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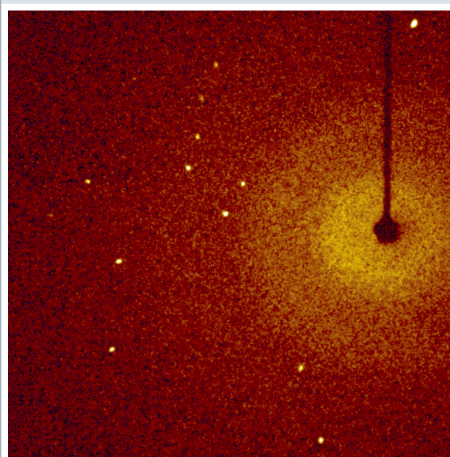
# Twinned L-aspartic acid

Martin Lutz\*

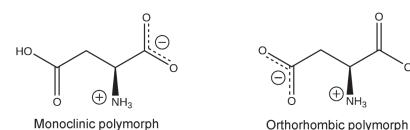
Structural Biochemistry, Bijvoet Centre for Biomolecular Research, Faculty of Science, Utrecht University, Universiteitsweg 99, 3584 CG Utrecht, The Netherlands. \*Correspondence e-mail: m.lutz@uu.nl

By improving the data quality with appropriate twin handling in the intensity integration, difference-Fourier maps show indications for asymmetric double-well hydrogen bonds between the carboxylate groups of the title molecule. Refinements with spherical scattering factors (IAM) and with non-spherical scattering factors (NoSpherA2) are consistent with this observation.

## Raw data



## Chemical scheme



**Bruker SMART data files and CBF files:** <https://doi.org/10.5281/zenodo.15432050>

**Metadata imgCIF file:** <https://doi.org/10.1107/S241431462500879X/ii4003img.cif>

## Introduction

The Cambridge Structural Database (CSD, version 5.46, November 2024; Groom *et al.*, 2016) contains seven entries for the enantiopure amino acid L-aspartic acid. Six of these entries are of the monoclinic polymorph with space group  $P2_1$ , and one entry is of the orthorhombic polymorph with space group  $P2_12_12_1$  (refcode LASPRT06; Illin, 2016). In the gas phase, the most stable form of aspartic acid is a neutral molecule with a neutral  $\text{NH}_2$  group and two neutral carboxylic acid groups (Li *et al.*, 2007). In the monoclinic L-aspartic acid, the molecule is zwitterionic with a positively charged  $\text{NH}_3$  group, the main chain carboxylate is deprotonated and negatively charged, while the side-chain carboxylic acid is protonated and neutral. The orthorhombic polymorph is also zwitterionic but here the main-chain carboxylic acid is protonated and the side-chain carboxylate is deprotonated (Fig. 1).

In both the monoclinic and the orthorhombic polymorphs, the neutral carboxylic acid group is connected by an intermolecular hydrogen bond to the negatively charged carboxylate group. This raises the question whether the bridging hydrogen atom is localized on one of the two groups. Unfortunately, the data quality of the six published monoclinic structures is not sufficient to answer this question. A problem is that this

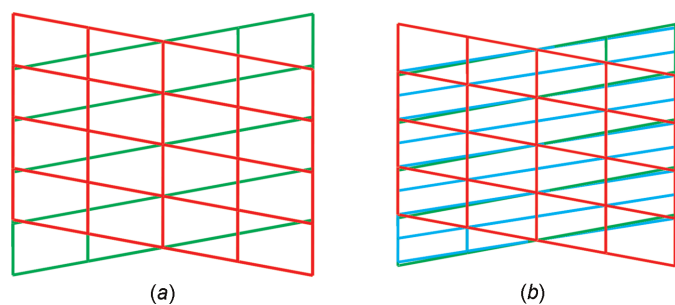
**Table 1**  
Experimental details.

