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-acetylamino-cinnamic acid (Iwamoto, Kashino & Haisa, 1989*b*).

The authors wish to thank Emeritus Professor Masao Haisa of Okayama University for his time spent with us on the work and Emeritus Professor Kenji Osaki of Kyoto University for discussion on the early work of solid-state reactions of cinnamamide. The Research Center for Protein Engineering, Institute for Protein Research, Osaka University, is gratefully acknowledged for the use of the facility.

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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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Table 1. *Final atomic coorindates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* $B_{\rm cq}$ *defined as* $B_{\rm eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}.\mathbf{a}_j$

	x	y	z	$B_{\rm eq}$(Å^2)
C(1)	− 1388 (4)	2068 (9)	271 (2)	3·78
C(2)	− 1867 (4)	3456 (8)	− 358 (2)	3·80
C(3)	− 3272 (4)	3656 (9)	− 426 (2)	3·91
C(4)	− 3709 (4)	4664 (8)	196 (2)	3·88
C(5)	− 3244 (3)	3217 (8)	806 (2)	3·33
C(6)	− 3783 (3)	3906 (9)	1443 (2)	3·89
C(7)	− 3411 (4)	2190 (9)	1999 (2)	3·80
C(8)	− 1989 (3)	1988 (8)	2131 (2)	3·41
C(9)	− 1475 (3)	1367 (8)	1482 (2)	3·13
C(10)	− 1789 (3)	3041 (7)	911 (2)	3·10
C(11)	− 87 (4)	832 (8)	1733 (2)	3·66
C(12)	− 55 (4)	104 (8)	2484 (2)	3·44
C(13)	716 (3)	1805 (8)	2948 (2)	3·07
C(14)	− 1439 (3)	99 (8)	2596 (2)	3·33
C(15)	1645 (4)	167 (9)	3327 (2)	4·15
C(16)	− 857 (4)	1980 (9)	3733 (2)	3·97
C(17)	508 (4)	1518 (8)	3669 (2)	3·50
C(18)	2084 (3)	1528 (8)	2854 (2)	3·48
C(19)	1164 (4)	5337 (9)	1066 (2)	4·33
C(20)	1412 (3)	3011 (8)	4163 (2)	3·33
C(21)	1293 (4)	2379 (11)	4880 (2)	5·16
C(22)	2760 (4)	2616 (8)	4022 (2)	3·48
C(23)	3715 (4)	4019 (9)	4477 (2)	3·96
C(24)	5046 (4)	3471 (10)	4343 (2)	4·61
C(25)	5229 (4)	3934 (9)	3627 (2)	4·32
C(26)	4227 (3)	2705 (9)	3159 (2)	3·65
C(27)	5351 (4)	6494 (10)	3492 (2)	5·32
N(1)	2907 (3)	3121 (6)	3301 (1)	2·99
O(1)	3699 (3)	5113 (7)	− 988 (1)	4·94
O(2)	− 5121 (2)	4094 (6)	1329 (1)	4·18
O(3)	1109 (2)	5373 (6)	4079 (1)	4·26
O(4)	2584 (2)	5387 (6)	3142 (1)	3·57
O(5)	3319 (4)	7045 (8)	1956 (2)	7·94

envelope form. The ring junctions *A*/*B*, *B*/*C*, *D*/*E* and *E*/*F* are *trans* and *C*/*D* is *cis*. An elaborate network of hydrogen bonds interlinks the molecules.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·541 (6)	C(15)—C(16)	1·535 (7)
C(2)—C(3)	1·515 (6)	C(16)—C(17)	1·524 (6)
C(3)—C(4)	1·517 (6)	C(17)—C(13)	1·511 (5)
C(3)—O(1)	1·449 (5)	C(13)—C(12)	1·539 (6)
C(4)—C(5)	1·526 (6)	C(17)—C(20)	1·571 (6)
C(5)—C(10)	1·566 (5)	C(20)—C(22)	1·540 (6)
C(5)—C(6)	1·533 (6)	C(20)—C(21)	1·515 (6)
C(6)—C(7)	1·522 (6)	C(20)—O(3)	1·427 (6)
C(6)—O(2)	1·443 (5)	C(22)—C(23)	1·532 (6)
C(7)—C(8)	1·534 (5)	C(22)—N(1)	1·513 (5)
C(8)—C(9)	1·529 (5)	N(1)—C(18)	1·509 (5)
C(8)—C(14)	1·524 (6)	C(18)—C(13)	1·525 (5)
C(9)—C(10)	1·519 (6)	C(23)—C(24)	1·534 (6)
C(10)—C(1)	1.525 (6)	C(24)—C(25)	1·507 (6)
C(10)—C(19)	1·521 (7)	C(25)—C(26)	1·527 (6)
C(9)—C(11)	1·557 (5)	C(25)—C(27)	1·533 (8)
C(11)—C(12)	1·573 (6)	C(26)—N(1)	1·513 (5)
C(12)—C(14)	1·543 (5)	N(1)—O(4)	1·400 (5)
C(14)—C(15)	1·520 (6)		

