We thank A. Johansson MSc for preliminary synthesis of the title compound.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: AB1137). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C50, 2022–2025

# **Two Dimethylmorpholinium Bromides**

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(Received 22 November 1993; accepted 25 April 1994)

## Abstract

In the title compounds 2-hydroxy-2-(4-methoxyphenyl)-4,4,6-trimethylmorpholinium bromide,  $C_{14}H_{22}NO_3^+.Br^-$ , (1), and 2-hydroxy-4,4,6-trimethyl-2-phenylmorpholinium bromide hydrate,  $C_{13}H_{20}NO_2^+.Br^-.xH_2O$ , (2), the morpholinium rings adopt a chair conformation with the magnitudes of the endocyclic torsion angles in (1) and (2) in the ranges 50.5 (7)–60.1 (7) and 49.4 (4)–58.3 (4)°,

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© 1994 International Union of Crystallography Printed in Great Britain – all rights reserved respectively. The hydroxyl group is in the axial position of the morpholinium ring in both structures with C—OH bond distances of 1.410 (9) and 1.409 (4) Å. In (2), the hydroxy H atom points towards a Br ion  $[O \cdots Br$ 3.246 (3),  $H \cdots Br$  2.55 (3) Å,  $O - H \cdots Br$  172 (3)°]. The OH  $\cdots Br$  interaction in (1) is longer and nonlinear  $[O \cdots Br$  3.427 (5) Å,  $O - H \cdots Br$  123 (8)°].

# Comment

As part of a larger study of structural changes in reactions involving tetrahedral intermediates and substituent effects in 2-substituted 2-hvdroxy-4.4-dimethylmorpholinium bromides (Lee et al., 1992), the syntheses of some morpholinium bromides (Garcia-Guaiardo, Fronczek & Gandour, 1986: Altbach et al., 1988: Garcia, Fronczek & Gandour, 1992a, 1993a) were performed. 2-Hydroxy-2-(4-methoxyphenyl)-4,4-dimethyl-6-methylmorpholinium bromide (1) was prepared by condensing 4-methoxy-2'-bromoacetophenone with 2-hydroxy-N, Ndimethylpropanol amine following the procedure described by Garcia (1986). Similarly, 2-hydroxy-4,4dimethyl-6-methyl-2-phenylmorpholinium bromide hydrate (2) was prepared by condensing 2'-bromoacetophenone with 2-hydroxy-N, N-dimethylpropanol amine. Crystals of (1) (m.p. 443-445 K) and (2) (m.p. 480-482 K) were grown by slow cooling of methanol.



average values of the O-CH<sub>3</sub> and The C(aromatic)-O distances and the C(aromatic)-O-CH<sub>3</sub> angles in (1) [1.42 (1) and 1.372 (8) Å, 119.0 (6)°, respectively] are in agreement with those determined by X-ray crystallography by Nyburg & Faerman (1986) (1.425, 1.371 Å; 117.7°). Structural data for the title compounds are also in agreement with those for 2-(4cyanophenyl)-2-hydroxy-4,4-dimethylmorpholinium bromide (Altbach et al., 1988), 2-hydroxy-4,4-dimethyl-2-(4-tolyl)morpholinium bromide (Garcia, Fronczek & Gandour, 1992a), 2-hydroxy-2-(2-methylphenyl)-4,4dimethylmorpholinium bromide (Garcia, Fronczek &

> Acta Crystallographica Section C ISSN 0108-2701 ©1994

Gandour, 1993*a*), 2-hydroxy-6-methyl-2-(4-nitrophenyl)-4,4-dimethylmorpholinium bromide (Garcia, Fronczek & Gandour, 1993*b*), 4,4-dimethyl-2-oxomorpholinium bromide (Garcia-Guajardo, Fronczek & Gandour, 1986) and *N*-(3-hydroxypropyl)-*N*,*N*-dimethyl-*N*-[2-oxo-2-(4-phenylphenyl)ethyl]ammonium bromide (Garcia, Fronczek & Gandour, 1992*b*). The pharmacological activity of related compounds is reported by Anderson *et al.* (1966) and Lee *et al.* (1992).

The morpholinium rings in (1) and (2) adopt chair conformations with the following torsion angles:  $\omega_1(O1-C2-C1-N)$  52.6 (8), 49.4 (4);  $\omega_2(C2-C1-N-C4)$  -50.5 (7), -50.5 (4);  $\omega_3(C1-N-C4-C3)$  52.5 (7), 54.0 (4);  $\omega_4(N-C4-C3-O1)$  -58.2 (8), -58.3 (4);  $\omega_5(C4-C3-O1-C2)$  60.1 (7), 57.8 (4) and  $\omega_6(C3-O1-C2-C1)$  -56.8 (7),







-52.1 (4)°. They are distorted from the ideal  $D_{3d}$  chair conformation of cyclohexane (Hargittai & Hargittai, 1986) which has torsion angles  $\omega_1$ ,  $\omega_2$ ,  $\omega_3$ ,  $\omega_4$ ,  $\omega_5$  and  $\omega_6$  of  $\pm 54.4^\circ$  (Hendrickson, 1967).

In (2), the partially populated water, O1W, is at a distance of 3.27 (1) Å from the bromide ion and 2.85 (1) Å from the morpholinium O atom O( $x + \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$ ).

