$\times\ 10^{12}\ e^2\ Å^{-3}$. This is comparable with the study of a very small crystal of piperazine silicate whose scattering efficiency was $200\ \times\ 10^{12}\ e^2\ Å^{-3}$ and which required a focused synchrotron radiation beam and electronic area detector for the data collection (Andrews *et al.*, 1988). However, in the piperazine silicate case the crystal mosaic spread was also very broad (approx 3°) compared with 0.6° here. Hence, the advantages of the high intensity and Cu $K\alpha$ wavelength of a laboratory rotating-anode diffractometer were adequate, compared with a laboratory sealed tube Mo $K\alpha$ diffractometer (Helliwell, Gallois, Kariuki, Kaucic & Helliwell, 1993), to obtain a data set sufficient to solve and refine the structure of this fructose deriviative. Obviously a much more intense synchrotron beam could be utilized to further improve the data-to-parameter ratio and will be the subject of a future study.

The structure was solved by direct methods. Due to the small crystal volume and rather poor quality, only the O atoms and five C atoms were refined anisotropically, the remaining non-H atoms being refined isotropically. H atoms attached to C atoms were placed in calculated positions which were updated after each refinement and assigned isotropic displacement parameters 20% greater than the $B_{\rm eq}$ value of the bonded atom. Those attached to the N and O atoms were not included. Full-matrix least-squares refinement of 127 parameters minimized the function $\sum w(|F_o| - |F_c|)^2$.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1985). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984); *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *PLUTO* (Motherwelll & Clegg, 1978). Software used to prepare material for publication: *TEXSAN*.

The authors wish to thank the CDCHT–ULA (Grants C-539-92 and C-404-89), the Intercambio Cientifico Commission, from ULA-Venezuela, and the British Council for financial support, and SUMA, Facultad de Ciencias, ULA-Venezuela, for computational support. Also the authors wish to thank the SERC for the funding of a Rigaku AFC-5*R* diffractometer. Professor J. R. Helliwell is thanked for discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Andrews, S. J., Papiz, M. Z., McMeeking, R., Blake, A. J., Lowe, B. M., Franklin, K. R., Helliwell, J. R. & Harding, M. M. (1988). *Acta Cryst.* B**44**, 73–77.

Baynes, J. W., Thorpe, S. R. & Murtiashaw, M. H. (1984). *Method. Enzymol.* **106**, 88–98.

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Technical Report 1984/1 Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, Netherlands

Cerami, A., Vlassara, H. & Browlee, M. (1987). *Sci. Ann.* **256**, 82–88.

Cohen, M. P. (1986). In *Diabetes and Protein Gycosylation. Measurement and Biological Relevance.* New York: Springer-Verlag.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol IV, Table 2.2A, Birmingham: The Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Funcke, W. & Klemer, A. (1976). *Carbohydr. Res.* **50**, 9.

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Helliwell, M., Gallois, B., Kariuki, B. M., Kaucic, V. & Helliwell, J. R. (1993). *Acta Cryst.* B**49**, 420–428.

Lowrey, C. H., Lyness, S. J. & Soeldner, S. (1985). *J. Biol. Chem.* **260**, 11611–11618.

Molecular Structure Corporation. (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Monnier, V. M., Kohn, R. R. & Cerami, A. (1984). *Proc. Natl Acad. Sci. USA*, **81**, 583.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* University of Cambridge, England.

Neglia, C. I., Cohen, H. J., Garber, A. R., Ellis, P. D., Thorpe, S. R. & Baynes, J. W. (1983). *J. Biol. Chem.* **258**, 14279.

Neglia, C. I., Cohen, H. J., Garber, A. R., Thorpe, S. R. & Baynes, J. W. (1985). *J. Biol. Chem.* **260**, 5406.

Shaklai, N., Garlick, R. L. & Bunn, H. F. (1984). *J. Biol. Chem.* **259**, 3812–3817.

Watkins, N. G., Thorpe, S. R. & Baynes, J. W. (1987). *J. Biol. Chem.* **260**, 10629–10630.

