

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Deacetyl tenuazonic acid

David Siegel,* Matthias Koch, Franziska Emmerling and Irene Nehls

Bundesanstalt für Materialforschung und -prüfung, Abteilung Analytische Chemie; Referenzmaterialien, Richard-Willstätter-Strasse 11, D-12489 Berlin-Adlershof, Germany

Correspondence e-mail: david.siegel@bam.de

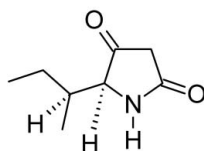
Received 23 April 2009; accepted 24 April 2009

Key indicators: single-crystal X-ray study; $T = 193$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 15.3.

The heterocycle in the title compound {systematic name: (5*S*)-5-[(1*S*)-1-methylpropyl]pyrrolidine-2,4-dione}, $\text{C}_8\text{H}_{13}\text{NO}_2$, is planar (r.m.s. deviation for all non-H atoms = 0.008 Å). The crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding.

Related literature

Tenuazonic acid (TA) is an *Alternaria* mycotoxin commonly encountered in food (Siegel, Rasenko *et al.*, 2009; Weidenbörner, 2001). The title compound is known to be formed upon boiling TA in 0.1 M HCl (Stickings, 1959). For the synthesis of the title compound, see: Lebrun *et al.* (1988). For the crystal structure of the tenuazonic acid copper (II) salt, see: Dippenaar *et al.* (1977) and for the 2,4-dinitrophenylhydrazone, see: Siegel, Merkel *et al.* (2009). For the structures of other pyrrolidine-2,4-diones, see, for example: Yu *et al.* (2007); Zhu *et al.* (2004); Ellis & Spek (2001).



Experimental

Crystal data

$\text{C}_8\text{H}_{13}\text{NO}_2$
 $M_r = 155.19$
 Monoclinic, $P2_1$
 $a = 5.0114$ (4) Å
 $b = 7.7961$ (4) Å
 $c = 10.9919$ (10) Å
 $\beta = 95.778$ (4)°

$V = 427.26$ (6) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 0.71$ mm⁻¹
 $T = 193$ K
 $0.44 \times 0.16 \times 0.16$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (CORINC; Dräger & Gattow, 1971)
 $T_{\min} = 0.744$, $T_{\max} = 0.993$
 (expected range = 0.669–0.893)

1866 measured reflections
 1571 independent reflections
 1558 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 3 standard reflections
 frequency: 60 min
 intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.098$
 $S = 1.06$
 1571 reflections
 103 parameters
 1 restraint

H-atom parameters constrained
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³
 Absolute structure: Flack (1983), 697 Friedel pairs
 Flack parameter: 0.1 (2)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.90	2.02	2.8963 (18)	164

Symmetry code: (i) $-x, y - \frac{1}{2}, -z + 1$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2937).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Dippenaar, A., Holzapfel, C. W. & Boeyens, J. C. A. (1977). *J. Chem. Crystallogr.* **7**, 189–197.
- Dräger, M. & Gattow, G. (1971). *Acta Chem. Scand.* **25**, 761–762.
- Ellis, D. D. & Spek, A. L. (2001). *Acta Cryst.* **C57**, 433–434.
- Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Lebrun, M. H., Nicolas, L., Boutar, M., Gaudemer, F., Ranomenjanahary, S. & Gaudemer, A. (1988). *Phytochemistry*, **27**, 77–84.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Siegel, D., Merkel, S., Koch, M., Emmerling, F. & Nehls, I. (2009). *Acta Cryst.* **E65**, o988–o989.
- Siegel, D., Rasenko, T., Koch, M. & Nehls, I. (2009). *J. Chromatogr. A* **1216**, 4582–4588.
- Stickings, C. E. (1959). *Biochem. J.* **72**, 332–340.
- Weidenbörner, M. (2001). In *Encyclopedia of Food Mycotoxins*. Berlin: Springer.
- Yu, G.-S., Xu, H.-Z. & Zhu, Y.-Q. (2007). *Acta Cryst.* **E63**, o3384.
- Zhu, Y.-Q., Song, H.-B., Li, J.-R., Yao, C.-S., Hu, F.-Z., Zou, X.-M. & Yang, H.-Z. (2004). *Acta Cryst.* **E60**, o196–o198.

supplementary materials

Acta Cryst. (2009). E65, o1201 [doi:10.1107/S1600536809015372]

Deacetyl tenuazonic acid

D. Siegel, M. Koch, F. Emmerling and I. Nehls

Comment

Tenuazonic acid (TA) is an *Alternaria* mycotoxin commonly encountered in food (Siegel, Rasenko *et al.*, 2009; Weidenbörner, 2001). The title compound is known to be formed upon boiling of TA in 0.1 M HCl (Stickings, 1959). It is therefore a possible degradation product which might also be encountered in food matrices.