Mo  $K\alpha$  radiation

Cell parameters from 25

 $0.40 \times 0.40 \times 0.24$  mm

1159 observed reflections

 $\lambda = 0.71073 \text{ Å}$ 

reflections

 $\theta = 10 - 13^{\circ}$  $\mu = 2.7 \text{ mm}^{-1}$ 

T = 295 K

Colorless

 $[I > \sigma(I)] \\ \theta_{\max} = 25^{\circ}$ 

 $h = 0 \rightarrow 13$ 

 $k = 0 \rightarrow 18$ 

 $l = 0 \rightarrow 10$ 

3 standard reflections

frequency: 167 min

intensity variation: <2%

Prism

## Experimental Compound (1)

# Crystal data

 $C_{14}H_{22}NO_3^{+}.Br^ M_r = 332.3$ Orthorhombic  $Pna2_1$  a = 11.341 (4) Å b = 15.692 (6) Å c = 8.479 (3) Å V = 1509 (2) Å<sup>3</sup> Z = 4 $D_x = 1.462$  Mg m<sup>-3</sup>

#### Data collection

Enraf-Nonius CAD-4 diffractometer  $\omega$ -2 $\theta$  scans Absorption correction: empirical  $T_{min} = 0.738, T_{max} =$ 0.999 1604 measured reflections 1418 independent reflections

#### Refinement

Br 01 02 03 N C1

C2

C3

C4

C5

Refinement on F	$\Delta \rho_{\rm max} = 0.87 \ {\rm e} \ {\rm \AA}^{-3}$
R = 0.0464	$\Delta \rho_{\rm min} = -0.28 \ {\rm e} \ {\rm \AA}^{-3}$
wR = 0.0468	Extinction correction:
S = 2.176	$(1 + gI_c)^{-1}$ applied to $F_c$
1159 reflections	Extinction coefficient:
176 parameters	8.7 (6) $\times 10^{-8}$
$w = 4F_o^2[\sigma^2(I)]$	Atomic scattering factors
$+ (0.02F_o^2)^2]^{-1}$	from International Tables
$(\Delta/\sigma)_{\rm max} = 0.03$	for X-ray Crystallography
•	(1974, Vol. IV)

# Table 1. Fractional atomic coordinates and equivalentisotropic displacement parameters (Å<sup>2</sup>) for (1)

# $B_{\rm eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_i^* \mathbf{a}_i . \mathbf{a}_j.$

x	у	Ζ	$B_{eq}$
0.67054 (6)	0.88346 (5)	0.00000	4.16(1
0.6600 (4)	0.5983 (3)	0.2187 (6)	2.5(1)
0.7691 (4)	0.6780 (3)	0.0378 (6)	3.0(1)
0.9940 (5)	0.8035 (3)	0.6793 (6)	3.5 (1)
0.8130 (4)	0.4881 (3)	0.0355 (7)	2.6 (1)
0.8581 (6)	0.5544 (4)	0.1504 (8)	2.5 (2)
0.7730 (6)	0.6288 (4)	0.1769 (9)	2.4 (1)
0.6116 (7)	0.5414 (4)	0.1049 (9)	2.8 (2)
0.6913 (6)	0.4635 (4)	0.0847 (9)	2.8 (2)
0.8190 (7)	0.5174 (5)	-0.130(1)	3.8 (2)