C(10)—C(1)—C(2)	113·5 (4)	C(17)—C(13)—C(18)	111·9 (3)
C(1)—C(2)—C(3)	110·2 (4)	C(8)—C(14)—C(12)	102·7 (3)
C(2)—C(3)—C(4)	111·7 (4)	C(8)—C(14)—C(15)	119·0 (4)
C(2)—C(3)—O(1)	109·5 (3)	C(12)—C(14)—C(15)	114·0 (4)
C(4)—C(3)—O(1)	108·5 (3)	C(14)—C(15)—C(16)	113·4 (4)
C(3)—C(4)—C(5)	110·2 (4)	C(15)—C(16)—C(17)	108·2 (4)
C(4)—C(5)—C(10)	111·7 (3)	C(16)—C(17)—C(13)	109·0 (4)
C(4)—C(5)—C(6)	114·1 (4)	C(16)—C(17)—C(20)	112·4 (3)
C(10)—C(5)—C(6)	112·7 (3)	C(13)—C(17)—C(20)	113·1 (3)
C(5)—C(6)—C(7)	110·6 (4)	C(13)—C(18)—N(1)	111·7 (3)
C(5)—C(6)—O(2)	111·5 (3)	C(17)—C(20)—C(22)	109·2 (3)
C(7)—C(6)—O(2)	109·6 (3)	C(17)—C(20)—C(21)	110·4 (4)
C(6)—C(7)—C(8)	110·2 (4)	C(17)—C(20)—O(3)	110·4 (3)
C(7)—C(8)—C(9)	109·6 (3)	C(21)—C(20)—C(22)	109·7 (4)
C(7)—C(8)—C(14)	118·0 (4)	C(21)—C(20)—O(3)	107·7 (3)
C(9)—C(8)—C(14)	101·4 (3)	C(22)—C(20)—O(3)	109·5 (3)
C(8)—C(9)—C(10)	115·2 (3)	C(20)—C(22)—N(1)	111·4 (3)
C(8)—C(9)—C(11)	102·2 (3)	C(20)—C(22)—C(23)	113·1 (3)
C(10)—C(9)—C(11)	119·4 (3)	N(1)—C(22)—C(23)	109·0 (3)
C(1)—C(10)—C(5)	107·4 (3)	C(22)—C(23)—C(24)	111·1 (4)
C(1)—C(10)—C(9)	110·0 (3)	C(23)—C(24)—C(25)	111·8 (4)
C(1)—C(10)—C(19)	109·7 (4)	C(24)—C(25)—C(26)	109·9 (4)
C(5)—C(10)—C(9)	105·7 (3)	C(24)—C(25)—C(27)	111·8 (4)
C(5)—C(10)—C(19)	112·6 (3)	C(26)—C(25)—C(27)	115·0 (4)
C(9)—C(10)—C(19)	111·3 (3)	C(25)—C(26)—N(1)	114·9 (4)
C(9)—C(11)—C(12)	105·7 (3)	C(18)—N(1)—C(22)	109·2 (3)
C(11)—C(12)—C(13)	110·8 (3)	C(18)—N(1)—C(26)	106·7 (3)
C(11)—C(12)—C(14)	103·8 (3)	C(18)—N(1)—O(4)	109·7 (3)
C(13)—C(12)—C(14)	112·0 (3)	C(22)—N(1)—C(26)	111·6 (3)
C(12)—C(13)—C(17)	112·2 (3)	C(22)—N(1)—O(4)	110·6 (3)
C(12)—C(13)—C(18)	108·5 (3)	C(26)—N(1)—O(4)	108·9 (3)