Raw data			
DOI	<a href="https://doi.org/10.5281/zenodo.15432050">https://doi.org/10.5281/zenodo.15432050</a>		
Data archive	Zenodo		
Data format	CBF		
Data collection			
Beamline/diffractometer			
Detector	APEXII		
Temperature (K)	150		
Radiation type	Mo $K\alpha$		
Wavelength (Å)	0.71073		
Beam centre (mm)	30.485, 30.847		
Detector axis	-Z		
Detector distance (mm)	41		
Swing angle (°)	-26.72		
Pixel size (mm)	0.12 × 0.12		
No. of pixels	512 × 512		
No. of scans	7		
Exposure time per frame (s)	5		
Scan axis	Start angle, increment per frame (°)	Scan range (°)	No. of frames
$\phi$ , $X(\omega = 164.659^\circ, \kappa = 46.226^\circ)$	74.659, -0.300	-360.0	1200
$\omega$ , $X(\kappa = -73.760^\circ, \phi = 10.746^\circ)$	-174.596, -0.300	-118.2	394
$\omega$ , $X(\kappa = -73.760^\circ, \phi = -142.253^\circ)$	-174.596, -0.300	-118.2	394
$\omega$ , $X(\kappa = 88.307^\circ, \phi = -148.967^\circ)$	203.008, -0.300	-87.6	292
$\omega$ , $X(\kappa = -73.760^\circ, \phi = -91.254^\circ)$	-174.596, -0.300	-118.2	394
$\omega$ , $X(\kappa = -73.760^\circ, \phi = 115.747^\circ)$	-174.596, -0.300	-118.2	394
$\omega$ , $X(\kappa = 88.307^\circ, \phi = 4.033^\circ)$	203.008, -0.300	-87.6	292

monoclinic polymorph is affected by twinning. A detailed analysis of the twinning is given by (Derissen *et al.*, 1968) while the chirality of the asymmetric center seems to be wrong in their description. The present study (Table 1) aims at obtaining better data of this twinned system.

### Data processing and refinement

Indexing of the reflections with the *DIRAX* program (Duisenberg, 1992) and a high tolerance finds the twin cell, which had first been described in 1931 by Bernal. In this monoclinic twin cell, the unit-cell parameters are  $a = 15.134$ ,  $b = 6.918$ ,  $c = 5.124$  Å,  $\beta = 99.02^\circ$ , and  $V = 529.81$  Å<sup>3</sup>. Among the *DIRAX* solutions there also is the true unit cell according to (Derissen *et al.*, 1968) as well as the second twin component. The volume of the true unit cell is half of the Bernal unit cell. The twin law is then a twofold rotation about  $uvw = [100]$ . The geometry of the twin lattice is shown in Fig. 1.



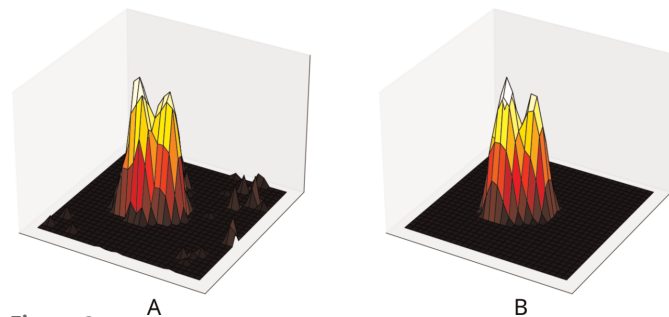
**Figure 1**  
Reciprocal space geometry in the twinned title compound. (a) The main lattice is drawn in red, the interfering lattice in green. (b) Overlay of Bernal's twin cell in cyan with the current twin interpretation in red and green.

As consequence of the twinning, intensity integration with the *Eval15* software (Schreurs *et al.*, 2010) was based on two orientation matrices. The profile prediction involved an isotropic mosaicity of  $0.275^\circ$ . An example of an overlapping reflection is displayed in Fig. 2.

The result file of the *Eval15* integration contains the non-overlapping reflections of twin component 1, the non-overlapping reflections of twin component 2 and the overlapping reflections of both components. This file was read into the *TWINABS* program (Sevvana *et al.*, 2019) for absorption correction, outlier rejection, error model and merging. After manual removal of space-group absences, the merged reflection file contains 1063 non-overlapping reflections of component 1 and 494 overlapping reflections. This file was used for the structure refinement. Further details are given in Table 2.