Acta Cryst. (1996). C**52**, 254–256

6-[1,2-(*Z*)-Bis(methoxycarbonyl)vinyl]amino-5-dimethylamino-2-methoxy-3-methyl-4(3*H*)-pyrimidinone

JOHN N. LOW,[a] GEORGE FERGUSON,[b] JUSTO COBO,[c] MANUEL NOGUERAS[c] AND ADOLFO SÁNCHEZ[c]

[a]*Department of Applied Physics and Electronic & Mechanical Engineering, University of Dundee, Dundee DD1 4HN, Scotland,* [b]*Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1, and* [c]*Departamento de Química Inorgánica y Orgánica, Facultad de Ciencias Experimentales, Universidad de Jaén, 23071 Jaén, Spain*

(*Received 15 August 1995; accepted 11 September 1995*)

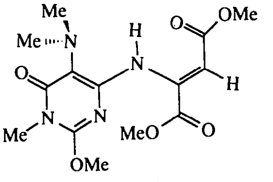
Abstract

The molecular conformation of the title compound, dimethyl (*Z*)-1-[5-dimethylamino-2-methoxy-3-methyl-4(3*H*)-oxo-6-pyrimidinylamino]ethylene-1,2-dicarboxylate, $C_{14}H_{20}N_4O_6$, in the solid state is determined by an intramolecular bifurcated hydrogen-bond system involving the H atom on the amino N6 atom and adjacent dimethylamino N5 and carbonyl O61 atoms [N6···N5 2.722 (2) and N6···O61 2.767 (2) Å].

Acta Crystallographica Section C
ISSN 0108-2701 ©1996

Comment

6-Vinylaminopyrimidines are intermediates in the synthesis of other fused heterocyclic derivatives such as imidazo[1,2-*c*]pyrimidines or pyrimido[3,4-*b*]pyrimidines, which are of interest because of their potential biological activity (Abignente *et al.*, 1991). The imidazo[1,2-*c*]pyrimidines have shown anti-inflammatory, analgesic and antipyretic effects. We report here the structure of the title compound, (I), which has been examined in order to determine details of the solid-state conformation.



(I)

A view of compound (I) with the atomic numbering scheme is shown in Fig. 1. Intramolecular bond distances are in accordance with accepted values (Table 2), but some of the bond angles are increased from values of 120° in order to relieve intramolecular overcrowding effects. The molecular conformation is determined by an intramolecular bifurcated hydrogen-bond system involving the H atom on the amino N6 atom and adjacent dimethylamino N5 and carbonyl O61 atoms; details are in Table 2. In the (*Z*)-1,2-di(methoxycarbonyl)vinyl chain, one group of atoms (N6, C61, C65, C62, C63, O61, O62, C64) is essentially planar [maximum deviation 0.041 (1) Å], while the plane of the carbomethoxy group (C65, O63, O64, C66) is inclined at an angle of 67.4 (1)° to that plane. Intermolecular distances correspond with van der Waals contacts.

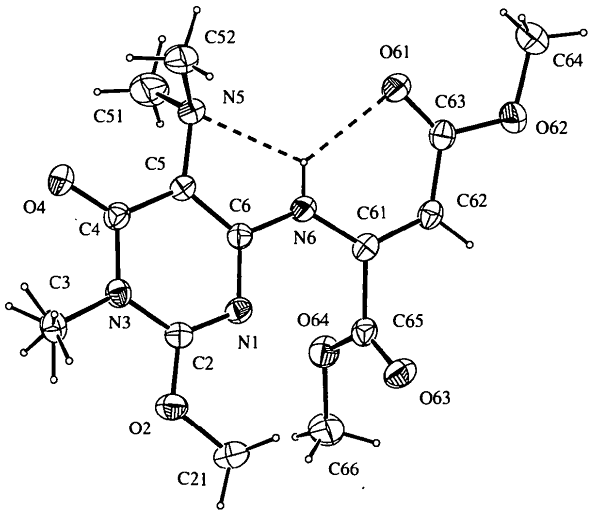


Fig. 1. The molecular structure of compound (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The C3 methyl group is disordered.

Experimental

The title compound was synthesized by the Michael addition of 6-amino-5-dimethylamino-2-methoxy-3-methyl-4(3*H*)-pyrimidinone to dimethyl acetylenedicarboxylate (DMAD) in acetonitrile under reflux (yield 33%). Crystals suitable for X-ray analysis were obtained by recrystallization from methanol.