C6	0.8914 (/)	0.4098	3(5) 0.052(1)	4.2 (2)	Refinemer	nt			
C7 C8	0.8227 (6)	0.6799	P(4) = 0.3151(9) R(4) = 0.4669(8)	2.2(1) 2.7(2)	Refineme	nt on F		$(\Delta/\sigma) = 0.03$	
C9	0.8372 (6)	0.7077	7 (5) 0.5932 (9)	2.9 (2)	R = 0.044	1		$\Delta a = 0.03$	3 -3
C10	0.9322 (6)	0.7625	5 (4) 0.5626 (8)	2.7 (2)	wR = 0.04	, ,,		$\Delta p_{\text{max}} = 0.45 \text{ Cr}$	<b>Å</b> −3
C11	0.9681 (6)	0.7756	5 (5) 0.4108 (8)	2.6 (2)	S = 1.389	)		$\Delta p_{\rm min} = -0.18  {\rm e}$	A
C12	0.9148 (6)	0.7355	5 (4) 0.2890 (9)	2.8 (2)	S = 1.500	) +:		Atomic scattering	g factors
	0.4909 (7)	0.5172	2(5) 0.159(1)	3.8 (2)		ections		from Internatio	onal lables
014	0.9073(7)	0.7652	(3) 0.859(1)	4.0 (2)	244 parar	neters		for X-ray Crys	tallography
					$w = 4F_o^2$	$\sigma^{-}(I)$		(1974, Vol. IV	)
Table 2	Salactor	lacomatr	ia naramatara (Å o	) for (1)	+ (0.	$(02F_o^2)^2$ ] <sup>-1</sup>			
	Tuble 2. betected geometric purumeters (A, *) jor (1)								
01—C2		1.414 (8)	C2—C7	1.53 (1)	Table 3.	Fractional	atomic	coordinates and	equivalent
01 - C3		1.425 (9)	C3-C4	1.53 (1)	isot	ropic displa	acement i	parameters (Å <sup>2</sup> ) f	for (2)
02		1.410 (9)	$C_3 - C_{13}$	1.49(1)					
03—C14		1.42(1)	$C_{7} = C_{12}$	1.37(1) 1 38(1)		$B_{eq} =$	$(8\pi^2/3)\Sigma_i$	$\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ .	
N-C1		1.514 (9)	C8—C9	1.38 (1)		x	ν	- 7	<i>B</i>
NC4		1.493 (9)	C9-C10	1.40(1)	Br	0.71862 (5)	0.67662	2 (4) 0.09612 (3)	5.18(1)
N—C5		1.48 (1)	C10-C11	1.37 (1)	01	0.7140 (3)	0.7343	(2) 0.5168 (2)	4.09 (6)
		1.522 (9)	CII—CI2	1.35 (1)	02	0.5427 (3)	0.8658	(2) 0.4807 (2)	4.45 (6)
		1.55(1)	o. <b>o</b> . o.			0.8453 (4)	0.8648	$\begin{array}{ccc} (2) & 0.3673 (2) \\ (2) & 0.4766 (2) \end{array}$	4.00(7)
$C_2 = -01 = C_3$	14	113.1 (5)	01 - C3 - C4	110.4 (6)	C2	0.6897(4)	0.8951	(3) 0.4700 (3) (3) 0.5285 (3)	3.48 (9) 3.51 (8)
C1 - N - C4	14	108 1 (5)	01 - 03 - 013 04 - 03 - 013	107.7 (6)	C3	0.7317 (5)	0.7021	$\begin{array}{c} (3) \\$	4.6(1)
C1-N-C5		112.4 (5)	N-C4-C3	111.8 (5)	C4	0.8676 (5)	0.7545	(3) 0.3667 (3)	4.5 (1)
C1NC6		107.4 (6)	C2C7C8	121.7 (6)	C5	0.9943 (5)	0.9145	(4) 0.3358 (3)	5.8 (1)
C4—N—C5		112.8 (5)	C2-C7-C12	119.2 (6)	C6	0.7126 (5)	0.8957	(3)    0.2918  (3)    0.1018  (3)	4.9 (1)
C4—N—C6		107.9 (5)	C8-C7-C12	118.8 (7)	C8	0.7010 (4)	0.8594	$\begin{array}{ccc} (3) & 0.6434 (3) \\ (3) & 0.6793 (3) \\ \end{array}$	3.27(8)
N-C1-C2		108.0 (6)	C/C8C9 C8C9C10	121.6 (6)	C9	0.6632 (5)	0.9714	(3) 0.7836(3)	5.3 (1)
01-C2-02		111.5 (5)	03-010-09	123.1 (6)	C10	0.7313 (5)	0.9052	(4) 0.8530 (3)	5.8 (1)
01—C2—C1		110.4 (5)	O3-C10-C11	117.2 (6)	C11	0.7885 (5)	0.8173	(3) 0.8184 (3)	5.4 (1)
01-C2-C7		108.7 (6)	C9-C10-C11	119.7 (6)	C12	0.7741 (5)	0.7946	(3) 0.7142 (3)	4.5 (1)
02—C2—C1		108.3 (6)	C10-C11-C12	121.1 (7)		0.7381(7)	0.3918	$\begin{array}{ccc} (3) & 0.41/0 (4) \\ (8) & 0.0874 (7) \end{array}$	7.9 (2)
02 - 02 - 07		111.5 (5)	C7—C12—C11	120.7 (7)	011	0.949 (1)	0.8703	(8) 0.0674 (7)	7.0 (3)
C1—C2—C7	~	100.5(0)	N 64 65 65		Table	Salaatad	a a a ma at mi	n manana tana (Å	a) fam ( <b>3</b> )
C201C3 C4NC1		60.1 (7) 	N = C1 = C2 = 02 02 = C2 = C7 C12	-69.8 (7)	Table 4	. Selecieu g	geometric	c parameters (A,	°) for (2)
N - C1 - C2 - C2	-01	- 50.5 (7) 52.6 (8)	$H_{2}H_{-0}^{2}$	38.3 (8)	01—C2	1	.417 (4)	C3-C4	1.502 (6)
		0 = 10 (0)		40(0)	01 - C3	1	.429 (5)	C3C13	1.511 (6)
Compound	1 (2)				N-C1	1	.409 (4) 502 (5)	$C_{7}$	1.370 (5)
Crystal dat	- (-) a				NC4	1	.504 (5)	C8—C9	1.382 (6)
Crysiai aan	u				NC5	1.	.505 (5)	C9-C10	1.369 (9)
$C_{13}H_{20}NO_{2}^{4}$	$Br^{-}xH$	$\mathbf{A}$	Ma Ka radiation		N 0/	1	EO1 (E)	C10-C11	1 369 (7)
(x = 1/3)		U	NO NO NO TAUTALION		N-00	1.	.501 (5)		1.505 (7)
		20	$\lambda = 0.71073 \text{ Å}$		N-C6 C1-C2 C2 C7	1.	.501 (5) .519 (5)	C11-C12	1.379 (5)
$M_r = 308.2$		20	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro	om 25	N-C6 C1-C2 C2-C7	1.	.501 (5) .519 (5) .511 (5)	C11—C12	1.379 (5)
$M_r = 308.2$ Monoclinic	:	20	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections	om 25	NC6 C1C2 C2C7 C2O1C	3 1	.501 (5) .519 (5) .511 (5) 15.1 (3)	C11-C12 C1-C2-C7	1.379 (5) 108.9 (3)
$M_r = 308.2$ Monoclinic $P2_1/n$		0	$\lambda = 0.71073$ Å Cell parameters fro reflections $\theta = 1-25^{\circ}$	m 25	NC6 C1C2 C2C7 C2O1C C1NC4 C1NC5	3 1. 10 3 1.	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3)	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13	1.379 (5) 108.9 (3) 110.7 (3) 106 8 (3)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 (	3) Å	0	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$	om 25	N-C6 C1-C2 C2-C7 C2-O1-C C1-N-C4 C1-N-C5 C1-N-C6	1 1 1 3 1 1 1 1 1 1	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3)	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535	3) Å (2) Å	0	ho Ka radiation $\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K	m 25	NC6 C1C2 C2C7 C201C C1NC4 C1NC5 C1NC6 C4NC5	3 1: 10 3 1: 10 10 11 10	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3) 09.6 (3)	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915	3) Å (2) Å (2) Å	0	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle	m 25	NC6 C1C2 C2C7 C201C C1NC4 C1NC5 C1NC6 C4NC5 C4NC6	3 1. 