### Data description

The true unit cell can be transformed to Bernal's twin cell with the matrix  $(1, 0, 2 / 0, 1, 0 / -1, 0, 0)$ . The determinant of this



**Figure 2**  
Height plot of the overlapping reflection between  $hkl = (245)$  of the main lattice, and  $hkl = (244)$  of the interfering lattice. (a) observed profile (central frame). (b) model profile as simulated by *Eval15*.

Table 2

Experimental details (continued).

Crystal data		
Chemical formula	C <sub>4</sub> H <sub>7</sub> NO <sub>4</sub>	
<i>M<sub>r</sub></i>	133.105	
Crystal system, space group	monoclinic, <i>P</i> 2 <sub>1</sub>	
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.1237(2), 6.9197(3), 7.6006(4)	
$\beta$ (°)	100.442(2)	
<i>V</i> (Å <sup>3</sup> )	265.013(19)	
<i>Z</i>	2	
$\mu$ (mm <sup>-1</sup> )	0.151	
Crystal size (mm)	0.32 × 0.16 × 0.05	
Data processing		
Absorption correction	multi-scan ( <i>TWINABS2012/I</i> ; Sevvana <i>et al.</i> , 2019)	
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.6541, 0.7460	
Number of measured, independent and observed <i>I</i> > 2σ( <i>I</i> ) reflections	11270, 1557, 1522	
<i>R</i> <sub>int</sub>	0.0272	
(sin θ/λ) <sub>max</sub> (Å <sup>-1</sup> )	0.704	
Refinement		
	<b>Independent atom model</b>	<b>NoSpherA2</b>
No. of reflections	1557	1557
No. of parameters	99	141
H-atom treatment	O–H freely, C–H riding model	freely
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.0258, 0.0685, 1.0547	0.0165, 0.0369, 0.9865
Twin fraction	0.537(3)	0.5372(15)
Weighting scheme <sup>†</sup>	<i>a</i> = 0.0460, <i>b</i> = 0.0143	<i>a</i> = 0.0189, <i>b</i> = 0.0080
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	0.3047, -0.1757	0.3701, -0.01923
Bond precision C–C (Å)	0.0015	0.0010

<sup>†</sup>*a* and *b* are parameters of the *SHELXL* weighting scheme  $w = 1/[\sigma^2(F_o^2) + (a \times P)^2 + b \times P]$  with  $P = (F_o^2 + 2F_c^2)/3$ .

matrix is 2 and the twin index is consequently 2. On the other hand, Bernal's primitive twin cell can be expressed as a C-centered pseudo-orthorhombic lattice with *a* = 5.124, *b* = 29.901, *c* = 6.920 Å,  $\alpha = \beta = 90^\circ$ ,  $\gamma = 90.74^\circ$ . The twin obliquity (Le Page, 2002) of the current system is thus  $\delta = 0.74^\circ$ . The twinning fulfills Mallard's criterion (Nespolo & Ferraris, 2005), which requires a twin index smaller than 6 and a twin obliquity smaller than 6°.

It should be noted that the twin operation needs to be a first kind operation (rotation) because a second kind operation (mirror) is not possible in this enantiopure crystal.

All hydrogen bonds are formed in the (001) plane. These hydrogen-bonded layers are connected by covalent carbon bonds into a three-dimensional network. The twin operation about vector *uvw* = [100] can be alternatively be described as a twofold rotation about vector *hkl* = (001) in the monoclinic system. Our model for the twin boundary is therefore based on the hydrogen-bonded layers. It should be noted that face

(001) is also most prominent in the Bravais–Friedel–Donnay–Harker morphology prediction (BFDH; Donnay & Harker, 1937), see Table 3.

Structure refinement with an independent-atom model in the *OLEX2* software (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015) shows a significant residual electron density on the O–H···O hydrogen bond (Fig. 3), which can be an indication for a double-well situation. The peak height for the modeled hydrogen atom and the residual peak are not equal. A ratio of 2:1 can be guessed. This makes it an asymmetric double-well hydrogen bond, such as is frequently observed in hydrogen bonds of moderate strength (Gilli & Gilli, 2010).

