Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-4-(4-Methylstyryl)pyridine–(*E*)-but-2-enedioic acid (2/1)

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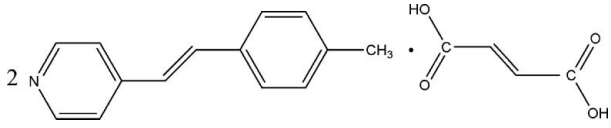
Received 29 June 2007; accepted 7 July 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.141; data-to-parameter ratio = 8.7.

In the title compound, $2C_{14}H_{13}N\cdot C_4H_4O_2$, the asymmetric unit consists of two crystallographically independent (*E*)-4-(4-methylstyryl)pyridine and one (*E*)-but-2-enedioic acid molecules, which are linked through O—H···N hydrogen bonds to form a linear arrangement.

Related literature

For general background, see: Chung *et al.* (1991); Yam *et al.* (1998). For related structures, see: Ayyappan *et al.* (2004); Lin *et al.* (2000).



Experimental

Crystal data

$2C_{14}H_{13}N\cdot C_4H_4O_4$
M_r = 506.58
Monoclinic, *Cc*
a = 21.952 (1) Å
b = 7.3477 (3) Å
c = 17.0000 (7) Å
β = 104.610 (2)°
V = 2653.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 291 (2) K
0.25 × 0.24 × 0.23 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.979, T_{max} = 0.981
12115 measured reflections
3033 independent reflections
2450 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.141
S = 1.00
3033 reflections
347 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.82	1.74	2.548 (3)	169
O3—H3···N2	0.82	1.74	2.528 (3)	161

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge financial support from the National Natural Science Foundation of China (grant No. 20473032), the PCSIRT of the Ministry of Education of China (grant No. IRT0422), the '111' project (grant No. B06009) and the Innovation Fund of Jilin University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2213).

References

Ayyappan, P., Sirokman, G., Evans, O. R., Warren, T. H. & Lin, W. B. (2004). *Inorg. Chim. Acta*, **357**, 3999–4004.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Chung, C. M. & Hasegawa, M. (1991). *J. Am. Chem. Soc.* **113**, 7311–7316.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Lin, W. B., Ma, L. & Evans, O. R. (2000). *Chem. Commun.* pp. 2263–2264.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yam, V. W. W., Lau, V. C. Y. & Wu, L. X. (1998). *J. Chem. Soc. Dalton Trans.* pp. 1461–1468.

  doi:10.1107/S1600536807033260

supplementary materials

Acta Cryst. (2007). E**63**, o3466 [**doi:10.1107/S1600536807033260**]

(*E*)-4-(4-Methylstyryl)pyridine-(*E*)-but-2-enedioic acid (2/1)

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Comment

(*E*)-4-(4-methylstyryl)pyridine and its ramifications have attracted extensive attention because of their photophysical, photochemical and electrochemical properties (Chung *et al.*, 1991; Yam *et al.*, 1998). They are also be used widely for building up supramolecular networks through covalent bonds with metals (Ayyappan *et al.*, 2004; Lin *et al.*, 2000). We report here the crystal structure of the title compound, (I).

The asymmetric unit of (I) contains two independent (*E*)-4-(4-methylstyryl)pyridine and one (E)-but-2-enedioic acid molecules and they are linked by stong O - H ··· N hydrogen bonds into a linear arrangement (Fig.1). The three molecules are all nearly planar and the diheral angles of (E)-but-2-enedioic acid molecule and two (*E*)-4-(4-methylstyryl)pyridine molecules are 4.86 (5)° and 16.51 (7)°, respectively.

Experimental

A mixture of 4-methylpyridine (0.93 g, 10 mmol)and 4-methylbenzaldehyde (0.96 g, 10 mmol) in Acetic anhydride (50 ml) was heated to 413 K for 6 h. After cooling, filtration and drying, (*E*)-4-(4-methylstyryl)pyridine was obtained. (E)-but-2-enedioic acid was used as purchased without further purification. (*E*)-4-(4-methylstyryl)pyridine (0.39 g, 2 mmol) and (E)-but-2-enedioic acid (0.12 g, 1 mmol) were dissolved in DMF (30 ml), the mixture was stirred for 2 h at room temperature. The filtrate was stand at room temperature for two months, colorless crystals suitable for single-crystal X-ray measurement were obtained

Refinement

All H atoms attached to C atoms and O atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.96 Å (methylene) and O—H = 0.82 Å with U_{iso}(H) = xU_{eq} where x = 1.2 for C(aromatic) and O atoms or 1.5 for methyl group.

