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The Structures of 1-(2′-hydroxybenzoyl)piperidine and 4-(2′-hydroxybenzoyl)morpholine

Franz Pertlik

Institut für Mineralogie und Kristallographie, Universität Wien, A-1010 Wien, Österreich

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1-(2′-hydroxybenzoyl)piperidine, $C_{12}H_{15}NO_2$, orthorhombic, $Pbc2_1-C_{2v}^5$; $a = 9.529$ (1), $b = 10.001$ (1), $c = 11.652$ (1) Å; $Z = 4$. The structure was determined from 1 510 single crystal X-ray data by direct methods and the refinement of the structure parameters yielded $R = 0.040$. There is one molecule in the asymmetric unit.

4-(2′-hydroxybenzoyl)morpholine, $C_{11}H_{13}NO_3$, orthorhombic, $P2_12_12_1-D_2^4$; $a = 13.006$ (1), $b = 17.258$ (2), $c = 9.125$ (1) Å; $Z = 8$. The structure parameters of the isotypic compound 4-(2′-hydroxythiobenzoyl)morpholine were used in a starting set of refinement; finally $R = 0.054$ was obtained for 1 900 single crystal X-ray data. There are two molecules in the asymmetric unit.

In both compounds the hydrogen atoms could be located by means of difference *Fourier* summations. The intermolecular hydrogen bonds with O . . . O distances of 2.649 (2) Å for the piperidine derivate and 2.688 (3) resp. 2.754 (3) Å for the morpholine derivate are typical for intermediate bond strengths.

(Keywords: 1-(2′-Hydroxybenzoyl)piperidine; 4-(2′-Hydroxybenzoyl)-morpholine; Molecular structure; Crystal structure; Intermolecular hydrogen bond)

Die Strukturen von 1-(2′-hydroxybenzoyl)piperidin und 4-(2′-hydroxybenzoyl)morpholin

1-(2′-hydroxybenzoyl)piperidin, $C_{12}H_{15}NO_2$, orthorhombisch, $Pbc2_1-C_{2v}^5$; $a = 9.529$ (1), $b = 10.001$ (1), $c = 11.652$ (1) Å; $Z = 4$. Die Struktur wurde aus 1 510 Einkristallröntgendaten mittels direkter Methoden bestimmt; die Strukturverfeinerung ergab $R = 0.040$. Es ist ein Molekül in der asymmetrischen Einheit.

4-(2′-hydroxybenzoyl)morpholin, $C_{11}H_{13}NO_3$, orthorhombisch, $P2_12_12_1-D_2^4$; $a = 13.006$ (1), $b = 17.258$ (2), $c = 9.125$ (1) Å; $Z = 8$. Die Strukturparameter der isotypen Verbindung 4-(2′-hydroxythiobenzoyl)morpholin wurden als Startsatz für die Strukturverfeinerung benutzt. Es wurde $R = 0.054$ aus 1 900 Einkristallröntgendaten erreicht. Es befinden sich zwei Moleküle in der asymmetrischen Einheit.

In beiden Verbindungen konnten mittels Differenz-*Fourier*-Summierungen die Wasserstoffatome lokalisiert werden. Die intermolekularen Wasserstoffbrükken mit O . . . O-Distanzen von 2.649 (2) Å für das Piperidinderivat und 2.688 (3) bzw. 2.754 (3) Å für das Morpholinderivat sind typisch für mittlere Bindungsstärken.

Introduction

The crystal structure determinations of 1-(2′-hydroxythiobenzoyl)piperidine, $C_{12}H_{15}NOS$, *HTBP*, and 4-(2′-hydroxythiobenzoyl)morpholine, $C_{11}H_{13}NO_2S$, *HTBM*, by single crystal techniques showed, that—in the crystalline state—in both these compounds the molecules are combined by hydrogen bonds of intermediate bond strength. This intermolecular connection by the bridging hydrogen bonds results for both compounds in a one-dimensional atomic arrangement. These "formal chains" are combined by *van der Waals* contacts [1, 2].

The present paper deals with the crystal structure determination of the two compounds 1-(2′-hydroxybenzoyl)piperidine, $C_{12}H_{15}NO_2$, *HBP*, and 4-(2′-hydroxybenzoyl)morpholine, $C_{11}H_{13}NO_3$, *HBM*, by single crystal X-ray techniques at room temperature. The aim was to compare the conformation of the molecules itself as well as to compare the molecular connections by hydrogen bonds [cf. 3]. Single crystals of *HBP* and *HBM* were prepared by Mag. *E. Steinwender*, Institute of Organic Chemistry, University of Vienna, according to [4].