In crystal structure refinements, the use of non-spherical scattering factors can improve the reliability of hydrogen-atom positions (Woińska *et al.*, 2016). In the present case, the use of the NoSpherA2 approach (Kleemiss *et al.*, 2021) in *OLEX2* improved the *R*-values significantly. As is common with this method, the hydrogen atoms were refined with anisotropic displacement parameters. The C–H and N–H

Table 3

BFDH morphology prediction as calculated with the *Mercury* software (Macrae *et al.*, 2006).

Face	Perp. distance	Relative area
(0, 0, 1)	13.3784	0.153
(0, -1, 1)	19.6933	0.081
(0, 1, 1)	19.6933	0.081
(1, 0, 0)	19.8458	0.086
(1, 0, -1)	21.8311	0.034
(1, -1, 0)	24.55	0.026
(1, 1, 0)	24.55	0.026
(1, -1, -1)	26.181	0.004
(1, 1, -1)	26.181	0.004

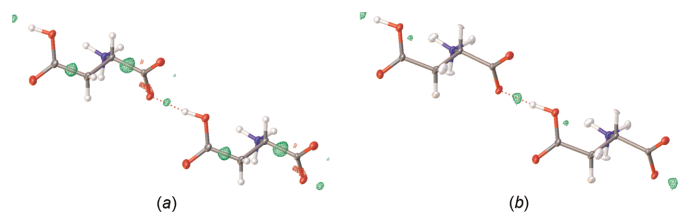


Figure 3

Residual electron density on the O–H···O hydrogen bond. (a) independent-atom model (contour level 0.16 e Å<sup>-3</sup>). (b) Non-spherical NoSpherA2 model (contour level 0.10 e Å<sup>-3</sup>).

hydrogen atoms could be refined this way but the O—H hydrogen atom becomes non-positive definite. In the final refinements, the O—H hydrogen atom was therefore refined isotropically. We believe that this refinement situation confirms the double-well potential as does the remaining residual electron density on the O—H···O hydrogen bond (Fig. 3).

### Acknowledgements

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### Conflict of interest

There are no conflicts of interest.

### Funding information

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### References

- Bernal, J. D. (1931). *Z. Krist. Crystal. Mater.* **78**, 363–369. <https://doi.org/10.1524/zkri.1931.78.1.363>
- Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* **A71**, 59–75. <https://doi.org/10.1107/S2053273314022207>
- Derissen, J. L., Endeman, H. J. & Peerdeman, A. F. (1968). *Acta Cryst.* **B24**, 1349–1354. <https://doi.org/10.1107/S0567740868004280>
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341. <https://doi.org/10.1107/S0021889808042726>
- Donnay, J. D. H. & Harker, D. (1937). *Am. Mineral.* **22**, 446–467.
- Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96. <https://doi.org/10.1107/S0021889891010634>
- Gilli, P. & Gilli, G. (2010). *J. Mol. Struct.* **972**, 2–10. *Horizons in hydrogen bond research 2009*. <https://www.sciencedirect.com/science/article/pii/S0022286010001262>
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **72**, 171–179. <https://doi.org/10.1107/S2052520616003954>
- Illin, A. I. (2016). Private Communication (refcode LASPRT06). CCDC, Cambridge, England.
- Kleemiss, F., Dolomanov, O. V., Bodensteiner, M., Peyrerimhoff, N., Midgley, L., Bourhis, L. J., Genoni, A., Malaspina, L. A., Jayatilaka, D., Spencer, J. L., White, F., Grundkötter-Stock, B., Steinhauer, S., Lentz, D., Puschmann, H. & Grabowsky, S. (2021). *Chem. Sci.* **12**, 1675–1692. <http://dx.doi.org/10.1039/D0SC05526C>
- Le Page, Y. (2002). *J. Appl. Cryst.* **35**, 175–181. <https://doi.org/10.1107/S0021889801021574>
- Li, Z., Matus, M. H., Velazquez, H. A., Dixon, D. A. & Cassady, C. J. (2007). *Int. J. Mass Spec.* **265**, 213–223. Jean H. Futrell Honour Issue. <https://www.sciencedirect.com/science/article/pii/S1387380607000681>
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457. <https://doi.org/10.1107/S002188980600731X>
- Nespolo, M. & Ferraris, G. (2005). *Z. Krist. Cryst. Mater.* **220**, 317–323. <https://doi.org/10.1524/zkri.220.4.317.61622>
- Schreurs, A. M. M., Xian, X. & Kroon-Batenburg, L. M. J. (2010). *J. Appl. Cryst.* **43**, 70–82. <https://doi.org/10.1107/S0021889809043234>
- Sevvana, M., Ruf, M., Usón, I., Sheldrick, G. M. & Herbst-Irmer, R. (2019). *Acta Cryst.* **D75**, 1040–1050. <https://doi.org/10.1107/S2059798319010179>
- Woińska, M., Grabowsky, S., Dominiak, P. M., Woźniak, K. & Jayatilaka, D. (2016). *Sci. Adv.* **2**, e1600192. <https://www.science.org/doi/abs/10.1126/sciadv.1600192>

