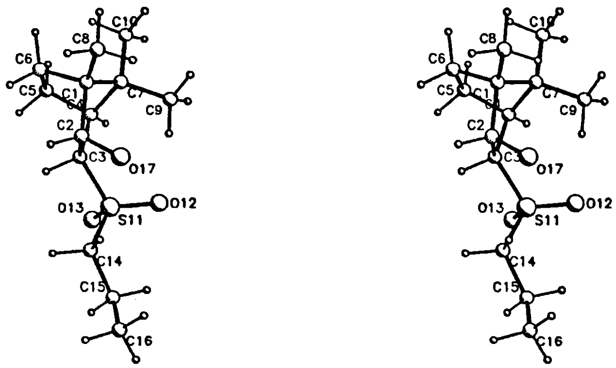

Fig. 1. Stereoscopic view of molecule *A* of compound (I) and atom-numbering scheme.

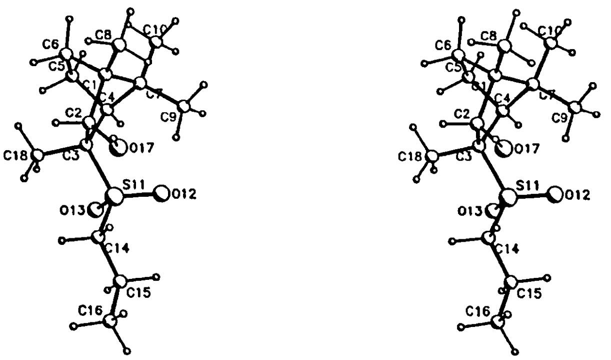

Fig. 2. Stereoscopic view of the molecule of compound (II) and atom-numbering scheme.

Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. The structure of (1*R*,2*S*,*S*)-3-*exo*-(prop-2-enylsulfinyl)-1,7,7-trimethylbicyclo[2.2.1]-heptan-2-ol (Goodridge, Hambley, Haynes & Ridley, 1988) is closely related to the title compounds.

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Structures of *o*-Aminobenzamide and *p*-Hydroxybenzamide Monohydrate

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Abstract. (I) *o*-Aminobenzamide, $C_7H_8N_2O$, $M_r = 136{\cdot}15$, monoclinic, $P2_1/c$, $a = 14{\cdot}173\,(3)$, $b = 6{\cdot}327\,(2)$, $c = 7{\cdot}803\,(2)$ Å, $\beta = 97{\cdot}98\,(2)°$, $V = 692{\cdot}9\,(3)$ Å^3, $Z = 4$, $D_m = 1{\cdot}31$, $D_x = 1{\cdot}306$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}71$ mm^{-1}, $F(000) = 288$, $T = 295$ K, $R = 0{\cdot}062$ for 1031 unique reflections. (II) *p*-Hydroxybenzamide monohydrate, $C_7H_7NO_2.H_2O$, $M_r = 155{\cdot}14$, monoclinic, $P2_1/c$, $a = 16{\cdot}546\,(3)$, $b = 11{\cdot}798\,(2)$, $c = 3{\cdot}8889\,(7)$ Å, $\beta = 100{\cdot}40\,(2)°$, $V = 746{\cdot}7\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}35$, $D_x = 1{\cdot}381$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}93$ mm^{-1}, $F(000) = 328$, $T = 295$ K, $R = 0{\cdot}040$ for 1024 unique reflections. Dihedral angles between the benzene ring and amide planes are 32·5 (2) for (I) and 14·6 (2)° for (II). In (I) an intramolecular hydrogen bond is formed between the *o*-amino and the amide groups. An intermolecular hydrogen bond is formed between the amide groups related by a 2_1 axis. The *o*-amino group accepts and donates the other intermolecular hydrogen bonds. In (II) a centrosymmetric dimer is formed by hydrogen bonds between the amide groups. The hydroxyl group

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Table 1. *Experimental details*

