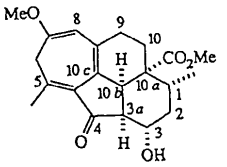
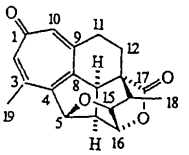
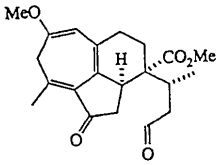
Related literature. The title compound (1) is a possible precursor to the natural product Harringtonolide (2) (Buta, Flippen & Lusby, 1978). (1) is formed *via* an intramolecular aldol reaction of the keto aldehyde (3), giving (1) as a 6:1 mixture with the alternative epimer (4) (Rogers, 1990).



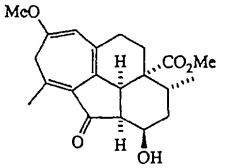
(1)



(2)



(3)



(4)

Table 3. *Selected contact distances* (Å) *for methyl* (1α,3α,3aα,10aα,10bα)-1,2,3,3a,6,9,10,10bα-*octahydro-3-hydroxy-7-methoxy-1,5-dimethyl-4-oxocyclohept*[*bc*]*acenaphthalene-10a*(4*H*)*-carboxylate* (1)

H(16)···O(4)*	2·03 (4)	H(26)···O(6)†	2·15 (4)
O(2)···O(4)*	2·890 (3)	O(7)···O(6)†	3·005 (3)
H(16)···O(4)	2·52 (3)	H(26)···O(6)	2·45 (3)
O(2)···O(4)	3·091 (2)	O(7)···O(6)	3·089 (2)
H(11*b*)···H(8)	2·26 (4)	H(11*c*)···H(8)	2·37 (4)
H(21*b*)···H(8′)	2·40 (4)	H(21*c*)···H(8′)	2·35 (5)

* Generated from the coordinate list by the operation ($-x$, $2-y$, $-z$).

† Generated from the coordinate list by the operation ($2-x$, $-y$, $-z$).

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Acta Cryst. (1991). C**47**, 2256–2258

Two Polymorphs of 5-Carbamoyl-4-methyl-6-phenyl-1,2,3-triazine

BY KENTARO YAMAGUCHI, TAKASHI ITOH, MAMIKO OKADA, AKIO OHSAWA AND GO MATSUMURA

School of Pharmaceutical Sciences, Showa University 1-5-8, *Hatanodai, Shinagawa-ku, Toyko* 142, *Japan*

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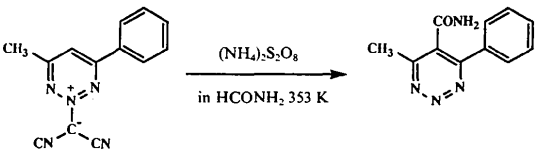
Abstract. $C_{11}H_{10}N_4O$, $M_r = 214{\cdot}23$, crystallizes in two different forms: (I) monoclinic, $P2_1/n$, $a = 14{\cdot}815$ (1), $b = 9{\cdot}477$ (1), $c = 7{\cdot}869$ (3) Å, $\beta = 98{\cdot}85$ (1)°, $V = 1091{\cdot}7$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}303$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·54050 Å, $\mu = 0{\cdot}692$ mm^{-1}, $F(000) = 448$, $T = 295$ K, final $R = 0{\cdot}048$ for 1512 reflections; (II) orthorhombic, $P2_12_12_1$, $a = 9{\cdot}299$ (1), $b = 14{\cdot}874$ (1), $c = 7{\cdot}557$ (4) Å, $V = 1045{\cdot}2$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}361$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·54050 Å, $\mu = 0{\cdot}723$ mm^{-1}, $F(000) = 448$, $T = 295$ K, final $R = 0{\cdot}050$ for 760 reflections. Bond distances and angles are quite similar in the two structures.