## full crystallographic data

*IUCrData* (2025). **10**, x250879 [https://doi.org/10.1107/S241431462500879X]

## Twinned L-aspartic acid

Martin Lutz

(Ia)

*Crystal data*

$C_4H_7NO_4$

$M_r = 133.10$

Monoclinic,  $P2_1$

$a = 5.1237$  (2) Å

$b = 6.9197$  (3) Å

$c = 7.6006$  (4) Å

$\beta = 100.442$  (2)°

$V = 265.01$  (2) Å<sup>3</sup>

$Z = 2$

$F(000) = 140.133$

$D_x = 1.668$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4378 reflections

$\theta = 4.0$ – $30.0$ °

$\mu = 0.15$  mm<sup>-1</sup>

$T = 150$  K

Plate, colourless

$0.32 \times 0.16 \times 0.05$  mm

*Data collection*

Bruker Kappa ApexII  
diffractometer

Radiation source: sealed tube

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
TWINABS-2012/1

$T_{\min} = 0.654$ ,  $T_{\max} = 0.746$

11270 measured reflections

1557 independent reflections

1522 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 30.0$ °,  $\theta_{\min} = 2.7$ °

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -10 \rightarrow 10$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.069$

$S = 1.06$

1557 reflections

99 parameters

1 restraint

5 constraints

Primary atom site location: dual

Secondary atom site location: difference Fourier  
map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.0143P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.31$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.18$  e Å<sup>-3</sup>

Absolute structure: Hooft, R.W.W., Straver,  
L.H., Spek, A.L. (2010). *J. Appl. Cryst.*, **43**,  
665–668.

Absolute structure parameter: 0.1 (2)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.68132 (15)	0.51119 (13)	0.01802 (11)	0.01613 (18)
O2	0.25187 (16)	0.52336 (13)	-0.10959 (10)	0.01648 (18)
O3	0.38789 (16)	0.48596 (13)	0.58289 (11)	0.01603 (17)