	(I)	(II)
Morphology	Plate developed (100)	Prismatic *c*
Size of specimen (mm)	0·10 × 0·35 × 0·23	0·20 × 0·20 × 0·30
Lattice-parameter refinement		
Number of reflections	20	20
2θ range (°)	12–43	18–48
Systematic absences	*h*0*l*, *l* odd	*h*0*l*, *h* odd
	0*k*0, *k* odd	0*k*0, *k* odd
$2\theta_{max}$ (°)	120	125
Range of *h*	−23 to 23	−18 to 18
k	0 to 11	0 to 13
l	0 to 16	0 to 4
Max. variation of standard reflections (%)	3·2	0·9
R_{int}	0·036 for 93 *hk*0 reflections	0·006 for 193 *hk*0 reflections
Number of		
unique reflections	1031	1204
reflections with $\|F_o\| > \sigma(F_o)$	928	1176
p	−0·0494	0·0271
q	0·0056	0·0001
r	3·6532	7·3270
R, *wR*	0·062, 0·058	0·040, 0·063
S	1·363	0·990
$(\Delta/\sigma)_{max}$ for non-H/H	0·21/0·72	0·13/0·71
$\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	0·20/−0·19	0·12/−0·21

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

$$B_{eq} = (4/3)\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
(I)				
O(1)	0·4339 (1)	0·0176 (2)	0·1649 (2)	5·59 (7)
N(1)	0·3973 (1)	0·3587 (2)	0·1179 (2)	4·85 (8)
N(2)	0·3428 (1)	−0·2378 (2)	−0·0912 (2)	5·88 (9)
C(1)	0·2815 (1)	0·0938 (3)	0·0127 (2)	3·84 (7)
C(2)	0·2680 (1)	−0·0984 (3)	−0·0800 (2)	4·49 (9)
C(3)	0·1768 (2)	−0·1497 (3)	−0·1578 (3)	5·8 (1)
C(4)	0·1012 (1)	−0·0212 (4)	−0·1444 (3)	6·6 (1)
C(5)	0·1134 (1)	0·1681 (4)	−0·0522 (3)	6·2 (1)
C(6)	0·2025 (1)	0·2215 (3)	0·0231 (2)	4·88 (9)
C(7)	0·3766 (1)	0·1544 (3)	0·1023 (2)	3·99 (8)
(II)				
O(1)	0·48247 (6)	0·85432 (9)	0·3440 (3)	4·07 (5)
O(2)	0·71273 (7)	0·43886 (9)	0·1520 (3)	4·05 (5)
O(*W*)	0·61869 (8)	0·2541 (1)	0·1940 (4)	4·65 (6)
N(1)	0·60445 (9)	0·9370 (1)	0·5090 (5)	4·28 (7)
C(1)	0·60084 (9)	0·7422 (1)	0·3214 (4)	2·84 (5)
C(2)	0·67974 (9)	0·7394 (1)	0·2449 (4)	3·29 (6)
C(3)	0·71610 (9)	0·6383 (1)	0·1855 (4)	3·49 (7)
C(4)	0·67435 (9)	0·5373 (1)	0·2051 (4)	2·93 (6)
C(5)	0·59530 (9)	0·5389 (1)	0·2757 (4)	3·43 (6)
C(6)	0·55889 (9)	0·6406 (1)	0·3308 (4)	3·40 (6)
C(7)	0·55896 (9)	0·8490 (1)	0·3918 (4)	3·18 (6)