Experimental. The title compound was prepared by the radical substitution reaction of 1,2,3-triazinium dicyanomethylide (III) with ammonium persulfate and formamide at 353 K (Minisci, Fontana & Vismara, 1990). The crystals, colorless prism (0·45 × 0·25 × 0·50 mm) (I) and clear needle (0·10 × 0·03 × 0·50 mm) (II) were recrystallized from methanol. Details of data collection and refinement are listed in Table 1. Intensity data were collected with a Rigaku AFC-5 four-circle diffractometer used in the ω–2θ scan mode, ω scan width $(1{\cdot}3 + 0{\cdot}41\tan\theta)°$ and scan



(III) (I), (II)

speed 16° min^{-1}. Intensity variation was less than 3% for both crystals. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using program package *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985) version of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson,

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Table 1. *Details of data collection and structure refinement*

	(I)	(II)
Data collection		
Cell-parameters determination No., θ range ( )	23, 56–60	25, 54–60
Max $(\sin\theta)/\lambda$ (Å^{-1})	0·56	0·56
Range h	−16 to 16	0 to 10
k	0 to 10	0 to 16
l	0 to 8	0 to 8
Standard reflexions No., interval (ref.)	3, 150	3, 150
No. of reflexions	1910	963
independent	1617	930
$F > 3\sigma(F)$	1512	760
Merging R for equivalent reflexions	0·01 141	–
Refinement		
R	0·048	0·050
wR	0·046	0·048
G*	1·718	1·107
$(\Delta/\sigma)_{max}$	0·11	0·08
$\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	0·23/−0·29	0·21/−0·23

$$*G = \sum w\{[|(|F_o|)^2 - (|F_c|)^2|]^2/(N_r - N_v)\}^{1/2}.$$

Table 2. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
(I)				
N(1)	0·1876 (1)	−0·0873 (2)	1·1726 (2)	3·43 (7)
N(2)	0·2578 (1)	−0·0371 (2)	1·2777 (2)	3·77 (7)
N(3)	0·3197 (1)	0·0458 (2)	1·2253 (2)	3·63 (7)
C(4)	0·3142 (2)	0·0756 (2)	1·0569 (3)	3·18 (8)
C(5)	0·2429 (1)	0·0239 (2)	0·9395 (3)	2·66 (7)
C(6)	0·1772 (1)	−0·0556 (2)	1·0035 (3)	2·79 (7)
C(7)	0·3901 (2)	0·1654 (5)	1·0244 (5)	5·04 (12)
C(8)	0·2408 (1)	0·0550 (2)	0·7504 (3)	2·91 (7)
O(9)	0·2193 (1)	0·1724 (1)	0·6921 (2)	4·01 (6)
N(10)	0·2651 (1)	−0·0513 (2)	0·6592 (3)	3·68 (7)
C(11)	0·0927 (1)	−0·1122 (3)	0·9030 (3)	2·99 (7)
C(12)	0·0430 (2)	−0·0354 (3)	0·7701 (3)	3·38 (8)
C(13)	−0·0374 (2)	−0·0882 (3)	0·6813 (4)	3·87 (9)
C(14)	−0·0696 (2)	−0·2174 (4)	0·7234 (4)	4·50 (10)
C(15)	−0·0210 (2)	−0·2943 (4)	0·8571 (4)	4·82 (11)
C(16)	0·0592 (2)	−0·2425 (3)	0·9463 (4)	3·93 (9)
(II)				
N(1)	0·7690 (4)	−0·1047 (2)	0·1307 (5)	3·79 (11)
N(2)	0·6933 (5)	−0·1793 (2)	0·1297 (7)	4·50 (14)
N(3)	0·5526 (5)	−0·1787 (2)	0·1581 (6)	4·45 (13)
C(4)	0·4845 (5)	−0·1003 (3)	0·1866 (7)	3·69 (14)
C(5)	0·5593 (5)	−0·0195 (3)	0·1900 (6)	2·92 (12)
C(6)	0·7072 (5)	−0·0245 (2)	0·1632 (6)	2·97 (13)
C(7)	0·3254 (7)	−0·1071 (5)	0·2137 (11)	5·56 (20)
C(8)	0·4789 (5)	0·0680 (3)	0·2068 (7)	3·32 (14)
O(9)	0·4034 (3)	0·0916 (2)	0·0795 (5)	4·59 (10)
N(10)	0·4908 (5)	0·1150 (2)	0·3546 (6)	3·92 (12)
C(11)	0·8086 (5)	0·0515 (3)	0·1615 (6)	2·94 (12)
C(12)	0·7719 (6)	0·1343 (3)	0·0882 (7)	3·66 (14)
C(13)	0·8728 (8)	0·2027 (4)	0·0859 (8)	4·90 (18)
C(14)	1·0058 (8)	0·1903 (4)	0·1597 (9)	5·69 (21)
C(15)	1·0435 (6)	0·1090 (4)	0·2289 (8)	5·16 (19)
C(16)	0·9465 (5)	0·0385 (3)	0·2297 (7)	3·97 (16)