H3	0.333 (5)	0.489 (5)	0.691 (3)	0.051 (6)*
O4	0.0023 (2)	0.33238 (17)	0.49259 (13)	0.0283 (2)
N1	0.14057 (19)	0.71721 (14)	0.18338 (12)	0.01218 (18)
H1a	-0.019 (3)	0.662 (3)	0.118 (2)	0.018 (4)*
H1b	0.102 (4)	0.754 (3)	0.295 (3)	0.023 (4)*
H1c	0.183 (4)	0.826 (4)	0.126 (3)	0.033 (5)*
C1	0.4400 (2)	0.53553 (15)	0.02088 (13)	0.01090 (19)
C2	0.3650 (2)	0.57621 (15)	0.20458 (14)	0.01019 (19)
H2	0.5217 (2)	0.63118 (15)	0.28706 (14)	0.0122 (2)*
C3	0.2852 (3)	0.38469 (16)	0.27988 (14)	0.0144 (2)
H3a	0.1335 (3)	0.33005 (16)	0.19522 (14)	0.0172 (2)*
H3b	0.4351 (3)	0.29300 (16)	0.28733 (14)	0.0172 (2)*
C4	0.2085 (2)	0.40003 (15)	0.46296 (14)	0.0136 (2)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0128 (3)	0.0202 (4)	0.0161 (3)	0.0004 (3)	0.0045 (3)	-0.0042 (3)
O2	0.0151 (4)	0.0252 (4)	0.0095 (3)	-0.0014 (4)	0.0030 (3)	-0.0030 (3)
O3	0.0178 (4)	0.0199 (4)	0.0109 (3)	-0.0024 (3)	0.0041 (3)	-0.0026 (3)
O4	0.0306 (5)	0.0407 (6)	0.0151 (4)	-0.0180 (5)	0.0085 (4)	-0.0031 (4)
N1	0.0144 (4)	0.0121 (4)	0.0108 (4)	0.0022 (4)	0.0041 (3)	0.0004 (3)
C1	0.0144 (4)	0.0095 (4)	0.0100 (4)	-0.0016 (4)	0.0054 (3)	-0.0006 (4)
C2	0.0120 (4)	0.0109 (5)	0.0080 (4)	0.0012 (3)	0.0027 (4)	-0.0009 (3)
C3	0.0239 (5)	0.0108 (4)	0.0093 (4)	-0.0009 (4)	0.0057 (4)	0.0006 (4)
C4	0.0203 (5)	0.0117 (4)	0.0090 (4)	-0.0003 (4)	0.0033 (4)	0.0013 (4)

*Geometric parameters (Å, °)*

O1—C1	1.2522 (13)	N1—C2	1.4943 (14)
O2—C1	1.2541 (13)	C1—C2	1.5398 (14)
O3—H3	0.91 (3)	C2—H2	1.0000
O3—C4	1.3136 (14)	C2—C3	1.5283 (15)
O4—C4	1.2136 (16)	C3—H3a	0.9900
N1—H1a	0.957 (18)	C3—H3b	0.9900
N1—H1b	0.941 (19)	C3—C4	1.5166 (14)
N1—H1c	0.92 (2)		
C4—O3—H3	110.1 (16)	C3—C2—N1	110.72 (9)
H1b—N1—H1a	106.0 (15)	C3—C2—C1	107.98 (8)
H1c—N1—H1a	109.8 (17)	C3—C2—H2	109.51 (6)
H1c—N1—H1b	108.0 (18)	H3a—C3—C2	108.64 (6)
C2—N1—H1a	111.4 (11)	H3b—C3—C2	108.64 (6)
C2—N1—H1b	111.2 (12)	H3b—C3—H3a	107.6
C2—N1—H1c	110.3 (13)	C4—C3—C2	114.47 (8)
O2—C1—O1	126.66 (10)	C4—C3—H3a	108.64 (6)
C2—C1—O1	116.74 (9)	C4—C3—H3b	108.64 (6)
C2—C1—O2	116.55 (9)	O4—C4—O3	124.69 (10)

C1—C2—N1	109.58 (8)	C3—C4—O3	113.79 (10)
H2—C2—N1	109.51 (5)	C3—C4—O4	121.49 (10)
H2—C2—C1	109.51 (5)		
O1—C1—C2—N1	143.71 (10)	O3—C4—C3—C2	−53.03 (11)
O1—C1—C2—C3	−95.61 (11)	O4—C4—C3—C2	129.02 (12)
O2—C1—C2—N1	−38.75 (11)	N1—C2—C3—C4	−60.79 (9)
O2—C1—C2—C3	81.93 (11)	C1—C2—C3—C4	179.25 (8)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O3—H3 $\cdots$ O2 <sup>i</sup>	0.91 (3)	1.66 (3)	2.5703 (12)	172 (3)
N1—H1 <i>a</i> $\cdots$ O1 <sup>ii</sup>	0.957 (18)	1.896 (18)	2.8420 (13)	169.2 (16)
N1—H1 <i>b</i> $\cdots$ O4 <sup>iii</sup>	0.941 (19)	1.870 (19)	2.8089 (13)	175.3 (17)
N1—H1 <i>c</i> $\cdots$ O1 <sup>iv</sup>	0.92 (2)	1.90 (2)	2.7965 (12)	166 (2)

