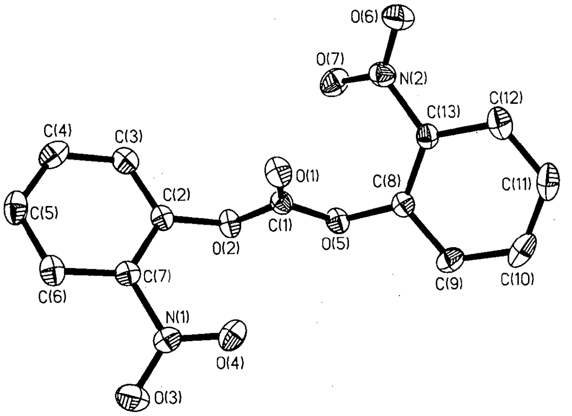

Fig. 1. Thermal-ellipsoid (50% probability) plot of (I).

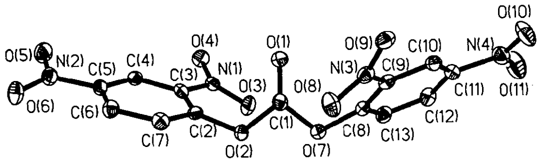

Fig. 2. Thermal-ellipsoid (50% probability) plot of (II).

and (II) are contained in Tables 2 and 3.* Figs. 1 and 2 illustrate the molecules with the numbering schemes employed.

Related literature. For additional information on related carbonate structures and chemistry, see Bendler (1985), Klug (1955), Perez & Scaringe (1987) and Henrichs & Luss (1988).

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52704 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of α-Truxillamide Monohydrate

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Abstract. 2,4-Diphenyl-1,3-cyclobutanedicarboxamide monohydrate, $C_{18}H_{18}N_2O_2.H_2O$, $M_r = 312{\cdot}18$, m.p. 551–553 K, orthorhombic, *Pbcn*, $a = 16{\cdot}710\,(4)$, $b = 21{\cdot}111\,(2)$, $c = 8{\cdot}901\,(1)$ Å, $V = 3139{\cdot}9\,(8)$ Å^3, $Z = 8$, $D_m = 1{\cdot}32\,(2)$, $D_x = 1{\cdot}321$ Mg m^{-3}, Cu $K\alpha(\lambda = 1{\cdot}54178$ Å), $\mu = 0{\cdot}75$ mm^{-1}, $F(000) = 1328$, $T = 295$ K, $R = 0{\cdot}069$ for 1988 unique reflections with $|F_o|$ larger than $2\sigma(F_o)$. Cinnamamide has been ascertained to be photodimerizable in the crystal form grown from an ethanol solution and the crystal structure of a monohydrate of the photodimer has been determined.

Experimental. Crystals of the monomer, cinnamamide, were grown from an ethanol solution by slow evaporation. The Weissenberg photographs of the crystals showed that the space group, lattice constants and intensity distribution are the same as those of the crystals grown from a chloroform solution (Iwamoto, Kashino & Haisa, 1989*a*). Photodimerization was effected by exposing the monomer crystals to sunlight for two months (the method used by Cohen, Schmidt & Sonntag, 1964). At first, the crystals of the anhydrate of the photodimer ($P2_1/a$, $Z = 4$) (Vaida, Shimon, van Mil, Ernst-Cabrera, Addadi, Leiserowitz & Lahav, 1989) were grown from an ethanol solution of the irradiated sample by slow evaporation. The crystals of the monohydrate of the photodimer were obtained from an ethanol solution of the anhydrate by recrystallization. D_m by flotation in aqueous KI. A colorless prism elongated along **c**; 0·12 × 0·07 × 0·30 mm. Systematic absences $0kl$ k odd, $h0l$ l odd, $hk0$ $h+k$ odd, space group

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Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i \beta_{ii}/a_{ii}^{*2}.$$