In the absence of significant anomalous scattering, the absolute structure could not be reliably determined and then the Friedel pairs were merged and any references to the Flack parameter were removed.

Figures

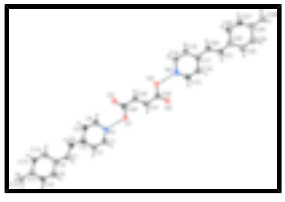


Fig. 1. The asymmetric unit of (I), with the atom- numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level. O - H ··· N hydrogen bonds are indicated by dashed lines. H atoms are represented as small spheres of arbitrary radii.

(*E*)-4-(4-Methylstyryl)pyridine–(E)-but-2-enedioic acid (2/1)

Crystal data

$2C_{14}H_{13}N·C_4H_4O_4$	F_{000} = 1072
M_r = 506.58	D_x = 1.268 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 9893 reflections
a = 21.952 (1) Å	θ = 3.8–54.9º
b = 7.3477 (3) Å	μ = 0.08 mm^{-1}
c = 17.0000 (7) Å	*T* = 291 (2) K
β = 104.610 (2)º	Block, colourless
V = 2653.4 (2) Å^3	0.25 × 0.24 × 0.23 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	3033 independent reflections
Radiation source: fine-focus sealed tube	2450 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 291(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	*h* = −28→28
T_{min} = 0.979, T_{max} = 0.981	*k* = −9→9
12115 measured reflections	*l* = −22→21

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.052	$w = 1/[\sigma^2(F_o^2) + (0.1053P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.141	$(\Delta/\sigma)_{max}$ = 0.024
S = 1.00	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
3033 reflections	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
347 parameters	Extinction correction: none
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C15	0.60752 (15)	0.6051 (5)	0.34113 (19)	0.0475 (8)
H15	0.5903	0.5805	0.3847	0.057*
C16	0.56773 (14)	0.6125 (5)	0.2632 (2)	0.0469 (7)
H16	0.5247	0.5945	0.2550	0.056*
C17	0.59308 (16)	0.6471 (4)	0.19811 (19)	0.0426 (7)
C18	0.65698 (16)	0.6731 (5)	0.2138 (2)	0.0509 (8)
H18	0.6756	0.6968	0.1714	0.061*
C19	0.69323 (16)	0.6642 (5)	0.2921 (2)	0.0503 (8)
H19	0.7364	0.6813	0.3013	0.060*
C20	0.55604 (15)	0.6522 (4)	0.11228 (18)	0.0439 (7)
H20	0.5785	0.6646	0.0730	0.053*
C21	0.49369 (15)	0.6406 (4)	0.08599 (19)	0.0432 (7)
H21	0.4707	0.6305	0.1249	0.052*
C22	0.45867 (17)	0.6426 (4)	0.0007 (2)	0.0463 (7)
C23	0.39503 (16)	0.6076 (5)	−0.0194 (2)	0.0508 (8)
H23	0.3752	0.5854	0.0219	0.061*
C24	0.35978 (17)	0.6046 (5)	−0.0993 (2)	0.0528 (8)
H24	0.3169	0.5792	−0.1106	0.063*
C25	0.38713 (17)	0.6386 (4)	−0.1625 (2)	0.0488 (9)
C26	0.3479 (2)	0.6359 (6)	−0.2505 (2)	0.0645 (11)
H26A	0.3148	0.5475	−0.2563	0.097*
H26B	0.3743	0.6045	−0.2857	0.097*
H26C	0.3298	0.7540	−0.2650	0.097*
C27	0.45072 (17)	0.6768 (5)	−0.1433 (2)	0.0528 (8)
H27	0.4701	0.7017	−0.1848	0.063*
C28	0.48591 (16)	0.6788 (5)	−0.0640 (2)	0.0534 (8)
H28	0.5287	0.7047	−0.0530	0.064*
N2	0.66980 (12)	0.6322 (4)	0.35565 (15)	0.0429 (6)
C1	0.90157 (14)	0.5770 (4)	1.00260 (17)	0.0405 (6)
H1	0.8618	0.5307	1.0005	0.049*
C2	0.94592 (15)	0.5850 (4)	1.07603 (17)	0.0427 (7)
H2	0.9354	0.5465	1.1231	0.051*
C3	1.00631 (14)	0.6502 (4)	1.08064 (18)	0.0414 (7)