Table 3. *Bond lengths* (Å) *and angles* (°) *and geometry of hydrogen bonds*

	(I)	(II)
O(1)—C(7)	1·240 (3)	1·247 (2)
O(2)—C(4)		1·357 (2)
N(1)—C(7)	1·327 (3)	1·315 (3)
N(2)—C(2)	1·391 (3)	
C(1)—C(2)	1·414 (3)	1·391 (3)
C(1)—C(6)	1·392 (3)	1·389 (3)
C(1)—C(7)	1·480 (3)	1·487 (3)
C(2)—C(3)	1·388 (3)	1·374 (3)
C(3)—C(4)	1·361 (4)	1·386 (3)
C(4)—C(5)	1·396 (4)	1·384 (3)
C(5)—C(6)	1·359 (3)	1·377 (3)
C(2)—C(1)—C(6)	118·4 (2)	118·6 (2)
C(2)—C(1)—C(7)	120·9 (2)	123·1 (2)
C(6)—C(1)—C(7)	120·6 (2)	118·3 (2)
N(2)—C(2)—C(1)	121·8 (2)	
N(2)—C(2)—C(3)	119·7 (2)	
C(1)—C(2)—C(3)	118·5 (2)	120·9 (2)
C(2)—C(3)—C(4)	121·5 (2)	120·0 (2)
O(2)—C(4)—C(3)		118·4 (2)
O(2)—C(4)—C(5)		121·8 (2)
C(3)—C(4)—C(5)	120·7 (3)	119·8 (2)
C(4)—C(5)—C(6)	118·5 (2)	119·9 (2)
C(1)—C(6)—C(5)	122·5 (2)	120·9 (2)
O(1)—C(7)—N(1)	121·3 (2)	120·9 (2)
O(1)—C(7)—C(1)	120·6 (2)	120·6 (2)
N(1)—C(7)—C(1)	118·1 (2)	118·4 (2)

D	*A*	*D*···*A* (Å)	*D*—H···*A* (°)
(I)			
N(2^{i})	O(1^{i})	2·749 (3)	115 (2)
N(1^{i})	O(1^{ii})	2·912 (3)	165 (2)
N(1^{i})	N(2^{iii})	3·070 (3)	148 (2)
N(2^{iv})	O(1^{i})	3·017 (3)	146 (3)
(II)			
N(1^{i})	O(1^{v})	2·959 (2)	179 (2)
N(1^{i})	O(2^{vi})	3·074 (2)	169 (2)
O(2^{i})	O(W^{i})	2·700 (1)	171 (2)
O(W^{vii})	O(1^{i})	2·748 (1)	168 (3)
O(W^{viii})	O(1^{i})	2·958 (1)	154 (3)

Symmetry codes: (i) x, y, z; (ii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $x, 1 + y, z$; (iv) $x, -\frac{1}{2} - y, \frac{1}{2} + z$; (v) $1 - x, 2 - y, 1 - z$; (vi) $\frac{3}{2} - x, -\frac{1}{2} + y, 1 - z$; (vii) $1 - x, 1 - y, -z$; (viii) $1 - x, 1 - y, 1 - z$.

accepts a hydrogen bond from the amide group of the molecule related by a 2_1 axis. In (II) there are three hydrogen bonds involving the water molecules which form a three-dimensional network.

Experimental. Experimental details are listed in Table 1. Crystals of (I) and (II) were grown by slow evaporation from ethanol solutions. Densities of the crystals of (I) and (II) were measured by flotation in ligroin–CCl_4 and benzene–CCl_4 solutions, respectively. Intensities were collected on Rigaku AFC-5 four-circle diffractometer equipped with rotating anode, by ω–2θ scan method [scan speed 4° min^{-1} in ω, scan range $(1{\cdot}2 + 0{\cdot}15\tan\theta)°$ in ω, Ni-filtered Cu $K\alpha$, 40 kV, 200 mA]. Background was measured for 4 s on either side of peak. Three standard reflections were measured after every 57 reflections for (I) and every 97 reflections for (II). Lorentz and polarization corrections were applied, but no absorption correction was made. All the unique reflections within $2\theta_{max}$ were used in refinements. The structures were solved by *MULTAN*78 and refined by block-diagonal least squares. The value minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma(F_o)^2 + p|F_o| + q|F_o|^2]$ for $|F_o| > 0$, and $w = r$ for $|F_o| = 0$. All H atoms were located on difference Fourier maps. The non-H atoms were refined anisotropically and the H atoms isotropically. For (II), secondary-extinction correction was applied for the strongest three reflections according to $I_{\text{corr}} = I_o(1 + 1{\cdot}7 \times 10^{-5}I_c)$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs used: *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *HBLS*-V and *DAPH* (Ashida, 1973), 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.

Final atomic parameters are listed in Table 2.* Thermal ellipsoids of the molecules with atomic numbering are shown in Fig. 1. Bond lengths and angles and geometry of the hydrogen bonds are listed in Table 3. Stereoscopic views of the crystal structures are shown in Fig. 2.