	x	y	z	$B_{\rm eq}$
O(*W*)	0·0462 (2)	0·0186 (1)	0·3782 (3)	4·5 (1)
O(1*A*)	0·1523 (2)	0·1144 (1)	0·4483 (3)	3·7 (1)
N(1*A*)	0·2289 (2)	0·1998 (1)	0·4880 (3)	3·9 (1)
C(1*A*)	0·0692 (2)	0·2211 (2)	0·0847 (4)	3·2 (2)
C(2*A*)	0·0079 (2)	0·2106 (2)	−0·0173 (4)	4·4 (2)
C(3*A*)	−0·0366 (3)	0·2599 (2)	−0·0733 (5)	5·0 (2)
C(4*A*)	−0·0210 (3)	0·3213 (2)	−0·0293 (5)	5·1 (2)
C(5*A*)	0·0394 (3)	0·3323 (2)	0·0737 (5)	4·9 (2)
C(6*A*)	0·0838 (2)	0·2824 (2)	0·1306 (4)	3·7 (2)
C(7*A*)	0·1149 (2)	0·1649 (2)	0·1470 (4)	3·2 (2)
C(8*A*)	0·1936 (2)	0·1758 (2)	0·2338 (4)	3·2 (2)
C(9*A*)	0·1897 (2)	0·1603 (2)	0·3991 (4)	3·0 (1)
O(1*B*)	0·2031 (2)	0·1850 (1)	−0·1674 (3)	4·1 (1)
N(1*B*)	0·1247 (2)	0·1023 (1)	−0·2242 (3)	3·8 (1)
C(1*B*)	0·2820 (2)	0·0719 (2)	0·1897 (4)	3·7 (2)
C(2*B*)	0·3454 (3)	0·0830 (2)	0·2852 (5)	4·8 (2)
C(3*B*)	0·3910 (3)	0·0329 (3)	0·3388 (5)	6·5 (3)
C(4*B*)	0·3736 (3)	−0·0281 (2)	0·3008 (5)	5·8 (2)
C(5*B*)	0·3111 (3)	−0·0395 (2)	0·2049 (6)	5·9 (3)
C(6*B*)	0·2650 (3)	0·0103 (2)	0·1491 (5)	4·9 (2)
C(7*B*)	0·2376 (2)	0·1283 (2)	0·1263 (4)	3·4 (2)
C(8*B*)	0·1591 (2)	0·1189 (2)	0·0363 (4)	3·4 (1)
C(9*B*)	0·1645 (2)	0·1383 (2)	−0·1271 (4)	3·3 (2)

Table 2. *Bond lengths* (Å) *and angles* (°), *and hydrogen-bond geometry* (Å *and* °), *with e.s.d.'s in parentheses*

	Portion *A*	Portion *B*
O(1)—C(9)	1·233 (5)	1·232 (4)
N(1)—C(9)	1·323 (5)	1·329 (4)
C(1)—C(2)	1·387 (6)	1·378 (6)
C(1)—C(6)	1·379 (6)	1·379 (6)
C(1)—C(7)	1·516 (6)	1·512 (6)
C(2)—C(3)	1·373 (6)	1·388 (6)
C(3)—C(4)	1·379 (7)	1·363 (7)
C(4)—C(5)	1·383 (6)	1·370 (7)
C(5)—C(6)	1·384 (6)	1·395 (6)
C(7)—C(8)	1·542 (6)	1·550 (5)
C(8)—C(9)	1·509 (6)	1·514 (4)
C(7*A*)—C(8*B*)	1·568 (5)	
C(8*A*)—C(7*B*)	1·569 (6)	
O(1)—C(9)—N(1)	122·3 (4)	122·0 (3)
O(1)—C(9)—C(8)	122·6 (4)	121·9 (3)
N(1)—C(9)—C(8)	115·2 (3)	116·1 (3)
C(1)—C(2)—C(3)	121·1 (4)	120·3 (4)
C(1)—C(6)—C(5)	120·7 (4)	120·2 (4)
C(1)—C(7)—C(8)	119·7 (3)	120·5 (3)
C(2)—C(3)—C(4)	120·5 (5)	121·2 (5)
C(2)—C(1)—C(6)	118·3 (4)	118·7 (4)
C(2)—C(1)—C(7)	119·1 (4)	118·3 (4)
C(3)—C(4)—C(5)	119·0 (5)	118·9 (5)
C(4)—C(5)—C(6)	120·4 (4)	120·7 (5)
C(6)—C(1)—C(7)	122·5 (4)	122·9 (4)
C(7)—C(8)—C(9)	114·8 (3)	114·3 (3)
C(1*A*)—C(7*A*)—C(8*B*)	119·5 (3)	
C(7*A*)—C(8*A*)—C(7*B*)	89·9 (3)	
C(8*A*)—C(7*A*)—C(8*B*)	90·3 (3)	
C(9*A*)—C(8*A*)—C(7*B*)	118·5 (3)	
C(1*B*)—C(7*B*)—C(8*A*)		120·4 (3)
C(7*B*)—C(8*B*)—C(7*A*)		89·7 (3)
C(8*B*)—C(7*B*)—C(8*A*)		90·0 (3)
C(9*B*)—C(8*B*)—C(7*A*)		117·7 (3)

Donor (*D*)	Acceptor (*A*)	*D*···*A*	H···*A*	*D*—H···*A*
O(*W*$^{\rm i}$)	O(1*A*$^{\rm i}$)	2·761 (4)	1·93 (4)	167 (4)
N(1*A*$^{\rm i}$)	O(1*B*$^{\rm ii}$)	3·113 (4)	2·15 (4)	172 (3)
N(1*B*$^{\rm i}$)	O(1*A*$^{\rm iii}$)	2·962 (4)	2·07 (4)	171 (4)
O(*W*$^{\rm i}$)	O(*W*$^{\rm iv}$)	2·755 (6)	2·08 (7)	149 (7)
N(1*A*$^{\rm i}$)	O(1*B*$^{\rm v}$)	3·020 (4)	2·09 (4)	173 (4)
N(1*B*$^{\rm i}$)	O(*W*$^{\rm vi}$)	3·011 (4)	2·14 (4)	168 (4)

Symmetry code: (i) x, y, z; (ii) $x, y, 1 + z$; (iii) $x, y, -1 + z$; (iv) $-x, y, \frac{1}{2} - z$; (v) $\frac{1}{2} - x, \frac{1}{2} - y, \frac{1}{2} + z$; (vi) $x, -y, -\frac{1}{2} + z$.