Whereas TA itself could so far only be crystallized as its copper (II) salt (Dippenaar *et al.*, 1977) or 2,4-dinitrophenyl-hydrazone (Siegel, Merkel *et al.*, 2009), the title compound is conveniently crystallized from hexane/ethyl acetate.

Each molecule (Fig. 1) is connected to two adjacent molecules *via* N—H···O hydrogen bonds. Along the *b* axis chains of symmetry equivalent molecules are formed (Fig. 2).

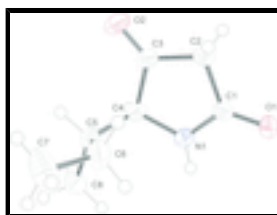
Experimental

The title compound was supplied by the workgroup of Professor R. Faust (University of Kassel, Germany) by synthesis according to a literature procedure (Lebrun *et al.*, 1988). For *x*-ray analysis, it was recrystallized several times from hexane:ethyl acetate 50:50 (v:v).

Refinement

The hydrogen atoms were located in difference maps but positioned with idealized geometry and refined using the riding model, with C—H = 0.98–1.00 Å or N—H = 0.90 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Figures



(5S)-5-[(1S)-1-methylpropyl]pyrrolidine-2,4-dione

Crystal data

$C_8H_{13}NO_2$	$F_{000} = 168$
$M_r = 155.19$	$D_x = 1.206 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Cu $K\alpha$ radiation
Hall symbol: P 2yb	$\lambda = 1.54178 \text{ \AA}$
$a = 5.0114 (4) \text{ \AA}$	Cell parameters from 25 reflections
$b = 7.7961 (4) \text{ \AA}$	$\theta = 67\text{--}69^\circ$
$c = 10.9919 (10) \text{ \AA}$	$\mu = 0.71 \text{ mm}^{-1}$
$\beta = 95.778 (4)^\circ$	$T = 193 \text{ K}$
$V = 427.26 (6) \text{ \AA}^3$	Block, yellow
$Z = 2$	$0.44 \times 0.16 \times 0.16 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.040$
Monochromator: graphite	$\theta_{\text{max}} = 69.9^\circ$
$T = 193 \text{ K}$	$\theta_{\text{min}} = 4.0^\circ$
$\omega/2\theta$ scans	$h = -6 \rightarrow 5$
Absorption correction: ψ scan (CORINC; Dräger & Gattow, 1971)	$k = -8 \rightarrow 9$
$T_{\text{min}} = 0.744$, $T_{\text{max}} = 0.993$	$l = -13 \rightarrow 13$
1866 measured reflections	3 standard reflections
1571 independent reflections	every 60 min
1558 reflections with $I > 2\sigma(I)$	intensity decay: 2%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.0771P]$
$wR(F^2) = 0.098$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1571 reflections	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
103 parameters	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
1 restraint	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.017 (4)
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 697 Friedel pairs
	Flack parameter: 0.1 (2)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	−0.0559 (2)	0.66044 (16)	0.50335 (11)	0.0386 (3)
O2	0.5911 (3)	0.74694 (19)	0.22570 (14)	0.0494 (4)
N1	0.2318 (3)	0.48864 (18)	0.41048 (11)	0.0303 (3)
H1	0.1866	0.3946	0.4519	0.036*
C1	0.1196 (3)	0.6392 (2)	0.43372 (14)	0.0305 (3)
C2	0.2400 (3)	0.7779 (2)	0.36022 (15)	0.0360 (4)
H2A	0.1012	0.8320	0.3021	0.043*
H2B	0.3265	0.8675	0.4145	0.043*
C3	0.4437 (3)	0.6853 (2)	0.29328 (15)	0.0335 (4)
C4	0.4354 (3)	0.4941 (2)	0.32393 (13)	0.0296 (3)
H4	0.6122	0.4594	0.3672	0.036*
C5	0.3757 (3)	0.3822 (2)	0.20975 (14)	0.0317 (4)
H5	0.5040	0.4161	0.1500	0.038*
C6	0.0924 (4)	0.4130 (3)	0.14885 (16)	0.0425 (4)
H6A	−0.0373	0.3594	0.1995	0.051*
H6B	0.0571	0.5380	0.1467	0.051*
C7	0.0441 (6)	0.3429 (4)	0.0202 (2)	0.0761 (8)
H7A	0.1721	0.3946	−0.0307	0.114*
H7B	−0.1391	0.3706	−0.0138	0.114*
H7C	0.0679	0.2181	0.0219	0.114*
C8	0.4266 (4)	0.1937 (2)	0.2416 (2)	0.0474 (5)
H8A	0.6088	0.1804	0.2821	0.071*
H8B	0.4071	0.1250	0.1666	0.071*
H8C	0.2967	0.1549	0.2966	0.071*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0498 (7)	0.0316 (6)	0.0377 (6)	−0.0010 (5)	0.0201 (5)	−0.0054 (5)
O2	0.0530 (8)	0.0418 (8)	0.0576 (8)	−0.0067 (6)	0.0262 (6)	0.0115 (6)
N1	0.0349 (7)	0.0268 (7)	0.0303 (6)	−0.0028 (5)	0.0094 (5)	0.0017 (5)
C1	0.0374 (8)	0.0264 (8)	0.0279 (7)	−0.0056 (6)	0.0046 (6)	−0.0030 (6)