1980). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\sum w[|(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}02(F_c)]$, $\sigma(F_o)$ was determined from counting statistics. All H atoms found on a difference map and located by calculations were refined, initial thermal parameters set at equivalent isotropic thermal

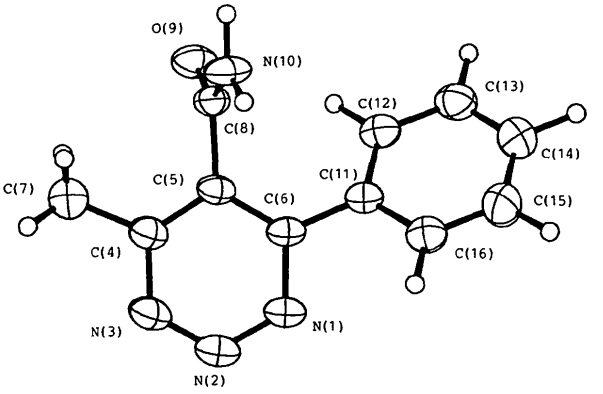

Fig. 1. *ORTEP* drawing of form (I). Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

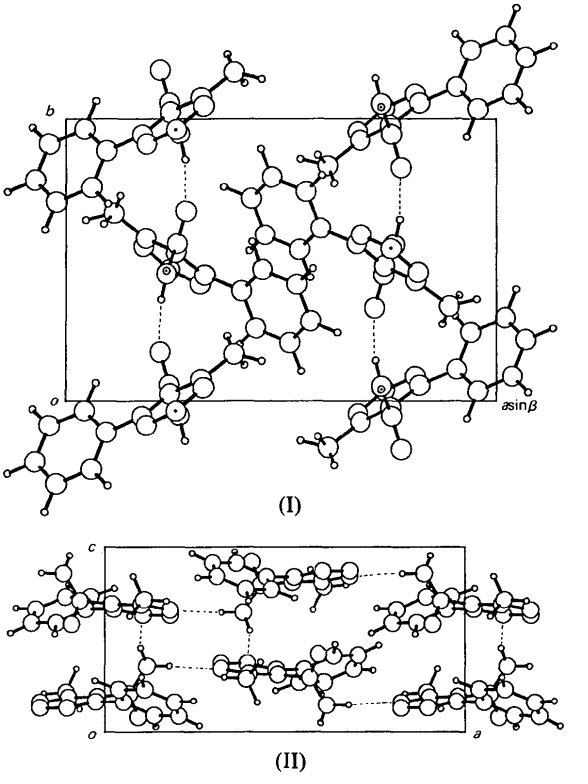

Fig. 2. Projections of the structures of forms (I) and (II). Dots on both N(2) and H(10*B*) indicate hydrogen bonds along the **c** direction in (I).