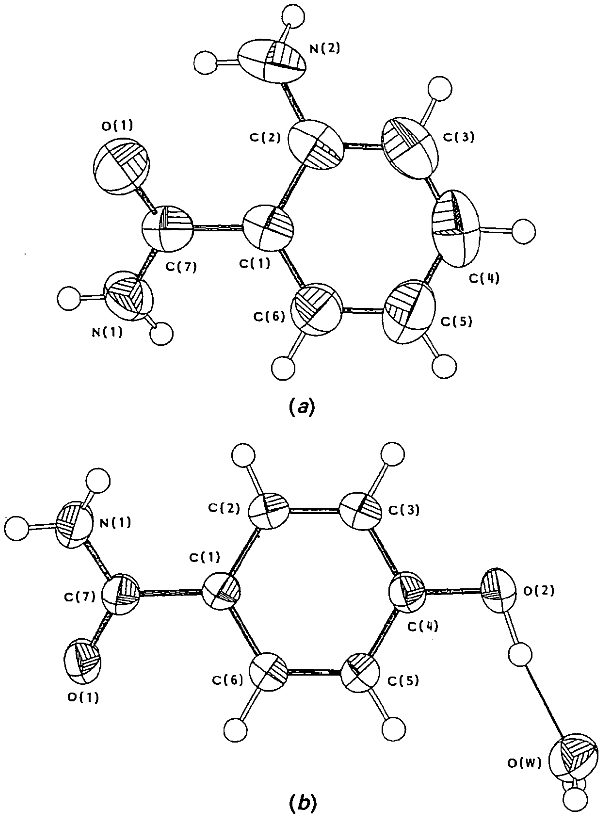

Fig. 1. *ORTEP* (Johnson, 1971) drawings of (*a*) *o*-aminobenzamide (I) and (*b*) *p*-hydroxybenzamide monohydrate (II). The thermal ellipsoids of the molecules are shown with atomic numbering. Ellipsoids of 50% probability are drawn for the non-H atoms; the H atoms are represented as spheres equivalent to $B = 1{\cdot}0$ Å^2.

Related literature. Packing modes of primary amides have been analysed in terms of the elements of symmetry-generating hydrogen-bonded networks from centrosymmetric amide pairs (Leiserowitz & Schmidt, 1969). The packing modes have also been discussed in view of crystal morphology engineering (Berkovitch-Yellin, van Mil, Addadi, Idelson, Lahav & Leiserowitz, 1985). The centrosymmetric dimers as observed in (II) have been found in benzamide (Blake & Small, 1972), *o*-hydroxybenzamide (salicylamide) (Sasada, Takano & Kakudo, 1964), *o*-nitrobenzamide (Fujimori, Tsukihara, Katsube & Yamamoto, 1972), *m*-nitrobenzamide (Nakata, 1987) and *p*-nitrobenzamide (Rienzo, Domenicano & Serantoni, 1977), while such dimers are not formed in (I), *p*-aminobenzamide (Alleaume, 1967) and *m*-hydroxybenzamide (Katsube, Sasada & Kakudo, 1966). In benzamide and *o*-nitrobenzamide the dimers are linked by a hydrogen bond between the amide groups related by a translation. This hydrogen bond is replaced by a hydrogen bond between the amide group and the substituent of the molecules related by a 2_1 axis in (II) and *m*-nitrobenzamide and by a glide plane in *o*-hydroxybenzamide and *p*-nitrobenzamide.