supplementary materials

C2	0.0462 (9)	0.0253 (8)	0.0378 (8)	-0.0062 (7)	0.0104 (7)	-0.0022 (7)
C3	0.0348 (8)	0.0312 (8)	0.0347 (8)	-0.0065 (6)	0.0043 (6)	0.0024 (7)
C4	0.0268 (7)	0.0308 (8)	0.0321 (7)	-0.0025 (6)	0.0063 (5)	0.0042 (7)
C5	0.0310 (8)	0.0312 (8)	0.0346 (8)	0.0006 (6)	0.0120 (6)	-0.0011 (6)
C6	0.0373 (9)	0.0512 (11)	0.0391 (9)	0.0028 (7)	0.0037 (7)	-0.0102 (8)
C7	0.0824 (18)	0.087 (2)	0.0547 (14)	0.0218 (14)	-0.0144 (12)	-0.0302 (13)
C8	0.0558 (11)	0.0317 (9)	0.0573 (12)	0.0060 (8)	0.0182 (8)	-0.0003 (8)

Geometric parameters (Å, °)

O1—C1	1.2338 (19)	C5—C8	1.526 (2)
O2—C3	1.199 (2)	C5—C6	1.526 (2)
N1—C1	1.337 (2)	C5—H5	1.0000
N1—C4	1.4640 (18)	C6—C7	1.512 (3)
N1—H1	0.9038	C6—H6A	0.9900
C1—C2	1.511 (2)	C6—H6B	0.9900
C2—C3	1.501 (2)	C7—H7A	0.9800
C2—H2A	0.9900	C7—H7B	0.9800
C2—H2B	0.9900	C7—H7C	0.9800
C3—C4	1.530 (2)	C8—H8A	0.9800
C4—C5	1.533 (2)	C8—H8B	0.9800
C4—H4	1.0000	C8—H8C	0.9800
C1—N1—C4	115.63 (14)	C6—C5—C4	111.45 (13)
C1—N1—H1	119.0	C8—C5—H5	107.6
C4—N1—H1	125.2	C6—C5—H5	107.6
O1—C1—N1	125.18 (14)	C4—C5—H5	107.6
O1—C1—C2	125.70 (14)	C7—C6—C5	114.04 (16)
N1—C1—C2	109.12 (14)	C7—C6—H6A	108.7
C3—C2—C1	104.25 (14)	C5—C6—H6A	108.7
C3—C2—H2A	110.9	C7—C6—H6B	108.7
C1—C2—H2A	110.9	C5—C6—H6B	108.7
C3—C2—H2B	110.9	H6A—C6—H6B	107.6
C1—C2—H2B	110.9	C6—C7—H7A	109.5
H2A—C2—H2B	108.9	C6—C7—H7B	109.5
O2—C3—C2	127.06 (17)	H7A—C7—H7B	109.5
O2—C3—C4	123.96 (16)	C6—C7—H7C	109.5
C2—C3—C4	108.98 (13)	H7A—C7—H7C	109.5
N1—C4—C3	101.98 (13)	H7B—C7—H7C	109.5
N1—C4—C5	115.10 (13)	C5—C8—H8A	109.5
C3—C4—C5	112.44 (13)	C5—C8—H8B	109.5
N1—C4—H4	109.0	H8A—C8—H8B	109.5
C3—C4—H4	109.0	C5—C8—H8C	109.5
C5—C4—H4	109.0	H8A—C8—H8C	109.5
C8—C5—C6	112.26 (15)	H8B—C8—H8C	109.5
C8—C5—C4	110.21 (14)		
C4—N1—C1—O1	179.64 (15)	C2—C3—C4—N1	-1.75 (16)
C4—N1—C1—C2	0.22 (18)	O2—C3—C4—C5	-57.6 (2)
O1—C1—C2—C3	179.26 (14)	C2—C3—C4—C5	122.08 (14)
N1—C1—C2—C3	-1.33 (18)	N1—C4—C5—C8	-75.53 (17)