Table 3. *Selected bond lengths* (Å), *angles* (°), *torsion angles* (°) *and hydrogen-bond lengths* (Å)

	(I)	(II)
N(1)—N(2)	1·314 (3)	1·314 (6)
N(2)—N(3)	1·321 (4)	1·325 (8)
N(3)—C(4)	1·345 (4)	1·344 (7)
C(4)—C(5)	1·383 (4)	1·390 (7)
C(5)—C(6)	1·384 (4)	1·392 (7)
C(6)—N(1)	1·350 (3)	1·347 (6)
C(4)—C(7)	1·490 (6)	1·497 (9)
C(5)—C(8)	1·513 (4)	1·506 (7)
C(8)—O(9)	1·227 (3)	1·242 (7)
C(8)—N(10)	1·319 (4)	1·323 (7)
C(6)—C(11)	1·475 (4)	1·473 (7)
C(6)—N(1)—N(2)	119·8 (2)	121·3 (4)
N(1)—N(2)—N(3)	122·6 (2)	121·4 (4)
N(2)—N(3)—C(4)	119·5 (2)	119·8 (4)
N(3)—C(4)—C(5)	120·5 (2)	121·1 (4)
C(4)—C(5)—C(6)	177·2 (2)	116·4 (4)
C(5)—C(6)—N(1)	120·0 (2)	119·7 (4)
N(3)—C(4)—C(7)	114·1 (2)	115·4 (5)
C(5)—C(4)—C(7)	125·3 (2)	123·4 (5)
C(4)—C(5)—C(8)	119·0 (2)	120·0 (4)
C(6)—C(5)—C(8)	123·7 (2)	123·3 (4)
C(5)—C(8)—O(9)	120·6 (2)	117·3 (4)
C(5)—C(8)—N(10)	114·8 (2)	119·1 (4)
O(9)—C(8)—N(10)	124·4 (2)	123·5 (4)
C(5)—C(6)—C(11)	126·0 (2)	126·3 (4)
N(1)—C(6)—C(11)	113·8 (2)	113·9 (4)
C(4)—C(5)—C(8)—O(9)	−75·2 (2)	−68·3 (4)
C(6)—C(5)—C(8)—N(10)	−75·8 (2)	−74·2 (5)
C(5)—C(6)—C(11)—C(12)	−37·8 (2)	−37·6 (5)
N(10)···O(9)	2·862 (3)	
N(10)···N(2)	2·990 (3)	
N(10)···N(1)		3·059 (7)
N(10)···N(3)		3·087 (6)

parameter of each bonded atom. Major computations performed on a PANAFACOM computer with the *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis program system. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic parameters are listed in Table 2.* Selected bond lengths, angles, torsion angles and hydrogen-bond lengths are listed in Table 3. Fig. 1 shows an *ORTEP* drawing (Johnson, 1965) of form (I) of the molecule with the atom labels. Fig. 2 gives the crystal structures.

Related literature. By comparison with the similar compound 4-methyl-6-phenyl-1,2,3-triazine (Yamaguchi, Ohsawa & Itoh, 1990), the bond lengths and angles are almost consistent in the triazine rings (differences are less than 0·01 Å and 0·5°, respectively).

* Tables of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54235 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of 3-(Substituted benzamido)-5,6,7,8-tetrahydro-5,8-methanoisoquinolines

BY HIROSHI NAKAI, MAMORU TAKASUKA, TADASHI IRIE AND HIROSHI TANIDA

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima-ku, Osaka 553, *Japan*

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Abstract. 3-Benzamido-5,6,7,8-tetrahydro-5,8-methanoisoquinoline (1): $C_{17}H_{16}N_2O$, $M_r = 264{\cdot}33$, monoclinic, $P2_1/c$, $a = 11{\cdot}168$ (4), $b = 13{\cdot}158$ (4), $c = 9{\cdot}678$ (8) Å, $\beta = 105{\cdot}68$ (3)°, $V = 1369$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}282$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}65$ mm^{-1}, $F(000) = 560$, $T = 295$ K, $R = 0{\cdot}076$ for 2005 observed reflections. 3-(*m*-Chlorobenzamido)-5,6,7,8-tetrahydro-5,8-methanoisoquinoline (2): $C_{17}H_{15}ClN_2O$, $M_r = 298{\cdot}77$, monoclinic, $P2_1/c$, $a = 11{\cdot}515$ (3), $b = 14{\cdot}241$ (2), $c = 9{\cdot}576$ (2) Å, $\beta = 108{\cdot}03$ (2)°, $V = 1493{\cdot}1$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}329$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu =$

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