Experimental. Crystallization from methanol, colourless needles, crystal used: 0·5 × 0·15 × 0·1 mm. D_m by flotation in bromoform/cyclohexane. Systematic absences: 0*k*0 for *k* odd, Syntex *P*2$_1$ diffractometer, graphite-monochromated Cu *Kα* radiation, room temperature, cell dimensions from 15 reflections, $15 < \theta < 35°$, intensity data ($h = 0$ to 11, $k = 0$ to 6, $l = -21$ to 21) by $\theta/2\theta$ scans, $2\theta \leq 110°$, two standards measured every 96 reflections, no significant systematic fluctuation. 1785 unique reflections, 1448 with $I \geq 2\sigma(I)$ considered observed and included in the refinement. Lp correction but none for absorption or extinction. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *MAGIC* (Declercq, Germain & Woolfson, 1979) and refined by block-diagonal least squares. $\Delta\rho$ map showed positions of all H atoms, refinement continued on positional parameters and anisotropic thermal parameters for non-H atoms (isotropic thermal parameters for H atoms were set to $B_{\rm eq}$ of the bonded atom, not refined). In the final cycle $R = 0{\cdot}047$, $wR = 0{\cdot}052$ for observed reflections only, $S = 1{\cdot}6$, max. shift/e.s.d. 0·08. Function minimized $\sum w(\Delta F)^2$, where $w = 1$ if $|F_o| < 30$ and $w = 30/|F_o|$ if $|F_o| \geq 30$. Max. and min. heights in final $\Delta\rho$ synthesis 0·15 and 0·20 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations except *MULTAN* and *MAGIC* were performed with a local version of the *NRC* system (Ahmed, Hall, Pippy & Huber, 1973). Final atomic coordinates of non-H atoms and equivalent isotropic *B*'s are listed in Table 1,* bond distances and angles in Table 2. A perspective drawing of the molecule and numbering of the atoms are shown in Fig. 1.

Related literature. The isolation and identification of the title compound have been previously described (Mašterová, Kettmann & Tomko, 1986). The

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52645 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

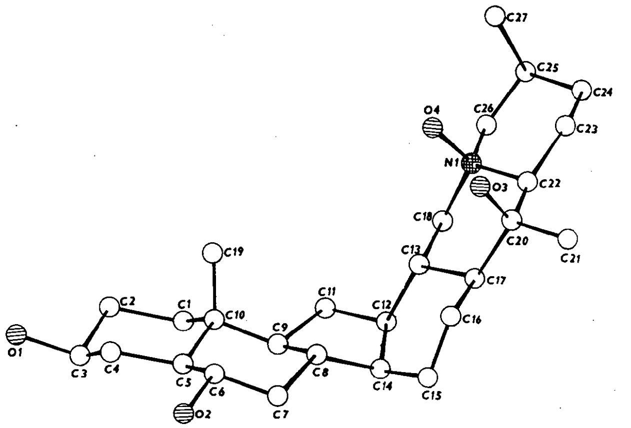

Fig. 1. A perspective view of verticine *N*-oxide in correct absolute configuration, also showing the atom numbering. H atoms are not drawn, for clarity.

molecular structure is similar to that of other related structures (Brisse, 1970; Itô, Fukazawa & Miyashita, 1976; Kettman, Mašterová & Tomko, 1982). Thus, the presence of an *N*-oxide arrangement did not change the usual *trans* configuration at the *E*/*F* ring junction as already observed with quaternization of verticinone with CH_3Br (Itô, Fukazawa & Okuda, 1968), obviously due to a smaller steric volume of the O atom compared with the methyl group.

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Structure of (1*R*,5*R*)-1-*p*-Menth-3-yloxy-2,4-dimethyl-*cis*-2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione*

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Abstract. $C_{16}H_{28}N_4O_3$, $M_r = 324{\cdot}43$, monoclinic, $P2_1$, $a = 10{\cdot}488\,(3)$, $b = 18{\cdot}432\,(6)$, $c = 9{\cdot}429\,(3)$ Å, $\beta = 90{\cdot}63\,(2)^\circ$, $V = 1823\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}182$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1{\cdot}54178$ Å, $\mu = 6{\cdot}38$ cm^{-1}, $F(000) = 704$, room temperature, $R = 0{\cdot}051$ for 2914 unique reflections $[I \geq 5\sigma(I)]$. The asymmetric unit contains two independent molecules. The title compound is the first example of a derivative of the bicyclol tautomer in the allantoin series and the key intermediate in the asymmetric synthesis of (−)-(*R*)-1,3-dimethylallantoin; the results of the X-ray structure determination confirm its diastereomeric identity and establish the absolute (1*R*,5*R*) configuration.

Experimental. The title compound was characterized by its melting point (550–551 K), $[\alpha]_D^{25^\circ C}$ −28° (*c* 1·6, Me_2SO), IR, ^{1}H- and ^{13}C-NMR spectra, as well as by regiospecific conversion into (−)-(*R*)-1,3-dimethylallantoin, $[\alpha]_D^{25^\circ C}$ −75° (*c* 3, H_2O) (Modrić, Drake & Poje, 1989). Recrystallization from ethanol;

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* *p*-Menth-3-yl is (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl.

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