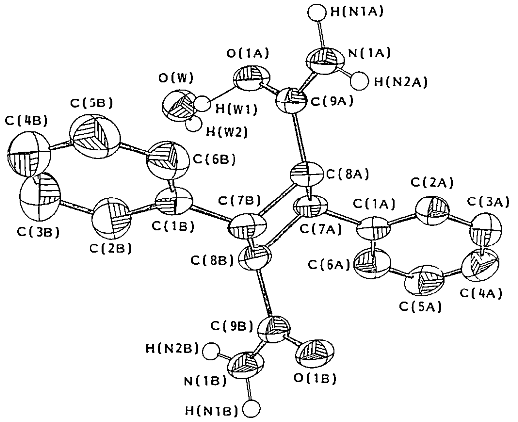

Fig. 1. The thermal ellipsoids with atomic numbering. Ellipsoids of 50% probability are drawn for the non-H atoms; the H atoms attached to N and O atoms are represented as spheres equivalent to $B = 1·0$ Å^2.

Pbcn. Rigaku AFC-5 four-circle diffractometer equipped with rotating anode (40 kV, 200 mA), Ni-filtered Cu *Kα*. 20 reflections within $22 \leq 2\theta \leq 39°$ were used to determine the lattice parameters. Intensity data were collected within $2\theta_{\rm max} = 125°$ ($0 \leq h \leq 19$, $0 \leq k \leq 24$, $-1 \leq l \leq 10$); ω–2θ scan method [scan speed 4° min^{-1} in ω, scan range $(1·2 + 0·15\tan\theta)°$ in ω], background measured for 4 s on either side of the peak; three standard reflections recorded after every 97 reflections, the fluctuation was within 1·7% in $|F_o|$. Lorentz and polarization corrections, no absorption correction. 2514 unique reflections were measured, $R_{\rm int} = 0·040$ for 354 *hkl* reflections. 1988 reflections with $|F_o|$ larger than $2\sigma(F_o)$ were used in structure analysis and refinement. The structure was solved by *MULTAN*78 and refined by block-diagonal least-squares (non-H atoms anisotropically). H atoms were determined from difference Fourier maps and refined isotropically; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = 1·0/[\sigma(F_o)^2 + 0·0406|F_o| + 0·0003|F_o|^2]$. Final $R = 0·069$ for 1988 reflections, $wR = 0·079$, $S = 1·174$ and $(\Delta/\sigma)_{\rm max} = 0·3$. The residual electron density in the final difference map was $-0·19$ to $0·22$ e Å^{-3}.

Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974). Programs *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *HBLS*-V and *DAPH* (Ashida, 1973), *MOLCON* (Fujii, 1979) and *ORTEP* (Johnson, 1971). Computations were carried out at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University, and at the Okayama University Computer Center. The final atomic parameters are listed in Table 1.* Thermal ellipsoids of the photodimer and water molecule are shown in Fig. 1 with the atomic numbering. Bond lengths and angles, and geometry of the hydrogen bonds are listed in Table 2.

Related literature. In contrast with the monomer crystals grown from a chloroform solution (Iwamoto, Kashino & Haisa, 1989*a*), the crystals grown from an ethanol solution are photoreactive. Evidence of the solid-state photoreaction of cinnamamide has been described by Osaki & Schmidt (1972). The molecule of the dimer has a pseudo center of symmetry in the crystals and the conformations around the C—C bonds of the cyclobutane ring are similar to those found in 4,4′-dimethyl-α-truxillic acid having $\bar{1}$ symmetry (Kashino, Oka & Haisa, 1989). The crystal structure is stabilized by the three-dimensional hydrogen-bond network involving the amide groups and the water molecules, as found in a tetrahydrate of the photodimer of α-acetylaminocinnamic acid (Iwamoto, Kashino & Haisa, 1989*b*).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52658 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Verticine *N*-Oxide Hydrate

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Abstract. $C_{27}H_{45}NO_4.H_2O$, $M_r = 465{\cdot}68$, monoclinic, $P2_1$, $a = 10{\cdot}830\ (2)$, $b = 5{\cdot}859\ (1)$, $c = 20{\cdot}187\ (4)$ Å, $\beta = 97{\cdot}26\ (2)°$, $V = 1270{\cdot}7\ (3)$ Å^3, $Z = 2$, $D_m = 1{\cdot}21\ (1)$, $D_x = 1{\cdot}217$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}59$ mm^{-1}, $F(000) = 512$, $T = 293$ K, $R = 0{\cdot}047$ for 1448 unique observed reflections. All the six-membered rings were found to have normal chair conformations and the five-membered ring takes an

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