1. 3 1. 10 10 11 10	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3) 09.6 (3) 11.2 (3)	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C8	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ (	3) Å (2) Å (2) Å (2) Å	0	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$	om 25	N-C6 C1-C2 C2-C7 C2-01-C C1-N-C4 C1-N-C5 C1-N-C6 C4-N-C5 C4-N-C5 C4-N-C6 N-C1-C2	3 1: 1, 3 1: 10 10 11 10 11 10 11 10 11 10	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3)	$\begin{array}{c} C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3-C13\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C7-C12\\ \end{array}$	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 118.3 (3)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup>		$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.06$ Colorless	m 25 6 mm	N-C6 C1-C2 C2-C7 C2-01-CC C1-N-C4 C1-N-C5 C1-N-C6 C4-N-C5 C4-N-C6 C5-N-C6 N-C1-C2 O1-C2-O	3 1 1 3 1 1 1 1 1 1 1 1 1 1 1 1 2 1 1	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 15.4 (3)	$\begin{array}{c} C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C7-C8\\ C7-C9\\ C8-C9\\ \end{array}$	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 112.8 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 7 = 4	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup>		$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.06$ Colorless	m 25 6 mm	N-C6 C1-C2 C2-C7 C2-01-C C1-N-C4 C1-N-C5 C1-N-C5 C4-N-C5 C4-N-C5 C4-N-C6 C5-N-C6 N-C1-C2 O1-C2-C O1-C2-C	3 1 10 10 10 10 10 10 10 10 10 10 10 10 10	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 10.7 (3) 10.4 (3)	$\begin{array}{c} C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C9\\ C8-C9\\ C8-C9-C10\\ \end{array}$	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 112.8 (4) 120.1 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_r = 1.388$	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Ma m <sup>-3</sup>		$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.06$ Colorless	m 25 6 mm	N-C6 C1-C2 C2-C7 C2-C7 C1-N-C4 C1-N-C5 C1-N-C5 C4-N-C5 C4-N-C5 C4-N-C6 N-C1-C2 O1-C2-C0 O1-C2-C O1-C2-C	3 1 10 10 10 10 10 11 11 12 11 11 11 11 11 11 11 11 11 11	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 08.3 (3) 12.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 10.7 (3) 10.4 (3) 07.4 (3) 07.4 (3)	$\begin{array}{c} C11-C12\\ C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C7-C12\\ C8-C9\\ C8-C9-C10\\ C9-C10-C11\\ C9-C10-C10\\ C9-C10\\ C9-C10\\ C9-C10-C10\\ C9-C10\\ C9-C1$	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 120.8 (4) 120.1 (4) 119.8 (4) 119.8 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup>		$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless	m 25 6 mm	N-C6 C1-C2 C2-C7 C2-01-CC C1-N-C4 C1-N-C5 C1-N-C6 C4-N-C5 C4-N-C5 C4-N-C6 C5-N-C6 N-C1-C2 01-C2-C 01-C2-C 01-C2-C 02-C2-C	2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	.501 (5) .519 (5) .511 (5) 15.1 (3) 07.6 (3) 07.8 (3) 07.8 (3) 11.2 (3) 07.8 (3) 15.4 (3) 10.7 (3) 10.4 (3) 07.4 (3) 07.1 (3) 12.4 (3)	$\begin{array}{c} C11-C12\\ C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C9\\ C8-C9-C10\\ C9-C10-C11\\ C10-C11-C12\\ C12-C12\\ C7-C12\\ C7-C12\\ C12-C12\\ C12-C12\\$	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 118.3 (3) 120.8 (4) 120.1 (4) 119.8 (4) 120.0 (4) 120.0 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup>		$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless	m 25 6 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-01-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C5\\ C4-N-C6\\ C5-N-C6\\ N-C1-C2\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 02-C2-C\\ 02-$	2 12 1	.501 (5)           .519 (5)           .511 (5)           15.1 (3)           07.6 (3)           08.3 (3)           12.4 (3)           09.6 (3)           11.2 (3)           07.8 (3)           15.4 (3)           10.7 (3)           10.4 (3)           07.4 (3)           07.1 (3)           12.4 (3)	$\begin{array}{c} C11-C12\\ C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C7-C12\\ C7-C8-C9\\ C8-C9-C10\\ C9-C10-C11\\ C10-C11-C12\\ C7-C12-C11\\ C10-C12-C12\\ C7-C12-C11\\ C10-C12-C12\\ C7-C12-C11\\ C10-C12-C12\\ C7-C12-C11\\ C10-C12-C12\\ C7-C12-C11\\ C10-C12-C12\\ C7-C12-C12\\ C10-C12-C12\\ C7-C12-C12\\ C10-C12-C12\\ C7-C12-C12\\ C10-C12-C12\\ C7-C12-C12\\ C10-C12-C12\\ C10-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C10-C12-C12\\ C$	1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 118.3 (3) 120.8 (4) 120.1 (4) 119.8 (4) 120.0 (4) 120.9 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion		$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless	m 25 6 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-01-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C5\\ C5-N-C6\\ N-C1-C2\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 02-C2-C\\ 02-C2-C\\ 02-C2-C\\ C2-01-C\\ C2-$	2 1 1 1 1 1 1 1 1 1 1 1 1 1	$\begin{array}{c} .501 (5) \\ .519 (5) \\ .511 (5) \\ 15.1 (3) \\ 07.6 (3) \\ 07.6 (3) \\ 07.6 (3) \\ 07.8 (3) \\ 09.6 (3) \\ 11.2 (3) \\ 07.8 (3) \\ 07.8 (3) \\ 10.7 (3) \\ 10.7 (3) \\ 10.7 (3) \\ 10.7 (3) \\ 10.4 (3) \\ 07.1 (3) \\ 12.4 (3) \\ 57.8 (4) \\ 59.5 (4) \end{array}$	$\begin{array}{c} C11-C12\\ C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C7-C12\\ C7-C8-C9\\ C8-C9-C10\\ C9-C10-C11\\ C10-C11-C12\\ C7-C12-C11\\ N-C1-C2-O2\\ C9-C7\\ C8-C9\\ C9-C7\\ C8-C9\\ C1-C8\\ C1-C8\\ C2-C7\\ C12-C1\\ C1-C2-C1\\ C1-C2-C2\\ C1-C8\\ C1-C$	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 118.3 (3) 120.8 (4) 120.1 (4) 120.0 (4) 120.9 (4) -71.2 (4)
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonit	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD-	4	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$	m 25 6 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-01-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C5\\ C4-N-C6\\ C5-N-C6\\ N-C1-C2\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 02-C2-C\\ 02-C2-C\\ C2-01-C\\ C2-01-C\\ C2-01-C\\ C2-01-C\\ C1-C2\\ C2-01-C\\ C1-C2\\ $	2 11 7 10 11 12 14 16 17 16 17 16 1 17 16 1 16 17 16 17 16 17 16 17 16 17 16 17 16 16 16 16 16 16 16 16 16 16	.501 (5) .519 (5) .511 (5) 15.11 (3) 07.6 (3) 07.6 (3) 07.6 (3) 112.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 07.4 (3) 07.4 (3) 07.1 (3) 12.4 (3) 57.8 (4) -50.5 (4) 49.4 (4)	$\begin{array}{c} C11-C12\\ C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3\\ C2-C7-C8\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C7-C8-C9\\ C8-C9-C10\\ C9-C10-C11\\ C10-C11-C12\\ C7-C12-C11\\ N-C1-C2-O2\\ 02-C2-C7-C8\\ H20H-O2-C2\\ O2-C2-C7-C8\\ H20H-O2-C2\\ O2-C2-C7-C8\\ H20H-O2-C2\\ O2-C2-C7\\ C8\\ C8\\ C9-C10\\ C1-C2\\ C7\\ C8\\ C1-C2\\ C7\\ C1-C2\\ C1-C2\\ C7\\ C7\\ C8\\ C1-C2\\ C7\\ C1-C2\\ C7\\ C8\\ C1-C2\\ C1-C2$	$\begin{array}{c} 1.359 \ (1)\\ 1.379 \ (5)\\ \hline \\ 108.9 \ (3)\\ 110.7 \ (3)\\ 106.8 \ (3)\\ 111.5 \ (4)\\ 111.5 \ (3)\\ 120.0 \ (3)\\ 121.6 \ (3)\\ 120.8 \ (4)\\ 120.1 \ (4)\\ 120.8 \ (4)\\ 120.0 \ (4)\\ 120.9 \ (4)\\ -71.2 \ (4)\\ -71.2 \ (4)\\ -71.2 \ (5)\\ 76 \ (2)\\ \end{array}$
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffracton	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter	4	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$	m 25 6 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-01-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C6\\ C5-N-C6\\ N-C1-C2\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 02-C2-C\\ 02-C2-C\\ C2-01-C\\ C2-01-C\\ C4-N-C1\\ N-C1-C2\\ \end{array}$	2 1 1 1 1 1 1 1 1 1 1 1 1 1	$\begin{array}{c} .501 (5) \\ .519 (5) \\ .511 (5) \\ 15.1 (3) \\ 07.6 (3) \\ 08.3 (3) \\ 12.4 (3) \\ 09.6 (3) \\ 11.2 (3) \\ 09.6 (3) \\ 11.2 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.4 (3) \\ 07.1 (3) \\ 12.4 (3) \\ 57.8 (4) \\ -50.5 (4) \\ 49.4 (4) \end{array}$	$\begin{array}{c} C11-C12\\ C11-C12\\ C1-C2-C7\\ 01-C3-C4\\ 01-C3-C13\\ C4-C3-C13\\ C4-C3-C13\\ C2-C7-C8\\ C2-C7-C8\\ C2-C7-C12\\ C8-C7-C12\\ C8-C9-C10\\ C9-C10-C11\\ C10-C11-C12\\ C7-C12-C11\\ C10-C11-C12\\ C7-C12-C11\\ N-C1-C2-O2\\ 02-C2-C7-C8\\ H2OH-O2-C2-O1\\ \end{array}$	$\begin{array}{c} 1.379 (5) \\ 1.379 (5) \\ 108.9 (3) \\ 110.7 (3) \\ 106.8 (3) \\ 111.5 (4) \\ 111.5 (3) \\ 120.0 (3) \\ 121.6 (3) \\ 120.8 (4) \\ 120.1 (4) \\ 120.9 (4) \\ 120.9 (4) \\ -71.2 (4) \\ -42.9 (5) \\ 1 \\ 76 (2) \end{array}$
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega$ -2 $\theta$ scans	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter	4	$\lambda = 0.71073 \text{ Å}$ Cell parameters from reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.000$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$	m 25 6 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-01-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C6\\ C5-N-C6\\ N-C1-C2\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 01-C2-C\\ 02-C2-C\\ 02-C2-C\\ C2-01-C\\ C2-01-C\\ C2-01-C\\ C2-01-C\\ C4-N-C1\\ N-C1-C2\\ R_{int} was 1\\ \end{array}$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{c} .501 (5) \\ .519 (5) \\ .511 (5) \\ 15.11 (3) \\ 07.6 (3) \\ 08.3 (3) \\ 12.4 (3) \\ 09.6 (3) \\ 11.2 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.4 (3) \\ 07.4 (3) \\ 07.1 (3) \\ 12.4 (3) \\ 57.8 (4) \\ -50.5 (4) \\ 49.4 (4) \\ \end{array}$	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C8 C2-C7-C12 C8-C7-C12 C7-C8-C9 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C12-C11 N-C1-C2-O2 O2-C2-C7-C8 H2OH-O2-C2-O1	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 118.3 (3) 120.8 (4) 120.1 (4) 120.9 (4) -71.2 (4) -42.9 (5) 1 76 (2) 0 redundant