C4	1.01803 (13)	0.7114 (4)	1.00812 (17)	0.0435 (7)
H4	1.0573	0.7590	1.0080	0.052*
C5	0.97168 (14)	0.7011 (4)	0.93701 (18)	0.0416 (7)
H5	0.9803	0.7419	0.8892	0.050*
C6	1.05832 (15)	0.6634 (5)	1.15393 (19)	0.0450 (7)
H6	1.0948	0.7226	1.1497	0.054*
C7	1.05689 (15)	0.5970 (4)	1.22547 (19)	0.0448 (7)
H7	1.0202	0.5387	1.2294	0.054*
C8	1.10877 (16)	0.6076 (4)	1.29986 (18)	0.0431 (7)
C9	1.09635 (15)	0.5528 (5)	1.37169 (19)	0.0496 (8)
H9	1.0566	0.5082	1.3712	0.059*
C10	1.14133 (15)	0.5627 (5)	1.44370 (18)	0.0458 (7)
H10	1.1310	0.5240	1.4908	0.055*
C11	1.20160 (15)	0.6274 (4)	1.44981 (19)	0.0411 (7)
C12	1.2495 (2)	0.6363 (5)	1.5301 (2)	0.0601 (10)
H12A	1.2286	0.6551	1.5727	0.090*
H12B	1.2780	0.7353	1.5296	0.090*
H12C	1.2727	0.5242	1.5394	0.090*
C13	1.21454 (16)	0.6831 (5)	1.3776 (2)	0.0491 (8)
H13	1.2544	0.7269	1.3782	0.059*
C14	1.16861 (18)	0.6742 (5)	1.3042 (2)	0.0524 (8)
H14	1.1784	0.7140	1.2569	0.063*
C29	0.86448 (15)	0.6534 (4)	0.73719 (18)	0.0410 (7)
C30	0.82186 (14)	0.6520 (4)	0.65343 (17)	0.0396 (7)
H30	0.8396	0.6712	0.6099	0.048*
C31	0.76064 (15)	0.6252 (4)	0.63758 (18)	0.0417 (7)
H31	0.7427	0.6083	0.6811	0.050*
C32	0.71838 (13)	0.6204 (4)	0.55357 (17)	0.0396 (7)
N1	0.91506 (12)	0.6349 (3)	0.93414 (14)	0.0363 (5)
O1	0.83709 (10)	0.6285 (3)	0.79486 (12)	0.0462 (5)
H1A	0.8624	0.6449	0.8388	0.069*
O2	0.92070 (10)	0.6735 (4)	0.74538 (13)	0.0568 (6)
O3	0.74528 (10)	0.6312 (4)	0.49519 (13)	0.0488 (6)
H3	0.7190	0.6548	0.4528	0.073*
O4	0.66121 (11)	0.6046 (4)	0.54446 (15)	0.0623 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C15	0.0463 (17)	0.063 (2)	0.0318 (15)	0.0077 (14)	0.0067 (13)	−0.0051 (14)
C16	0.0359 (16)	0.0521 (17)	0.0466 (17)	0.0040 (12)	−0.0009 (13)	−0.0070 (14)
C17	0.0474 (17)	0.0378 (15)	0.0364 (15)	0.0037 (12)	−0.0012 (12)	−0.0046 (12)
C18	0.0479 (17)	0.064 (2)	0.0386 (16)	−0.0007 (15)	0.0075 (13)	−0.0005 (14)
C19	0.0410 (16)	0.067 (2)	0.0397 (17)	−0.0015 (14)	0.0033 (13)	0.0002 (14)
C20	0.0434 (17)	0.0549 (19)	0.0300 (14)	0.0017 (13)	0.0033 (12)	−0.0010 (12)
C21	0.0415 (16)	0.0472 (17)	0.0379 (15)	0.0023 (12)	0.0044 (13)	−0.0026 (13)
C22	0.0475 (17)	0.0399 (15)	0.0425 (17)	0.0016 (12)	−0.0055 (13)	−0.0022 (13)
C23	0.0495 (18)	0.0551 (19)	0.0440 (17)	0.0007 (14)	0.0045 (14)	−0.0001 (14)

