

Table 3. *Hydrogen bonds in the crystal structure of  $\beta,\beta$ -trehalose*

Symmetry operations: (1)  $x, y, z$ ; (2)  $y, x, -z$ ; (3)  $x - y, -y, \frac{1}{2} - z$ . The last three digits specify a lattice translation. Each O of H<sub>2</sub>O is hydrogen bonded to four other O's. Each O of sugar OH is bonded to two other O's. O(1) and O(5) are not hydrogen bonded. O(X)→O(Y) means O(X)—H···O(Y). O(X)↔O(Y) means O(X)—H···O(Y) and O(X)···H—O(Y) (50:50) (i.e. disorder in H).

Hydrogen bond	Symmetry operation	Distances (Å)						Angles (°)	
		O—H	H—O	O···O	O···H	H···O	O—H···O	O···H—O	
O(4)↔O(2)	[2,100]	0.80	0.79	2.726	1.95	1.93	173	167	
O(11)↔O(2)	[1,110]	0.83	0.81	2.814	2.02	1.99	172	166	
O(11)→O(3)	[2,000]	0.82	—	2.848	—	2.03	176	—	
O(11)→O(6)	[1,100]	—	0.81	2.733	1.93	—	—	171	
O(11)↔O(11)	[3,000]	0.82	0.82	2.835	2.01	2.01	180	180	
O(12)→O(3)	[1,100]	—	0.81	2.808	2.00	—	—	174	
O(12)↔O(4)	[2,000]	0.81	0.82	2.736	1.94	1.95	163	163	
O(12)→O(6)	[3,110]	0.82	—	2.712	—	1.90	—	170	
O(12)↔O(12)	[2,000]	0.82	0.82	2.752	1.93	1.93	180	180	
E.s.d.'s		±0.05		±0.003		±0.05		±13	

H atoms were located from a difference map. Their positions were refined with fixed isotropic thermal parameters. Final C—H distances range from 0.97 to 1.00 Å, O—H from 0.79 to 0.83 Å. H atoms attached to O(2), O(4) and one of the H atoms of O(11) and O(12) were disordered as indicated by the difference map. Each of these were assigned two positions with site occupancy factors of 0.5. The disorder is required in order to describe the hydrogen-bonding scheme. The disordered model gave a final *R* of 0.029 against an *R* of 0.042 for the non-disordered model. The structure was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1990).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55632 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1014]

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## Structure of *N*-(3-Phthalimidooxypropyl)-trifluoroacetamide

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## Abstract

The title compound consists of a planar phthalimide ring linked via a single N—O bond [1.377 (6) Å] and a propyl chain to an amide function. The packing is stabilized by hydrogen bonds formed between the amide groups.

## Comment

In connection with the synthesis of some polyamine analogues, the title intermediate was prepared (Kong Thoo Lin, 1992) from 3-amino-1-propanol by selectively *N*-trifluoroacetylating with ethyl trifluoroacetate/triethylamine in methanol followed by *O*-toluene-*p*-sulfonation in pyridine. Reaction with

*N*-hydroxyphthalimide/triethylamine in dimethylformamide afforded the product (66% yield) which was crystallized from 2-propanol for crystal structure determination. The final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1. The molecular structure with labelling is depicted in Fig. 1. Selected bond distances and bond angles are given in Table 2.

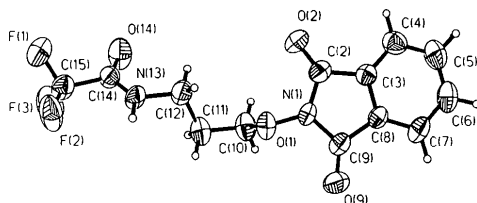


Fig. 1. View of the molecule with labelling. Thermal ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii (SHELXTL/PC).

The phthalimide moiety is planar [mean deviation from best plane through 11 non-H atoms = 0.010 (7) Å]. Its dimensions are in good agreement with those reported for phthalimide (Matzat, 1972) except for the angles around N(1), C(2) and C(9) which differ as a result of the N(1) substitution, and with those reported for diphtalimide phthalate (Bates & Teuber, 1985). N(1) deviates by only 0.041 (5) Å from the plane through its neighbours, and can be considered as being almost in this plane. The N(1)—O(1) distance of 1.377 (6) Å corresponds to a single N—O bond [N—O distance in C<sub>2</sub>—Nsp<sup>2</sup>—O—C = 1.397 (11) Å; Allen *et al.*, 1987]. The O(1)—C(10) bond is located almost perpendicular to