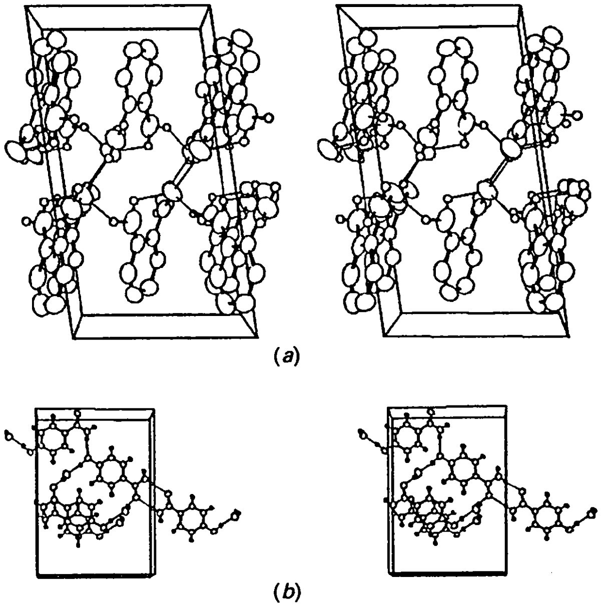

Fig. 2. Stereoviews of the molecular packing of (I) and (II). Hydrogen bonds are shown by thin lines. (*a*) *o*-Aminobenzamide (I); the *a* axis is vertical, the *b* axis is into the plane of paper and the *c* axis is horizontal. (*b*) *p*-Hydroxybenzamide monohydrate (II); the *a* axis is vertical, the *b* axis is horizontal and the *c* axis is into the plane of paper.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54134 (10 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 the Macrocyclic Ligand Tetrakis(2-hydroxyethyl)cyclam at 123 K

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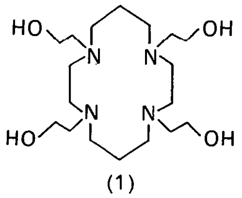
Abstract. 1,4,8,11-Tetrakis(2-hydroxyethyl)-1,4,8,11-tetraazacyclotetradecane, $C_{18}H_{40}N_4O_4$, $M_r = 376{\cdot}54$, monoclinic, $P2_1/n$, $a = 9{\cdot}945\,(1)$, $b = 9{\cdot}562\,(1)$, $c = 11{\cdot}116\,(1)$ Å, $\beta = 100{\cdot}03\,(1)^\circ$, $V = 1040{\cdot}9\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}201$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}079$ mm^{-1}, $F(000) = 416$, $T = 123\,(2)$ K, $R = 0{\cdot}041$ for 1513 unique reflections. The macrocyclic ligand crystallizes in the *RSSR* (*trans* IV) configuration on a crystallographic centre of symmetry, with the electron pairs on 1,8-related N atoms directed away from the centre of the ring. Thus it is not possible from this structure to determine accurately the dimensions of the metal ion binding cavity. Intramolecular hydrogen bonding brings together 1,4-related hydroxyl groups in an association which also involves the N atoms to which they are appended. There is no indication of significant intermolecular hydrogen bonding.

Experimental. Suitable crystals of the title compound, (1), were obtained from the reaction of 1,4,8,11-tetraazacyclotetradecane (cyclam) with excess oxirane in ethanol (Madeyski, Michael & Hancock, 1984) followed by recrystallization of the crude product at 279 K from a $10^{-2}M$ solution in propan-2-ol. The data crystal was a colourless prism $0{\cdot}21 \times 0{\cdot}25 \times 0{\cdot}23$ mm. Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$-scan mode. Unit-cell parameters from 25 accurately centred reflections ($12{\cdot}6 \le \theta \le 18{\cdot}8^\circ$). 3225 reflections measured with $\theta \le 23^\circ$, 494 with $\sigma(I)/I \le 1{\cdot}0$ rejected on fast scan, range $-10 \le h \le 11$, $0 \le k \le 10$, $-12 \le l \le 12$. Three standard reflections (111, $\bar{1}0\bar{5}$, $\bar{2}20$) measured periodically showed no significant variation. Data merged to give 1513 unique reflections, $R_{\text{int}} = 1{\cdot}7\%$, 37 with $I \le 0$.



(1)

The *XTAL System of Crystallographic Programs* (Hall & Stewart, 1989) was used for all computer calculations. Structure was solved by direct methods, H atoms located in difference map. Refined by full-matrix least squares of 198 parameters based on F^2 using *SFLSX* (Hall, Spadaccini, Olthof-Hazekamp & Dreissig, 1989). Scale factor, positional parameters for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms refined. On F^2, final $R = 0{\cdot}054$, $wR = 0{\cdot}058$, $w = 1{\cdot}0/[\sigma(F^2)]^2$ where $\sigma(F^2) = \sigma(I)/(2{\cdot}0|F|)$, $S = 2{\cdot}32$. A structure factor calculation after the

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