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega$ -2 $\theta$ scans Absorption	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter correction	4 1:	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$ $k = 0 \rightarrow 16$	m 25 5 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-O1-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C6\\ C5-N-C6\\ N-C1-C2\\ O1-C2-C\\ O1-C2-C\\ O1-C2-C\\ O1-C2-C\\ O2-C2-C\\ O2-C2-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C4-N-C1\\ N-C1-C2\\ R_{int} was idata. For \\ \end{array}$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{c} .501 (5) \\ .519 (5) \\ .511 (5) \\ 15.11 (3) \\ 07.6 (3) \\ 08.3 (3) \\ 12.4 (3) \\ 09.6 (3) \\ 11.2 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.4 (3) \\ 07.4 (3) \\ 07.1 (3) \\ 12.4 (3) \\ 07.1 (3) \\ 12.4 (3) \\ 57.8 (4) \\ -50.5 (4) \\ 49.4 (4) \\ ed for (1) \\ roxyl H a \end{array}$	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C12 C8-C7-C12 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C8-C9 C2-C7-C8 H2OH-O2-C2-O1 O2-C2-C7-C8 H2OH-O2-C2-O1 O2-C2-C7-C8 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-C7-C12 O2-C2-C7-C8 C2-O2 O2-C2-C7-C8 C2-O2 O2-C2-C7-C8 C2-O2 O2-C2-C7-C8 C2-O2 O2-C2-C7-C8 C2-O2 O2-C2-C7-C8 C3-C12 O2-C2-C12 O2-C12-C12 O2-C12-C12 O2-C12-C12 O2-C12-C12 O2-C12-C12-C12 O2-C12-C12-C12 O2-C12-C12-C12-C12 O2-C12-C12-C12-C12-C12-C12-C12-C12-C12-C1	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 120.8 (4) 120.1 (4) 120.9 (4) -71.2 (4) -42.9 (5) 76 (2) o redundant sotropically.
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega$ -2 $\theta$ scans Absorption empirical	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter correction	4 1:	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$ $k = 0 \rightarrow 16$ $l = -15 \rightarrow 15$	m 25 6 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-O1-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C4-N-C5\\ C5-N-C6\\ N-C1-C2\\ O1-C2-C\\ O1-C2-C\\ O1-C2-C\\ O2-C2-C\\ O2-C2-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C2-O1-C\\ C4-N-C1-C\\ N-C1-C2\\ R_{int} was idata. For All other I$	1 1 1 1 1 1 1 1	.501 (5) .519 (5) .511 (5) 15.11 (3) 07.6 (3) 07.6 (3) 11.2 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 07.8 (3) 15.4 (3) 07.4 (3) 07.4 (3) 07.1 (3) 12.4 (3) 07.1 (3) 12.4 (3) 57.8 (4) -50.5 (4) 49.4 (4) ed for (1) kroxyl H a re placed i	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C12 C8-C7-C12 C7-C8-C9 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C12-C11 N-C1-C2-O2 O2-C2-C7-C8 H2OH-O2-C2-O1 O as there were n atom was refined in n calculated position	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 121.6 (3) 120.8 (4) 120.1 (4) 120.9 (4) -71.2 (4) -42.9 (5) 1 76 (2) o redundant sotropically. ons with $B =$
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38 ($ V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega - 2\theta$ scans Absorption empirical $T_{min} = 0.9$	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter correction 914, $T_{max}$	4 n:	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$ $k = 0 \rightarrow 16$ $l = -15 \rightarrow 15$ 3 standard reflection	m 25 5 mm	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-O1-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C5-N-C6\\ C5-N-C6\\ N-C1-C2\\ O1-C2-C\\ O1-C2-C\\ O1-C2-C\\ O2-C2-C\\ O2-C2-C\\ C2-O1-C\\ C2-C\\ C2-O1-C\\ C2-C\\ C2-O1-C\\ C2-C\\ C2-C\\ C2-C\\ C4-N-C1\\ N-C1-C2\\ R_{int} was idata. For \\ All other I\\ 1.3 \times B_{en} \end{array}$	$1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\$	$\begin{array}{c} .501 (5) \\ .519 (5) \\ .511 (5) \\ 15.11 (3) \\ 07.6 (3) \\ 08.3 (3) \\ 12.4 (3) \\ 09.6 (3) \\ 11.2 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 07.8 (3) \\ 15.4 (3) \\ 15.4 (3) \\ 07.4 (3) \\ 07.4 (3) \\ 07.4 (3) \\ 07.1 (3) \\ 12.4 (3) \\ 07.4 (3) \\ 57.8 (4) \\ -50.5 (4) \\ 49.4 (4) \\ ed for (1) \\ roxyl Ha \\ re placed i \\ ed C atom \\ \end{array}$	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C8 C2-C7-C12 C7-C8-C9 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C12-C11 N-C1-C2-O2 O2-C2-C7-C8 H2OH-O2-C2-O2 O3 as there were n tom was refined in n calculated position Refinement of th	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 120.8 (4) 120.1 (4) 120.9 (4) -71.2 (4) -42.9 (5) 1 76 (2) o redundant sotropically. ons with $B =$ e inversion-