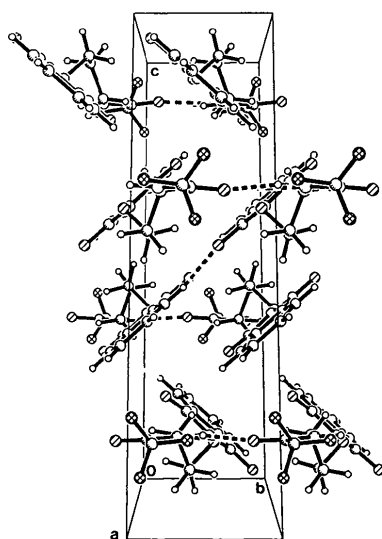


Fig. 2. Molecular packing and hydrogen bonding as seen in projection down *a*. Hydrogen bonds are marked with broken lines (SHELXTL/PC).

the phthalimide plane [C(2)—N(1)—O(1)—C(10) = 85.7 (5)°]. The conformation of the chain O(1) to C(14) can be described as *-ap* [N(1)—O(1)—C(10)—C(11) = -169.6 (4)°], *+sc* [O(1)—C(10)—C(11)—C(12) = 66.1 (6)°], *+ap* [C(10)—C(11)—C(12)—N(13) = 179.2 (5)°], *-ac* [C(11)—C(12)—N(13)—C(14) = -107.4 (5)°], *+ap* [C(12)—N(13)—C(14)—C(15) = 176.6 (5)°].

The hydrogen bonding and molecular packing are illustrated in Fig. 2. The amide N atom N(13) is hydrogen bonded to the amide O atom O(14) in the molecule translated in the *y* direction [N(13)⋯O(14) 2.788 (6), N(13)—H(13) 0.96, H(13)⋯O(14) 1.84 Å, N(13)—H(13)⋯O(14) 169.8°]. Molecules related by an inversion centre show a close contact between O(9) and H(7) (2.45 Å).

## Experimental

### Crystal data

C<sub>13</sub>H<sub>11</sub>F<sub>3</sub>N<sub>2</sub>O<sub>4</sub>  
*M<sub>r</sub>* = 316.2  
 Monoclinic  
*P*2<sub>1</sub>/*n*  
*a* = 15.800 (2) Å  
*b* = 4.985 (1) Å  
*c* = 18.875 (3) Å  
 $\beta$  = 111.21 (1)°  
*V* = 1386.0 (3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.516 Mg m<sup>-3</sup>

Cu K $\alpha$  radiation  
 $\lambda$  = 1.54178 Å  
 Cell parameters from 38 reflections  
 $\theta$  = 6–38°  
 $\mu$  = 1.188 mm<sup>-1</sup>  
*T* = 289 K  
 Needles  
 0.30 × 0.15 × 0.10 mm  
 White

### Data collection

Siemens P4-PC diffractometer  
 2 $\theta$ / $\theta$  scans  
 Absorption correction: none  
 1509 measured reflections  
 1444 independent reflections  
 924 observed reflections  
 [*F* > 4.0 $\sigma$ (*F*)]

*R*<sub>int</sub> = 0.014  
 $\theta_{\max}$  = 50.45°  
*h* = 0 → 15  
*k* = 0 → 4  
*l* = -18 → 17  
 3 standard reflections monitored every 50 reflections  
 intensity variation: 2%

### Refinement

Refinement on *F*  
 Final *R* = 0.0459  
*wR* = 0.0506  
*S* = 1.16  
 924 reflections  
 199 parameters  
 H atoms: riding model, fixed *U*

$w = 1/[\sigma^2(F) + 0.008F^2]$   
 $(\Delta/\sigma)_{\max} = 0.203$   
 $\Delta\rho_{\max} = 0.20 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$   
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)

*U*<sub>eq</sub> is defined as one third of the trace of the orthogonalized *U*<sub>*ij*</sub> tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
N(1)	0.2728 (3)	0.0437 (9)	0.3842 (2)	0.051 (2)
C(2)	0.2869 (4)	-0.1408 (12)	0.3345 (3)	0.051 (3)
C(3)	0.3828 (3)	-0.0913 (11)	0.3421 (3)	0.043 (2)