Crystal data

$C_{14}H_{20}N_4O_6$
$M_r = 340.34$
Monoclinic
$P2_1/c$
$a = 8.7777\ (7)$ Å
$b = 16.8264\ (18)$ Å
$c = 11.3773\ (9)$ Å
$\beta = 93.077\ (7)°$
$V = 1678.0\ (3)$ Å^3
$Z = 4$
$D_x = 1.347$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 24 reflections
$\theta = 9.70$–$17.10°$
$\mu = 0.107$ mm^{-1}
$T = 294\ (1)$ K
Prism
0.35 × 0.35 × 0.30 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3876 measured reflections
3649 independent reflections
2578 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.020$
$\theta_{\rm max} = 26.90°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: 2.0%

Refinement

Refinement on F^2
$R(F) = 0.0386$
$wR(F^2) = 0.1169$
$S = 1.058$
3649 reflections
221 parameters
H atoms riding (*SHELXL*93 defaults, C—H 0.93–0.96, N—H 0.86 Å)
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.1984P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.224$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.183$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.019 (2)
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)*

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

	x	y	z	$U_{\rm eq}$
N1	0.19663 (14)	0.47983 (7)	0.96155 (10)	0.0383 (3)
C2	0.2819 (2)	0.53966 (8)	0.99381 (12)	0.0374 (3)
O2	0.29166 (14)	0.60372 (6)	0.92629 (9)	0.0492 (3)
C21	0.2098 (2)	0.60130 (11)	0.81324 (14)	0.0571 (5)
N3	0.36895 (14)	0.54446 (7)	1.09562 (10)	0.0392 (3)
C3	0.4594 (2)	0.61550 (10)	1.1259 (2)	0.0501 (4)
C4	0.3713 (2)	0.48146 (9)	1.17793 (12)	0.0404 (3)
O4	0.45224 (14)	0.48739 (7)	1.26896 (10)	0.0560 (3)
C5	0.2767 (2)	0.41529 (9)	1.14412 (12)	0.0390 (3)
N5	0.2639 (2)	0.34681 (8)	1.21654 (11)	0.0499 (4)
C51	0.3978 (3)	0.29618 (13)	1.2195 (2)	0.0821 (7)
C52	0.2137 (3)	0.36489 (15)	1.3347 (2)	0.0748 (6)

C6	0.1951 (2)	0.41790 (8)	1.03830 (12)	0.0362 (3)
N6	0.10380 (15)	0.35396 (7)	1.00521 (10)	0.0419 (3)
C61	0.0149 (2)	0.34444 (8)	0.90399 (12)	0.0370 (3)
C62	−0.0948 (2)	0.28872 (9)	0.88973 (12)	0.0420 (4)
C63	−0.1352 (2)	0.23432 (9)	0.98169 (13)	0.0436 (4)
O61	−0.07274 (14)	0.22914 (7)	1.07893 (10)	0.0554 (3)
O62	−0.25496 (15)	0.18862 (8)	0.94632 (10)	0.0608 (4)
C64	−0.3063 (3)	0.13186 (14)	1.0302 (2)	0.0818 (7)
C65	0.0416 (2)	0.39400 (9)	0.79632 (12)	0.0406 (3)
O63	−0.05085 (14)	0.43827 (7)	0.75195 (10)	0.0557 (3)
O64	0.17414 (13)	0.37463 (7)	0.75262 (9)	0.0510 (3)
C66	0.2054 (3)	0.40910 (15)	0.64033 (15)	0.0709 (6)