C24	0.0426 (17)	0.0528 (19)	0.054 (2)	0.0020 (14)	−0.0047 (14)	−0.0045 (15)
C25	0.053 (2)	0.0395 (18)	0.0427 (18)	0.0021 (13)	−0.0086 (15)	−0.0037 (13)
C26	0.067 (2)	0.069 (3)	0.0405 (19)	0.0032 (17)	−0.0161 (17)	−0.0050 (16)
C27	0.0517 (18)	0.0572 (19)	0.0474 (19)	0.0003 (15)	0.0083 (15)	0.0081 (15)
C28	0.0417 (16)	0.0551 (19)	0.057 (2)	−0.0011 (14)	0.0001 (15)	0.0048 (16)
N2	0.0395 (14)	0.0538 (15)	0.0313 (13)	0.0044 (11)	0.0011 (10)	−0.0059 (11)
C1	0.0396 (14)	0.0480 (16)	0.0318 (14)	0.0002 (12)	0.0054 (11)	0.0000 (12)
C2	0.0487 (16)	0.0478 (16)	0.0286 (14)	0.0045 (13)	0.0043 (12)	0.0037 (12)
C3	0.0414 (15)	0.0398 (16)	0.0367 (16)	0.0076 (12)	−0.0019 (12)	−0.0091 (12)
C4	0.0379 (14)	0.0484 (16)	0.0423 (17)	−0.0037 (13)	0.0066 (13)	−0.0050 (13)
C5	0.0418 (15)	0.0453 (16)	0.0378 (15)	0.0014 (12)	0.0103 (13)	0.0006 (13)
C6	0.0427 (16)	0.0513 (17)	0.0363 (15)	−0.0025 (13)	0.0013 (13)	−0.0024 (13)
C7	0.0437 (15)	0.0439 (16)	0.0445 (18)	0.0014 (13)	0.0069 (14)	−0.0007 (13)
C8	0.0499 (17)	0.0396 (16)	0.0337 (15)	0.0079 (13)	−0.0006 (13)	−0.0020 (12)
C9	0.0435 (16)	0.0543 (19)	0.0467 (18)	−0.0002 (14)	0.0037 (14)	−0.0020 (15)
C10	0.0492 (17)	0.0552 (18)	0.0311 (14)	0.0028 (14)	0.0063 (12)	0.0009 (13)
C11	0.0435 (16)	0.0387 (16)	0.0351 (15)	0.0058 (12)	−0.0010 (13)	−0.0010 (12)
C12	0.058 (2)	0.062 (2)	0.049 (2)	0.0056 (16)	−0.0080 (17)	−0.0022 (16)
C13	0.0457 (17)	0.0525 (18)	0.0475 (18)	−0.0027 (14)	0.0089 (14)	0.0020 (15)
C14	0.071 (2)	0.0507 (18)	0.0362 (17)	0.0089 (16)	0.0157 (16)	0.0067 (13)
C29	0.0411 (16)	0.0510 (17)	0.0287 (14)	0.0015 (13)	0.0048 (12)	−0.0077 (12)
C30	0.0381 (16)	0.0531 (17)	0.0269 (14)	0.0014 (12)	0.0069 (12)	−0.0012 (12)
C31	0.0368 (15)	0.0555 (19)	0.0319 (15)	0.0043 (12)	0.0070 (12)	0.0029 (13)
C32	0.0305 (14)	0.0493 (17)	0.0357 (15)	0.0032 (12)	0.0022 (11)	0.0019 (13)
N1	0.0340 (12)	0.0419 (13)	0.0289 (11)	0.0027 (9)	0.0006 (9)	−0.0017 (9)
O1	0.0397 (11)	0.0734 (15)	0.0235 (9)	0.0009 (10)	0.0044 (8)	0.0016 (10)
O2	0.0336 (11)	0.1018 (19)	0.0336 (10)	−0.0073 (11)	0.0058 (9)	−0.0056 (12)
O3	0.0359 (10)	0.0776 (16)	0.0285 (11)	0.0020 (10)	0.0002 (8)	−0.0010 (10)
O4	0.0340 (11)	0.106 (2)	0.0431 (12)	0.0028 (12)	0.0025 (9)	0.0082 (13)

Geometric parameters (Å, °)