C(4)	0.4328 (4)	-0.2142 (12)	0.3055 (3)	0.061 (3)
C(5)	0.5205 (4)	-0.1269 (14)	0.3222 (4)	0.073 (3)
C(6)	0.5564 (4)	0.0774 (14)	0.3734 (4)	0.071 (3)
C(7)	0.5052 (4)	0.2034 (12)	0.4102 (3)	0.063 (3)
C(8)	0.4180 (3)	0.1131 (10)	0.3933 (3)	0.044 (2)
C(9)	0.3459 (3)	0.2085 (12)	0.4211 (3)	0.050 (2)
O(1)	0.1894 (2)	0.0764 (7)	0.3909 (2)	0.056 (2)
O(2)	0.2319 (3)	-0.2992 (9)	0.2979 (2)	0.072 (2)
O(9)	0.3478 (2)	0.3869 (8)	0.4649 (2)	0.074 (2)
C(10)	0.1809 (3)	-0.1178 (12)	0.4447 (3)	0.063 (3)
C(11)	0.0834 (3)	-0.1074 (12)	0.4384 (3)	0.062 (3)
C(12)	0.0192 (3)	-0.2037 (11)	0.3623 (3)	0.058 (3)
N(13)	-0.0749 (3)	-0.1952 (9)	0.3582 (2)	0.053 (2)
C(14)	-0.1203 (3)	-0.4119 (13)	0.3597 (3)	0.049 (3)
C(15)	-0.2153 (4)	-0.3647 (12)	0.3594 (4)	0.059 (3)
O(14)	-0.0931 (3)	-0.6402 (8)	0.3644 (3)	0.090 (2)
F(1)	-0.2755 (2)	-0.5044 (8)	0.3055 (2)	0.108 (2)
F(2)	-0.2421 (2)	-0.1163 (7)	0.3503 (2)	0.095 (2)
F(3)	-0.2227 (2)	-0.4471 (9)	0.4225 (2)	0.113 (2)

Table 2. Geometric parameters (Å, °)

N(1)—C(2)	1.389 (8)	N(1)—C(9)	1.382 (6)
N(1)—O(1)	1.377 (6)	C(2)—C(3)	1.490 (8)
C(2)—O(2)	1.192 (6)	C(3)—C(4)	1.369 (9)
C(3)—C(8)	1.375 (7)	C(4)—C(5)	1.375 (9)
C(5)—C(6)	1.377 (9)	C(6)—C(7)	1.393 (10)
C(7)—C(8)	1.373 (7)	C(8)—C(9)	1.495 (9)
C(9)—O(9)	1.208 (7)	O(1)—C(10)	1.445 (7)
C(2)—N(1)—C(9)	115.1 (5)	C(2)—N(1)—O(1)	122.2 (4)
C(9)—N(1)—O(1)	122.4 (5)	N(1)—C(2)—C(3)	103.4 (4)
N(1)—C(2)—O(2)	124.6 (6)	C(3)—C(2)—O(2)	132.0 (6)
C(2)—C(3)—C(4)	129.0 (5)	C(2)—C(3)—C(8)	109.2 (5)
C(4)—C(3)—C(8)	121.7 (5)	C(3)—C(4)—C(5)	117.4 (5)
C(4)—C(5)—C(6)	121.4 (7)	C(5)—C(6)—C(7)	121.2 (6)
C(6)—C(7)—C(8)	116.8 (5)	C(3)—C(8)—C(7)	121.5 (5)
C(3)—C(8)—C(9)	108.5 (4)	C(7)—C(8)—C(9)	130.0 (5)
N(1)—C(9)—C(8)	103.8 (5)	N(1)—C(9)—O(9)	126.3 (6)
C(8)—C(9)—O(9)	129.9 (5)	N(1)—O(1)—C(10)	108.7 (3)

Data collection and reduction: *P3/PC Data Collection* (Siemens, 1989). Program package used to solve and refine the structure: *SHELXTL/PC* (Siemens, 1990). Refinement was by full-matrix least-squares methods.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55684 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1028]

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## An Original Linkage Between Arabinose and a Pyranopyrimidine Derivative via a Sulfur Atom

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## Abstract

The structure of 5-methyl-2-[(2,3,4-tri-*O*-acetyl- $\beta$ -D-arabinopyranosyl)thio]-3,4-dihydro-7*H*-pyrano[2,3-*d*]pyrimidine-4,7-dione, C<sub>19</sub>H<sub>20</sub>N<sub>2</sub>O<sub>10</sub>S, is described. This compound consists of a sugar ring and a planar base (pyranopyrimidine derivative) linked unusually by an S atom. The sugar ring is in a <sup>4</sup>C<sub>1</sub> chair conformation and makes dihedral angles of 74.5 (4) and 75.7 (4)° with the pyran and pyrimidine rings. The S atom occupies an axial position on the sugar ring and lies 2.461 (9) Å out of the corresponding mean plane.

## Comment

Synthetic nucleosides are widely studied and used therapeutically as antiviral (Lin *et al.*, 1991), anti-tumor (Ramasamy *et al.*, 1990) and antibiotic compounds (Knapp & Shieh, 1991). Our aim is to synthesize nucleosides by substituting the heterocyclic base (pyrimidine or purine) by a pyranopyrimidine ring (1). From this compound, the nucleosides (2) were synthesized and their antiviral

† This publication is dedicated to Marie-Eve Rolland.