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

**(Ib)***Crystal data*

C<sub>4</sub>H<sub>7</sub>NO<sub>4</sub>

$M_r = 133.10$

Monoclinic,  $P2_1$

$a = 5.1237$  (2) Å

$b = 6.9197$  (3) Å

$c = 7.6006$  (4) Å

$\beta = 100.442$  (2)°

$V = 265.01$  (2) Å<sup>3</sup>

$Z = 2$

$F(000) = 140.133$

$D_x = 1.668$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4378 reflections

$\theta = 4.0$ – $30.0^\circ$

$\mu = 0.15$  mm<sup>−1</sup>

$T = 150$  K

Plate, colourless

$0.32 \times 0.16 \times 0.05$  mm

*Data collection*

Bruker Kappa ApexII

diffractometer

Radiation source: sealed tube

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

TWINABS-2012/1

$T_{\min} = 0.654$ ,  $T_{\max} = 0.746$

11270 measured reflections

1557 independent reflections

1522 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 30.0^\circ$ ,  $\theta_{\min} = 2.7^\circ$

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -10 \rightarrow 10$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.037$

$S = 0.99$

1557 reflections

141 parameters

1 restraint

0 constraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2 + 0.008P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.37$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.19$  e Å<sup>−3</sup>

Absolute structure: Hooft, R.W.W., Straver,  
L.H., Spek, A.L. (2010). *J. Appl. Cryst.*, 43,  
665-668.

Absolute structure parameter: 0.1 (2)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.68076 (10)	0.51134 (8)	0.01788 (7)	0.01490 (12)
O2	0.25205 (10)	0.52322 (8)	-0.10925 (6)	0.01523 (12)
O3	0.38709 (11)	0.48573 (9)	0.58294 (7)	0.01495 (12)
H3	0.324 (3)	0.498 (3)	0.6996 (18)	0.032 (3)*
O4	0.00246 (15)	0.33224 (10)	0.49271 (8)	0.02682 (15)
N1	0.14053 (14)	0.71719 (9)	0.18330 (9)	0.01114 (13)
H1a	-0.026 (2)	0.6565 (18)	0.1121 (16)	0.024 (3)
H1b	0.101 (3)	0.761 (2)	0.3010 (17)	0.036 (3)
H1c	0.195 (3)	0.8350 (19)	0.1147 (16)	0.030 (3)
C1	0.43986 (14)	0.53548 (10)	0.02129 (8)	0.00959 (13)
C2	0.36425 (15)	0.57630 (10)	0.20459 (9)	0.00931 (13)
H2a	0.5401 (19)	0.6369 (18)	0.2944 (14)	0.020 (3)
C3	0.28535 (18)	0.38494 (11)	0.28029 (10)	0.01333 (14)
H3a	0.122 (3)	0.3189 (19)	0.1867 (16)	0.034 (3)
H3b	0.468 (3)	0.291 (2)	0.2925 (18)	0.038 (4)
C4	0.20931 (16)	0.40001 (10)	0.46282 (9)	0.01254 (14)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0111 (2)	0.0199 (3)	0.0145 (2)	0.0005 (2)	0.00426 (19)	-0.0048 (2)
O2	0.0133 (2)	0.0251 (3)	0.0077 (2)	-0.0014 (3)	0.00291 (19)	-0.0029 (2)
O3	0.0162 (3)	0.0190 (3)	0.0103 (2)	-0.0025 (2)	0.0039 (2)	-0.0023 (2)
O4	0.0289 (3)	0.0405 (4)	0.0128 (2)	-0.0194 (3)	0.0083 (2)	-0.0043 (3)
N1	0.0130 (3)	0.0111 (3)	0.0097 (3)	0.0027 (3)	0.0032 (3)	0.0001 (3)
H1a	0.016 (6)	0.014 (6)	0.038 (8)	0.004 (6)	-0.008 (6)	-0.003 (7)
H1b	0.045 (8)	0.030 (8)	0.032 (7)	0.014 (7)	0.002 (7)	-0.004 (6)
H1c	0.049 (9)	0.015 (7)	0.029 (7)	0.006 (7)	0.018 (7)	0.002 (6)
C1	0.0105 (3)	0.0106 (3)	0.0084 (3)	-0.0010 (3)	0.0034 (2)	-0.0011 (2)
C2	0.0119 (3)	0.0101 (3)	0.0065 (3)	-0.0006 (3)	0.0031 (3)	-0.0013 (2)
H2a	0.012 (6)	0.022 (6)	0.026 (6)	-0.012 (5)	0.005 (5)	-0.004 (5)
C3	0.0227 (4)	0.0100 (3)	0.0081 (3)	-0.0010 (3)	0.0050 (3)	0.0002 (3)
H3a	0.057 (9)	0.019 (7)	0.027 (7)	-0.014 (7)	0.011 (7)	-0.004 (6)
H3b	0.034 (8)	0.045 (10)	0.035 (8)	0.027 (8)	0.007 (7)	0.004 (7)
C4	0.0184 (3)	0.0123 (3)	0.0077 (3)	-0.0017 (3)	0.0043 (3)	0.0008 (2)