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega$ -2 $\theta$ scans Absorption empirical $T_{min} = 0.9$ 0.994	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter correction 914, $T_{max}$	4 1: 4	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$ $k = 0 \rightarrow 16$ $l = -15 \rightarrow 15$ 3 standard reflection frequency: 177 m	m 25 6 mm 18	N-C6 C1-C2 C2-C7 C2-O1-C C1-N-C4 C1-N-C5 C1-N-C6 C4-N-C5 C4-N-C6 C5-N-C6 C5-N-C6 N-C1-C2 O1-C2-C O1-C2-C O2-C2-C O2-C2-C C2-O1-C2 C2-C2 C2-C1-C2 C2-C2 C2-C2-C2 C2-C2 C2-C2-C2-C2 C2-C2-C2-C2-C2 C2-C2-C2-C2-C2-C2-C2-C2-C2-C2-C2-C2-C2-C	$1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\$	$\begin{array}{l} \text{.501 (5)} \\ \text{.519 (5)} \\ \text{.511 (5)} \\ \text{15.11 (3)} \\ \text{07.6 (3)} \\ \text{08.3 (3)} \\ \text{12.4 (3)} \\ \text{09.6 (3)} \\ \text{11.2 (3)} \\ \text{07.8 (3)} \\ \text{15.4 (3)} \\ \text{07.8 (3)} \\ \text{15.4 (3)} \\ \text{07.7 (3)} \\ \text{10.4 (3)} \\ \text{07.4 (3)} \\ \text{07.4 (3)} \\ \text{07.1 (3)} \\ \text{12.4 (3)} \\ \text{57.8 (4)} \\ \text{-50.5 (4)} \\ \text{49.4 (4)} \\ \text{ed for (1)} \\ \text{re placed i} \\ \text{re placed i} \\ \text{re placed i} \\ \text{red C atom} \\ \text{led } R = 0 \end{array}$	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C4 O1-C3-C13 N-C4-C3 C2-C7-C8 C2-C7-C12 C7-C8-C9 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C2-C11 N-C1-C2-O2 O2-C2-C7-C8 H2OH-O2-C2-O2 O3 as there were n n calculated positio . Refinement of th 0.0465, $wR = 0.04$	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 120.8 (4) 120.9 (4) 120.9 (4) -71.2 (4) -42.9 (5) 76 (2) o redundant sotropically. ons with $B =$ e inversion- 71 and $S =$
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega$ -2 $\theta$ scans Absorption empirical $T_{min} = 0.9$ 0.994 2889 measu	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter correction 914, $T_{max}$ red reflec	4 1: 4 = 1: 1:	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$ $k = 0 \rightarrow 16$ $l = -15 \rightarrow 15$ 3 standard reflection frequency: 177 m intensity variation	m 25 5 mm 5 mm in 1: -3.4%	N-C6 C1-C2 C2-C7 C2-01-C C1-N-C4 C1-N-C5 C1-N-C6 C4-N-C5 C4-N-C6 C5-N-C6 N-C1-C2 O1-C2-C O1-C2-C O2-C2-C O2-C2-C C2-O1-C C2-O1-C C2-O1-C C2-O1-C C2-O1-C C2-O1-C R <sub>int</sub> was i data. For All other J 1.3 × $B_{eq}$ related str 2.187.	$1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\$	5.01 (5) 5.19 (5) 5.11 (5) 15.11 (3) 07.6 (3) 07.6 (3) 12.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 07.4 (3) 07.4 (3) 07.4 (3) 07.4 (3) 07.1 (3) 12.4 (3) 57.8 (4) -50.5 (4) 49.4 (4) ed for (1) broxyl H a re placed i ed C atom led $R = 0$	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C8 C2-C7-C12 C7-C8-C9 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C2-C1 N-C1-C2-O2 O2-C2-C7-C8 H2OH-O2-C2-O3 A sthere were n tom was refined in n calculated positio . Refinement of th .0465, $wR = 0.04$	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 120.8 (4) 120.9 (4) 120.9 (4) -71.2 (4) -71.2 (4) -72.9 (5) 76 (2) o redundant sotropically. ons with $B =$ e inversion- 71 and $S =$
$M_r = 308.2$ Monoclinic $P2_1/n$ a = 8.451 ( b = 13.535 c = 12.915 $\beta = 93.38$ ( V = 1474.8 Z = 4 $D_x = 1.388$ Data collect Enraf-Nonin diffractom $\omega$ -2 $\theta$ scans Absorption empirical $T_{min} = 0.9$ 0.994 2889 measu 2583 independent	3) Å (2) Å (2) Å (2)° (9) Å <sup>3</sup> Mg m <sup>-3</sup> tion us CAD- neter correction 914, $T_{max}$ red reflect endent ref	4 $a = $ $b = 1$ $b = 1$ $b = 1$ $b = 1$	$\lambda = 0.71073 \text{ Å}$ Cell parameters fro reflections $\theta = 1-25^{\circ}$ $\mu = 2.8 \text{ mm}^{-1}$ T = 298  K Needle $0.38 \times 0.10 \times 0.00$ Colorless $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^{\circ}$ $h = 0 \rightarrow 10$ $k = 0 \rightarrow 16$ $l = -15 \rightarrow 15$ 3 standard reflection frequency: 177 m intensity variation (linear correction)	m 25 5 mm 5 mm in a: -3.4% on)	$\begin{array}{c} N-C6\\ C1-C2\\ C2-C7\\ C2-O1-C\\ C1-N-C4\\ C1-N-C5\\ C1-N-C6\\ C4-N-C5\\ C5-N-C6\\ C5-N-C6\\ N-C1-C2\\ O1-C2-C\\ O1-C2-C\\ O1-C2-C\\ O2-C2-C\\ O2-C2-C\\ C2-O1-C\\ C2-O1-C\\ C2-C\\ C2-O1-C\\ C2-C\\ C2-O1-C\\ C2-C\\ C2-C\\ C2-C\\ C2-C\\ C2-C\\ C3-C2-C\\ C4-N-C1\\ N-C1-C2\\ C2\\ C2-C\\ C4-N-C1\\ N-C1-C2\\ C2-C\\ C2-$	$1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\$	5.01 (5) 5.19 (5) 5.11 (5) 15.11 (3) 07.6 (3) 07.6 (3) 12.4 (3) 09.6 (3) 11.2 (3) 07.8 (3) 15.4 (3) 07.4 (3) 07.4 (3) 07.4 (3) 07.1 (3) 12.4 (3) 07.1 (3) 12.4 (3) 57.8 (4) -50.5 (4) 49.4 (4) ed for (1) broxyl H a re placed i ed C atom led $R = 0$ ms were re	C11-C12 C1-C2-C7 O1-C3-C4 O1-C3-C13 C4-C3-C13 N-C4-C3 C2-C7-C12 C7-C12 C8-C7-C12 C7-C8-C9 C8-C9-C10 C9-C10-C11 C10-C11-C12 C7-C12-C11 N-C1-C2-O2 O2-C2-C7-C8 H2OH-O2-C2-O3 A sthere were n ttom was refined in n calculated positio A Refinement of th 0.0465, $wR = 0.04$	1.379 (5) 1.379 (5) 108.9 (3) 110.7 (3) 106.8 (3) 111.5 (4) 111.5 (3) 120.0 (3) 121.6 (3) 120.8 (4) 120.9 (4) 120.9 (4) -71.2 (4) -71.2 (4) -76 (2) o redundant sotropically. ons with $B =$ e inversion- 71 and $S =$