Table 1. *Summary of crystal data, data for intensity measurements and crystal structure refinement*

	HBP	*HBM*
a [Å]	9.529 (1)	13.006 (1)
b [Å]	10.001 (1)	17.258 (2)
c [Å]	11.652 (1)	9.125 (1)
ρ_{calc} [g cm^{-3}] and cell content	1.228; $Z = 4$	1.344; $Z = 8$
Space group	$Pbc2_1 - C_{2v}^5$	$P2_12_12_1 - D_2^4$
μMoKα [cm^{-1}]	0.48	0.59
Number of variables	196	375
R and R_w; $w = 1/[\sigma(F_0)]^2$	0.040 and 0.035	0.054 and 0.038
2 θ/ω scan mode with step width in [°]	0.030	0.035
Steps/reflection $+(\alpha_1,\alpha_2)$-splitting	45	45
Time/step	0.5 to 1.5	0.5 to 1.5
Range of data; max. sin Θ/λ [Å^{-1}]	0.70	0.70
Independent reflections	1 915	3 359
Reflections with $F_0 > 2\sigma_{F_0}$ resp. $3\sigma_{F_0}$	1 510	1 900
Secondary extinction: g [7]	1.8 (1) · 10^{-2}	n. d.

Stoe four-circle diffractometer; program system STRUCSY on an ECLIPSE S/140; graphite monochromatized MoKα radiation; 3 standard reflections; dimensions of the cut crystals $0.4 \times 0.4 \times 0.4 mm^3$

Experimental

Table 1 presents crystal data, data for intensity measurements, and finally obtained reliability indexes for the structure investigations of *HBP* and *HBM* by X-ray techniques. The lattice parameters were calculated from 48 (*HBP*) and 56 (*HBM*) accurate measured 2θ angles. The collected intensities were corrected for absorption (empirical φ scans of three reflections) as well as for *Lorentz* and polarization effects. For *HBP* the positions of the atoms C, O, and N were found by direct methods. For *HBM* the atomic coordinates given for the isotypic compound *HTBM* [2] were used in a starting set of refinement. Subsequent difference *Fourier* maps showed the positions of the hydrogen atoms. The atomic coordinates for all atoms in consideration of the anisotropic temperature parameters for the atoms C, O, and N as well as of isotropic ones for the hydrogen atoms were refined by least-squares techniques. Complex scattering functions for neutral atoms were taken from [5]. Table 2 gives the final structural parameters.

A trial to determine the absolute configuration failed for both structures.

Results and Discussion

The interatomic bond lengths and bond angles for the non-hydrogen atoms are compiled in Fig. 1. Also the conformation of the molecules and the numbering schemes of atoms are given in this figure. The mean values of the C–H distances at the atoms C(4) to C(7) (benzene ring) are 0.96 Å for *HBP* and 0.98 Å (molecule 1) resp. 0.94 Å (molecule 2) for *HBM*. At the piperidine resp. morpholine rings the average C–H dis-

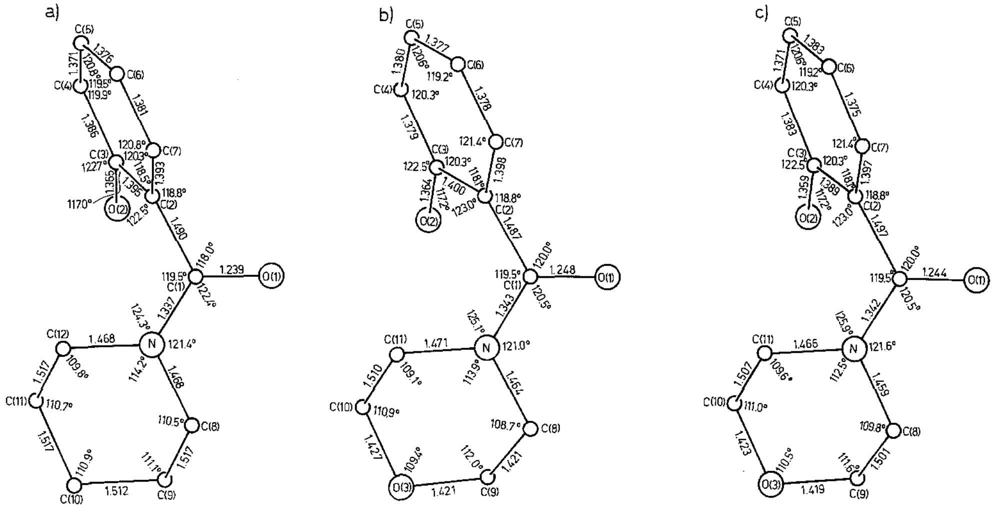

Fig. 1. The molecule of 1-(2′-hydroxybenzoyl)piperidine (*a*) as well as the two molecules of 4-(2′-hydroxybenzoyl)morpholine (*b* molecule 1; *c* molecule 2) in views perpendicular to the plane C(2), O(1), N. The numbering scheme used in text and tables, the interatomic distances [Å] (e.s.d.'s ⩽ 0.006 Å) and the bond angles [°] (e.s.d.'s ⩽ 0.5°) are given