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

O1—C1	1.2505 (9)	N1—C2	1.4911 (10)
O2—C1	1.2536 (8)	C1—C2	1.5383 (10)
O3—H3	1.001 (14)	C2—H2a	1.109 (10)

O3—C4	1.3090 (9)	C2—C3	1.5271 (10)
O4—C4	1.2175 (10)	C3—H3a	1.096 (13)
N1—H1a	1.013 (12)	C3—H3b	1.128 (13)
N1—H1b	1.001 (13)	C3—C4	1.5111 (10)
N1—H1c	1.032 (13)		
C4—O3—H3	111.1 (8)	C3—C2—N1	110.92 (6)
H1b—N1—H1a	108.5 (11)	C3—C2—C1	108.02 (5)
H1c—N1—H1a	109.5 (10)	C3—C2—H2a	109.7 (6)
H1c—N1—H1b	109.0 (11)	H3a—C3—C2	109.9 (6)
C2—N1—H1a	110.0 (7)	H3b—C3—C2	104.9 (8)
C2—N1—H1b	112.3 (8)	H3b—C3—H3a	109.8 (11)
C2—N1—H1c	107.4 (7)	C4—C3—C2	114.58 (6)
O2—C1—O1	126.40 (6)	C4—C3—H3a	109.3 (6)
C2—C1—O1	117.06 (6)	C4—C3—H3b	108.2 (7)
C2—C1—O2	116.49 (6)	O4—C4—O3	124.31 (7)
C1—C2—N1	109.67 (6)	C3—C4—O3	114.14 (7)
H2a—C2—N1	110.0 (6)	C3—C4—O4	121.52 (7)
H2a—C2—C1	108.5 (5)		
O1—C1—C2—N1	143.52 (7)	O3—C4—C3—C2	-53.08 (7)
O1—C1—C2—C3	-95.49 (8)	O4—C4—C3—C2	128.79 (8)
O2—C1—C2—N1	-38.83 (8)	N1—C2—C3—C4	-60.75 (7)
O2—C1—C2—C3	82.16 (7)	C1—C2—C3—C4	179.04 (6)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 $\cdots$ O2 <sup>i</sup>	1.001 (14)	1.572 (14)	2.5702 (7)	174.6 (13)
N1—H1a $\cdots$ O1 <sup>ii</sup>	1.013 (12)	1.843 (13)	2.8435 (9)	168.9 (11)
N1—H1b $\cdots$ O4 <sup>iii</sup>	1.001 (13)	1.811 (13)	2.8086 (9)	174.5 (13)
N1—H1c $\cdots$ O1 <sup>iv</sup>	1.032 (13)	1.773 (13)	2.7977 (9)	171.0 (11)

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