 $[l > \sigma(l)]$ 

water molecule which were not located. Refinement of the water molecule as fully occupied led to  $B = 40 \text{ Å}^2$ . Refinement

of the occupation factor for O1W led to x = 0.334 (5). The values quoted for the density *etc.* are for x = 1/3.

Programs used included *MolEN* (Fair, 1990) and *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, Hatom coordinates and torsion angles have been deposited with the IUCr (Reference: CR1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C50, 2025–2027

# 2,2-Dimethyl-5-(dimethylamino)indan-1,3-dione

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(Received 22 November 1993; accepted 16 May 1994)

### Abstract

The title compound,  $C_{13}H_{15}NO_2$ , lies on a crystallographic mirror plane. The indan-1,3-dione and the dimethyl-

amino systems are therefore coplanar. The C—C—C angle formed by the *gem*-dimethyl groups is 110.3 (1)° and the C—C—C—O torsion angles which they form with the carbonyl O atoms have magnitudes of 61.6 (1) and 61.3 (1)°. The C==O distances are 1.217 (2) and 1.210 (2) Å. The *N*-methyl groups lying on the crystallographic mirror plane have their atoms disordered into half-populated positions. The N—C(methyl) distances are 1.432 (2) and 1.447 (2) Å.

# Comment

As part of an ongoing study on nucleophilic aromatic substitution reactions with the fluoride ion, a number of substituted indan-1,3-diones were required (Enas, Garcia, Mathis & Gerdes, 1993). The crystal structures of a number of substituted indan-1,3-diones have been studied over the last 20 years on account of the well known anticoagulant activity of the parent compound in vitamin K-dependent biosynthesis (Ernster, Lind & Rase, 1972; Bravic, Gaultier & Hauw, 1974; Csöregh & Eckstein, 1979). The title compound, (I), was prepared in four steps from the known 2,2-dimethyl-1-indanone (Ohkata, Akiyama, Wada, Shun, Toda & Hanafusa, 1984), affording yellow needles when recrystallized by slow cooling and evaporation of ethanol.



The geometry about the N atom is perfectly planar, with angle magnitudes for C11—N—C5, C5—N—C12 and C11—N—C12 of 121.5 (1), 121.4 (1) and 117.1 (1)°, respectively. The gem-dimethyl groups are almost perfectly gauche to the carbonyl O atoms with magnitudes for C10—C9—C1—O1 and C10—C9—C8—O2 of 61.6 (1) and -61.3 (1)°, respectively, deviating only slightly from the perfect gauche conformation value of 60° (Klyne & Prelog, 1960).

Structural data for 2-ethyl-2-nitroindan-1,3-dione (Garcia, Enas, Chang & Fronczek, 1993), 5-amino-2,2dimethylindan-1.3-dione and 2.2-dimethyl-5-nitroindan-1,3-dione (Garcia, Enas & Fronczek, 1993), 2-nitroindan-1,3-dione dihydrate (Selenius & Lundgren, 1980), and 2-(2-nitrobenzylidene)indan-1,3-dione (Varghese, Srinivasan, Ramadas & Padmanabhan, 1986) are in agreement with those of the title compound. Examples of planar dimethylamino-aromatic groups in agreement with that of the title compound are trans-1-(2-chloro-4dimethylaminophenyl)-2-nitroethylene (Cameron. Cowley & Thompson, 1974), N, N-dimethyl-2,4-dinitro-3-toluidine (Maurin & Krygowski, 1987) and 9-(4dimethylamino-2-methylphenyl)-10-methylacridinium chloride dihydrate (Reiss, Goubitz & Heijdenrijk, 1989).