C15—N2	1.341 (4)	C3—C6	1.465 (4)
C15—C16	1.392 (4)	C4—C5	1.372 (4)
C15—H15	0.9300	C4—H4	0.9300
C16—C17	1.382 (5)	C5—N1	1.324 (4)
C16—H16	0.9300	C5—H5	0.9300
C17—C18	1.374 (5)	C6—C7	1.318 (4)
C17—C20	1.481 (4)	C6—H6	0.9300
C18—C19	1.369 (5)	C7—C8	1.475 (4)
C18—H18	0.9300	C7—H7	0.9300
C19—N2	1.329 (4)	C8—C9	1.377 (5)
C19—H19	0.9300	C8—C14	1.386 (5)
C20—C21	1.331 (4)	C9—C10	1.367 (4)
C20—H20	0.9300	C9—H9	0.9300
C21—C22	1.460 (4)	C10—C11	1.385 (5)
C21—H21	0.9300	C10—H10	0.9300
C22—C23	1.376 (5)	C11—C13	1.390 (5)
C22—C28	1.404 (5)	C11—C12	1.500 (4)

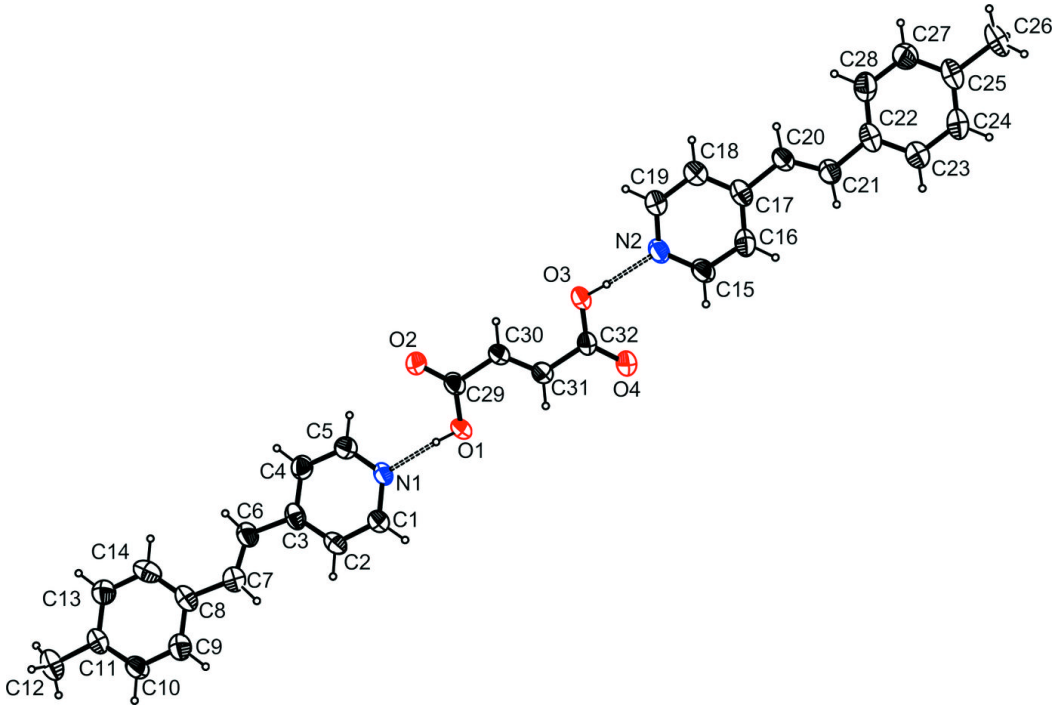
C23—C24	1.383 (5)	C12—H12A	0.9600
C23—H23	0.9300	C12—H12B	0.9600
C24—C25	1.380 (6)	C12—H12C	0.9600
C24—H24	0.9300	C13—C14	1.394 (5)
C25—C27	1.380 (5)	C13—H13	0.9300
C25—C26	1.527 (5)	C14—H14	0.9300
C26—H26A	0.9600	C29—O2	1.216 (4)
C26—H26B	0.9600	C29—O1	1.286 (4)
C26—H26C	0.9600	C29—C30	1.494 (4)
C27—C28	1.373 (5)	C30—C31	1.317 (4)
C27—H27	0.9300	C30—H30	0.9300
C28—H28	0.9300	C31—C32	1.494 (4)
C1—N1	1.341 (4)	C31—H31	0.9300
C1—C2	1.377 (4)	C32—O4	1.231 (4)
C1—H1	0.9300	C32—O3	1.279 (4)
C2—C3	1.393 (5)	O1—H1A	0.8200
C2—H2	0.9300	O3—H3	0.8200
C3—C4	1.396 (4)		
N2—C15—C16	122.3 (3)	C2—C3—C6	126.6 (3)
N2—C15—H15	118.8	C4—C3—C6	116.9 (3)
C16—C15—H15	118.8	C5—C4—C3	120.0 (3)
C17—C16—C15	119.1 (3)	C5—C4—H4	120.0
C17—C16—H16	120.4	C3—C4—H4	120.0
C15—C16—H16	120.4	N1—C5—C4	122.2 (3)
C18—C17—C16	117.9 (3)	N1—C5—H5	118.9
C18—C17—C20	117.9 (3)	C4—C5—H5	118.9
C16—C17—C20	124.2 (3)	C7—C6—C3	124.4 (3)
C19—C18—C17	119.8 (3)	C7—C6—H6	117.8
C19—C18—H18	120.1	C3—C6—H6	117.8
C17—C18—H18	120.1	C6—C7—C8	125.4 (3)
N2—C19—C18	123.3 (3)	C6—C7—H7	117.3
N2—C19—H19	118.3	C8—C7—H7	117.3
C18—C19—H19	118.3	C9—C8—C14	117.0 (3)
C21—C20—C17	126.3 (3)	C9—C8—C7	117.5 (3)
C21—C20—H20	116.9	C14—C8—C7	125.5 (3)
C17—C20—H20	116.9	C10—C9—C8	121.2 (3)
C20—C21—C22	124.9 (3)	C10—C9—H9	119.4
C20—C21—H21	117.6	C8—C9—H9	119.4
C22—C21—H21	117.6	C9—C10—C11	123.1 (3)
C23—C22—C28	116.6 (3)	C9—C10—H10	118.5
C23—C22—C21	119.5 (3)	C11—C10—H10	118.5
C28—C22—C21	124.0 (3)	C10—C11—C13	116.0 (3)
C22—C23—C24	121.9 (3)	C10—C11—C12	121.4 (3)
C22—C23—H23	119.1	C13—C11—C12	122.6 (3)
C24—C23—H23	119.1	C11—C12—H12A	109.5
C25—C24—C23	121.1 (3)	C11—C12—H12B	109.5
C25—C24—H24	119.5	H12A—C12—H12B	109.5
C23—C24—H24	119.5	C11—C12—H12C	109.5
C27—C25—C24	117.7 (3)	H12A—C12—H12C	109.5

