1241$
$S = 1.125$
2640 reflections
212 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0795P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = -0.009$
$\Delta\rho_{\text{max}} = 0.34$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.32$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
C31	−0.2366 (9)	0.7979 (2)	0.2543 (3)	0.0512 (10)
C4	−0.0535 (9)	0.7946 (2)	0.1954 (2)	0.0532 (10)
Cl1	0.0670 (3)	0.70295 (6)	0.16638 (8)	0.0797 (4)
C5	0.044 (1)	0.8606 (3)	0.1598 (3)	0.0656 (13)
C6	−0.047 (1)	0.9322 (3)	0.1840 (3)	0.0718 (14)
C7	−0.2227 (9)	0.9387 (2)	0.2444 (3)	0.0582 (11)
Cl2	−0.3216 (3)	1.03049 (6)	0.27599 (8)	0.0887 (5)
C71	−0.3215 (8)	0.8708 (2)	0.2810 (2)	0.0498 (10)
N1	−0.4970 (8)	0.8804 (2)	0.3425 (2)	0.0546 (10)
N2	−0.6019 (6)	0.8164 (2)	0.3765 (2)	0.0445 (8)
C8	−0.7762 (8)	0.8232 (2)	0.4315 (2)	0.0435 (9)
C9	−0.8683 (9)	0.8991 (2)	0.4623 (2)	0.0486 (10)
O1	−1.0478 (7)	0.8927 (2)	0.5170 (2)	0.0647 (9)
O2	−0.7875 (6)	0.9632 (1)	0.4391 (2)	0.0639 (8)
C10	0.895 (1)	0.7488 (2)	0.4641 (3)	0.0478 (11)
C11	−0.771 (1)	0.6757 (2)	0.4294 (4)	0.0561 (13)
C12	−0.8832 (9)	0.6010 (2)	0.4616 (2)	0.0490 (10)
O3	−0.7433 (8)	0.53929 (2)	0.4456 (2)	0.0915 (12)
O4	−1.0835 (7)	0.5972 (2)	0.4997 (2)	0.0795 (11)

Table 2. *Selected geometric parameters (Å, °) for (1)*

Cl1—C4	1.744 (4)	C4—C31	1.371 (5)
Cl2—C7	1.724 (4)	C5—C6	1.364 (7)
O1—C9	1.310 (5)	C6—C7	1.368 (6)
O2—C9	1.229 (4)	C7—C71	1.406 (5)
O3—C12	1.287 (5)	C8—C9	1.469 (5)
O4—C12	1.201 (5)	C8—C10	1.506 (5)
N1—N2	1.343 (4)	C10—C11	1.511 (6)
N1—C71	1.387 (5)	C11—C12	1.496 (5)
N2—C8	1.296 (4)	C31—C71	1.388 (5)
C4—C5	1.367 (6)		
N2—N1—C71	119.0 (3)	O2—C9—C8	123.7 (3)
N1—N2—C8	120.8 (3)	C8—C10—C11	112.3 (4)
C5—C4—C31	122.5 (4)	C10—C11—C12	113.2 (4)
C4—C5—C6	118.3 (4)	O3—C12—O4	121.7 (3)
C5—C6—C7	121.4 (4)	O3—C12—C11	113.9 (4)
C6—C7—C71	120.1 (4)	O4—C12—C11	124.4 (4)
N2—C8—C9	123.7 (3)	C4—C31—C71	119.4 (3)
N2—C8—C10	117.8 (3)	N1—C71—C7	117.9 (3)
C9—C8—C10	118.5 (3)	N1—C71—C31	123.6 (3)
O1—C9—O2	122.4 (3)	C7—C71—C31	118.3 (3)
O1—C9—C8	113.9 (3)		
N2—N1—C71—C7	177.8 (3)	C10—C11—C12—O4	12.0 (6)
C10—C8—C9—O1	0.5 (5)		

Compound (2)

Crystal data

$C_{11}H_{10}Cl_2N_2O_4$
$M_r = 305.12$
Triclinic
$P\overline{1}$
$a = 7.279\ (1)$ Å
$b = 8.805\ (2)$ Å
$c = 11.753\ (2)$ Å
$\alpha = 73.15\ (1)°$
$\beta = 71.27\ (1)°$
$\gamma = 71.09\ (1)°$
$V = 660.2\ (2)$ Å^3
$Z = 2$
$D_x = 1.535$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 24 reflections
$\theta = 19.87–23.43°$
$\mu = 4.63$ mm^{-1}
$T = 295\ (3)$ K
Prismatic
0.22 × 0.14 × 0.11 mm
Yellowish

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2908 measured reflections
2687 independent reflections
2687 observed reflections [$I > 3\sigma(I)$]
$R_{\text{int}} = 0.0237$

$\theta_{\text{max}} = 74.14°$
$h = -9 \rightarrow 0$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 13$
3 standard reflections monitored every 80 reflections
frequency: 120 min
intensity decay: 7.8%

Refinement

Refinement on F^2
$R(F) = 0.0617$
$wR(F^2) = 0.1647$

$(\Delta/\sigma)_{\text{max}} < 0.001$
$\Delta\rho_{\text{max}} = 0.653$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.381$ e Å^{-3}

$S = 1.114$
2687 reflections
212 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1313P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)*