C1—C2—C3—O2	-178.44 (17)	C3—C4—C5—C8	168.27 (14)
C1—C2—C3—C4	1.90 (17)	N1—C4—C5—C6	49.81 (19)
C1—N1—C4—C3	0.95 (16)	C3—C4—C5—C6	-66.39 (17)
C1—N1—C4—C5	-121.07 (15)	C8—C5—C6—C7	-70.6 (3)
O2—C3—C4—N1	178.57 (16)	C4—C5—C6—C7	165.2 (2)

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

<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>
N1—H1 \cdots O1 ⁱ	0.90	2.02	2.8963 (18)	164

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

Fig. 1

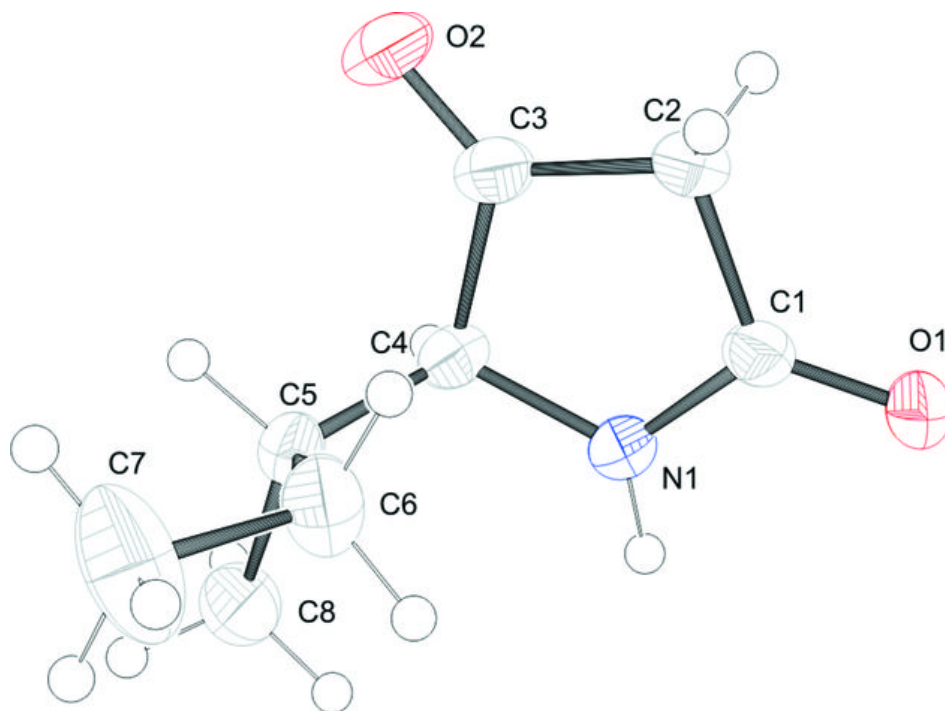


Fig. 2