C27—C25—C26	121.5 (4)	H12B—C12—H12C	109.5
C24—C25—C26	120.8 (3)	C11—C13—C14	121.0 (3)
C25—C26—H26A	109.5	C11—C13—H13	119.5
C25—C26—H26B	109.5	C14—C13—H13	119.5
H26A—C26—H26B	109.5	C8—C14—C13	121.7 (3)
C25—C26—H26C	109.5	C8—C14—H14	119.2
H26A—C26—H26C	109.5	C13—C14—H14	119.2
H26B—C26—H26C	109.5	O2—C29—O1	125.9 (3)
C28—C27—C25	121.3 (4)	O2—C29—C30	118.9 (3)
C28—C27—H27	119.3	O1—C29—C30	115.1 (3)
C25—C27—H27	119.3	C31—C30—C29	123.9 (3)
C27—C28—C22	121.4 (3)	C31—C30—H30	118.1
C27—C28—H28	119.3	C29—C30—H30	118.1
C22—C28—H28	119.3	C30—C31—C32	123.7 (3)
C19—N2—C15	117.5 (3)	C30—C31—H31	118.2
N1—C1—C2	120.9 (3)	C32—C31—H31	118.2
N1—C1—H1	119.6	O4—C32—O3	124.3 (3)
C2—C1—H1	119.6	O4—C32—C31	119.3 (3)
C1—C2—C3	120.7 (3)	O3—C32—C31	116.3 (3)
C1—C2—H2	119.6	C5—N1—C1	119.7 (3)
C3—C2—H2	119.6	C29—O1—H1A	109.5
C2—C3—C4	116.5 (3)	C32—O3—H3	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···N1	0.82	1.74	2.548 (3)	169
O3—H3···N2	0.82	1.74	2.528 (3)	161

Fig. 1


C27
C26
C28
C25
C22
C24
C18
C20
C19
C23
C21
C17
N2
C16
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C15
O2
C30
C32
C29
C31
O4
C5
O1
C4
N1
C6
C1
C3
C2
C14
C7
C13
C8
C9
C11
C12
C10