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

	x	y	z	U_{eq}
C31	0.2377 (4)	0.5017 (4)	0.0033 (3)	0.0436 (6)
C4	0.2649 (5)	0.4747 (4)	0.1194 (3)	0.0482 (7)
Cl1	0.3086 (2)	0.2750 (1)	0.20406 (8)	0.0665 (3)
C5	0.2628 (5)	0.5992 (4)	0.1696 (3)	0.0545 (8)
C6	0.2328 (5)	0.7561 (4)	0.0985 (3)	0.0543 (8)
Cl2	0.2377 (2)	0.9156 (1)	0.1551 (1)	0.0912 (4)
C7	0.2049 (5)	0.7901 (4)	−0.0173 (3)	0.0499 (7)
C71	0.2072 (4)	0.6617 (3)	−0.0654 (2)	0.0420 (6)
N1	0.1808 (4)	0.7001 (3)	−0.1832 (2)	0.0474 (6)
N2	0.2147 (4)	0.5800 (3)	−0.2412 (2)	0.0423 (5)
C8	0.1869 (5)	0.6122 (3)	−0.3493 (2)	0.0412 (6)
C9	0.1122 (5)	0.7791 (3)	−0.4184 (2)	0.0423 (6)
O1	0.0891 (5)	0.7827 (3)	−0.5248 (2)	0.0611 (7)
O2	0.0748 (4)	0.9033 (2)	−0.3788 (2)	0.0518 (6)
C10	0.2288 (6)	0.4704 (3)	−0.4096 (3)	0.0483 (7)
C11	0.3140 (6)	0.3063 (3)	−0.3361 (3)	0.0512 (8)
C12	0.3921 (5)	0.1728 (3)	−0.4082 (3)	0.0459 (7)
O3	0.4133 (6)	0.0254 (3)	−0.3403 (2)	0.080 (1)
O4	0.4338 (5)	0.1990 (3)	−0.5188 (2)	0.0728 (8)

Table 4. *Selected geometric parameters (Å, °) for (2)*

Cl1—C4	1.735 (3)	C4—C31	1.383 (4)
Cl2—C6	1.734 (4)	C5—C6	1.381 (4)
O1—C9	1.304 (4)	C6—C7	1.373 (4)
O2—C9	1.228 (3)	C7—C71	1.397 (4)
O3—C12	1.302 (3)	C8—C9	1.479 (4)
O4—C12	1.205 (3)	C8—C10	1.510 (4)
N1—N2	1.336 (4)	C10—C11	1.503 (4)
N1—C71	1.388 (3)	C11—C12	1.503 (3)
N2—C8	1.286 (3)	C31—C71	1.392 (4)
C4—C5	1.381 (5)		
N2—N1—C71	119.8 (2)	O2—C9—C8	122.8 (2)
N1—N2—C8	121.0 (2)	C8—C10—C11	113.8 (2)
C5—C4—C31	122.9 (3)	C10—C11—C12	112.6 (2)
C4—C5—C6	117.3 (2)	O3—C12—O4	122.8 (3)
C5—C6—C7	122.4 (3)	O3—C12—C11	113.8 (2)
C6—C7—C71	119.0 (3)	O4—C12—C11	123.4 (2)
N2—C8—C9	124.5 (2)	C4—C31—C71	118.1 (3)
N2—C8—C10	118.2 (2)	N1—C71—C7	117.5 (2)
C9—C8—C10	117.3 (2)	N1—C71—C31	122.2 (2)
O1—C9—O2	123.0 (2)	C7—C71—C31	120.3 (2)
O1—C9—C8	114.2 (2)		
N2—N1—C71—C7	168.9 (3)	C10—C11—C12—O4	19.3 (6)
C10—C8—C9—O1	0.8 (5)		

Table 5. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(1)				
N1—H1···O2	0.93 (5)	1.91 (5)	2.622 (4)	132 (4)
N1—H1···Cl2	0.93 (5)	2.51 (5)	2.928 (3)	108 (3)
O1—H11···O2^{i}	0.98 (5)	1.72 (5)	2.692 (4)	176 (5)
O3—H3···O4^{ii}	1.09 (7)	1.57 (7)	2.648 (4)	169 (5)
(2)				
N1—H1···O2	1.01 (4)	1.93 (4)	2.634 (3)	124 (3)
O1—H11···O2^{iii}	1.01 (5)	1.69 (5)	2.695 (3)	178 (4)
O3—H3···O4^{iv}	0.99 (7)	1.69 (7)	2.678 (3)	176 (7)

Symmetry codes: (i) $-2-x, 2-y, 1-z$; (ii) $-2-x, 1-y, 1-z$; (iii) $-x, 2-y, -1-z$; (iv) $1-x, -y, -1-z$.

As a result of poorly developed crystal faces, an analytical absorption correction was not possible, while experimental absorption corrections (*DIFABS*; Walker & Stuart, 1983) turned out to be inappropriate, giving unrealistic transmission factors.

The structures were solved by direct methods. All non-H atoms were located through difference Fourier synthesis and refined by full-matrix least-squares methods with anisotropic displacement parameters. All H atoms were determined from successive difference Fourier syntheses; in compound (1), atoms H11, H111 and H112 were normalized to the theoretical values and in compound (2), atoms H3, H31, H7, H11, H102 and H1 were normalized. H atoms were refined with an isotropic displacement parameter in the final run. Molecular geometries were calculated by the program *PLATON* (Spek, 1990*a*).

For both compounds, data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *HELENA* (Spek, 1990*b*); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *PLATON*93 (Spek, 1990*a*) and *ORTEP*II (Johnson, 1976), *PLUTON*93 (Spek 1991); software used to prepare material for publication: *PLATON*93.

This work was supported by the Ministry of Science and Technology, Republic of Croatia, grant No. 1–07–179.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1241). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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