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

N1—C2	1.296 (2)	N5—C52	1.469 (2)
N1—C6	1.360 (2)	N6—C6	1.382 (2)
O2—C2	1.329 (2)	N6—C61	1.365 (2)
C2—N3	1.355 (2)	C61—C62	1.348 (2)
O2—C21	1.440 (2)	C61—C65	1.511 (2)
N3—C3	1.466 (2)	C62—C63	1.448 (2)
N3—C4	1.414 (2)	O61—C63	1.211 (2)
O4—C4	1.228 (2)	O62—C63	1.346 (2)
C4—C5	1.430 (2)	O62—C64	1.440 (2)
N5—C5	1.425 (2)	O63—C65	1.193 (2)
C5—C6	1.368 (2)	O64—C65	1.330 (2)
N5—C51	1.450 (3)	O64—C66	1.443 (2)
C6—N6—C61	128.16 (12)	N6—C61—C65	120.85 (13)
N6—C61—C62	123.74 (13)	C61—C62—C63	123.63 (13)
C62—C61—C65	115.30 (12)		
C4—C5—N5—C51	−73.3 (2)	N6—C61—C65—O64	67.8 (2)
C4—C5—N5—C52	56.9 (2)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···O61	0.86	2.14	2.767 (2)	129
N6—H6···N5	0.86	2.32	2.722 (2)	109

A study of $\Delta\rho$ map sections showed that the H atoms on atom C3 were disordered. This disorder was allowed for by means of the AFIX 127 option in *SHELXL*93 (Sheldrick, 1993). The diagram (Fig. 1) was prepared using *ORTEP*II (Johnson, 1976), as implemented in *PLATON* (Spek, 1995*a*). Examination of the structure with *PLATON* showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD*-4/*PC Software* (Enraf–Nonius, 1992). Cell refinement: Enraf–Nonius *SET*4 (de Boer & Duisenberg, 1984) and *CELDIM*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*. Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93. Molecular graphics: *NRCVAX*94, *PLATON* and *PLUTON* (Spek 1995*b*). Software used to prepare material for publication: *NRCVAX*94, *SHELXL*93 and WordPerfect.

GF thanks NSERC (Canada) for Research Grants and JC thanks Junta de Andalucia (Spain) for a Research Grant.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1307). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Abignente, A., Arena, F., Caprariis, P., Luraschi, E., Sacchi, A., Lampa, E., Berrino, L. & Donnoli, D. (1991). *Il Farm.* **46**, 1099–1110.

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* **A40**, C-410.

Enraf–Nonius (1992). *CAD-4/PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Spek, A. L. (1995*a*). *PLATON. Molecular Geometry Program.* Version of July 1994. University of Utrecht, Utrecht, The Netherlands.

Spek, A. L. (1995*b*). *PLUTON. Molecular Graphics Program.* Version of July 1994. University of Utrecht, Utrecht, The Netherlands.

Acta Cryst. (1996). **C52**, 256–259

3-(4-Bromophényl)-1-[4-(4-bromophényl)-3-butène-2-one-1-yl]-2-[3-(2,6,6-triméthyl-1-cyclohexène-1-yl)-2-propène-1-one-1-yl]-1,2,3,4,5,6,7,8-octahydro-8,8-diméthylnaphtalène

DARIA GINDEROW

Laboratoire de Minéralogie et Cristallographie, URA 09 CNRS, Université Pierre et Marie Curie, T16, 4 Place Jussieu, 75252 Paris CEDEX 05, France

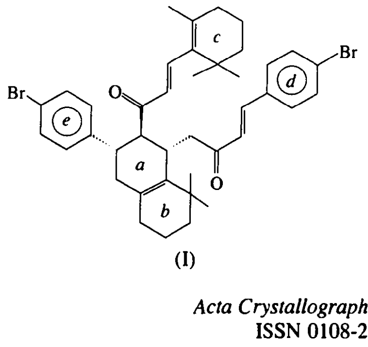
(*Reçu le* 11 *mars* 1995, *accepté le* 3 *juillet* 1995)

Abstract

The structure of a new octahydronaphthalene is reported ($C_{40}H_{46}Br_2O_2$). Disorder in the crystals arises from the coexistence of the two possible puckered conformations of the dimethylcyclohexene rings.

Commentaire

Les recherches concernant les rétinoïdes (Valla, Andriamialisoa & Giraud, 1994; Andriamialisoa, Valla, Zennache, Giraud & Potier, 1993; Giraud, Andriamialisoa, Valla, Zennache & Potier, 1994) ont amené ces auteurs à synthétiser de nouveaux analogues parmi lesquels le composé (I) dont la formule a été



(I)

ISSN 0108-2701