Table 2. *Atomic coordinates and anisotropic temperature parameters (hydrogen atoms isotropic) for 1-(2′-hydroxybenzoyl)piperidine* (a) *and 4-(2′-hydroxybenzoyl)morpholine* (b *molecule 1*; c *molecule 2*). *The e.s.d.s are given in parentheses*

$$ATF = \exp\left(-2\pi^2 \sum_{i=1}^{3} \sum_{j=1}^{3} U_{ij} h_i h_j a_i^* a_j^* \right)$$

a)	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	0.1861(2)	0.4011(2)	0.5	0.0484(9)	0.0531(10)	0.0307(8)	0.0010(9)	-0.0014(9)	-0.0012(9)
C(2)	0.2818(2)	0.4442(2)	0.4059(2)	0.0424(9)	0.0480(10)	0.0378(9)	0.0039(8)	-0.0029(9)	0.0060(9)
C(3)	0.2871(2)	0.3793(2)	0.3001(2)	0.0454(9)	0.0542(11)	0.0390(10)	0.0028(9)	-0.0016(9)	0.0041(9)
C(4)	0.3838(2)	0.4182(2)	0.2177(2)	0.0560(11)	0.0727(14)	0.0430(11)	0.0048(11)	0.0101(10)	0.0085(11)
C(5)	0.4731(2)	0.5227(3)	0.2395(2)	0.0500(11)	0.0764(15)	0.0682(15)	-0.0011(12)	0.0145(12)	0.0185(14)
C(6)	0.4706(2)	0.5876(2)	0.3436(3)	0.0523(12)	0.0633(13)	0.0853(17)	-0.0130(11)	0.0005(14)	0.0068(14)
C(7)	0.3757(2)	0.5482(2)	0.4265(2)	0.0535(11)	0.0582(11)	0.0551(13)	-0.0015(10)	-0.0053(11)	-0.0002(10)
C(8)	-0.0455(2)	0.4040(2)	0.5907(2)	0.0562(11)	0.0869(16)	0.0432(11)	0.0075(11)	0.0098(11)	0.0032(12)
C(9)	-0.1704(2)	0.3241(2)	0.5481(2)	0.0560(11)	0.0763(15)	0.0773(16)	0.0028(11)	0.0139(13)	0.0114(14)
C(10)	-0.2420(2)	0.3940(2)	0.4491(3)	0.0523(11)	0.0754(15)	0.0867(17)	-0.0020(11)	-0.0070(13)	0.0056(15)
C(11)	-0.1382(2)	0.4246(2)	0.3539(2)	0.0626(12)	0.0765(15)	0.0570(13)	0.0096(12)	-0.0161(13)	-0.0009(12)
C(12)	-0.0156(2)	0.5055(2)	0.3995(2)	0.0497(10)	0.0735(14)	0.0509(12)	0.0069(10)	-0.0026(11)	0.0135(11)
N	0.0504(1)	0.4344(2)	0.4956(2)	0.0458(8)	0.0703(11)	0.0365(8)	0.0045(8)	0.0001(8)	0.0053(9)
O(1)	0.2379(1)	0.3407(2)	0.5827(1)	0.0597(8)	0.1101(12)	0.0473(8)	0.0097(9)	-0.0013(7)	0.0302(9)
O(2)	0.1946(2)	0.2768(2)	0.2833(2)	0.0746(9)	0.0728(10)	0.0446(8)	-0.0214(8)	0.0141(8)	-0.0135(8)

	x/a	y/b	z/c	U_{iso}		x/a	y/b	z/c	U_{iso}
H(1)	0.204(2)	0.240(3)	0.215(3)	0.090(9)	H(9b)	-0.137(2)	0.233(2)	0.525(2)	0.083(7)
H(4)	0.382(2)	0.375(2)	0.147(2)	0.052(5)	H(10a)	-0.322(2)	0.345(2)	0.416(2)	0.090(7)
H(5)	0.536(2)	0.550(2)	0.179(2)	0.074(7)	H(10b)	-0.283(2)	0.478(2)	0.484(2)	0.079(7)
H(6)	0.539(2)	0.660(2)	0.357(2)	0.078(7)	H(11a)	-0.183(2)	0.472(2)	0.285(2)	0.070(6)
H(7)	0.374(2)	0.590(2)	0.500(2)	0.072(6)	H(11b)	-0.101(2)	0.341(2)	0.317(2)	0.084(7)
H(8a)	0.009(2)	0.352(2)	0.649(2)	0.065(6)	H(12a)	-0.054(2)	0.595(2)	0.431(2)	0.074(7)
H(8b)	-0.082(2)	0.495(2)	0.621(2)	0.077(7)	H(12b)	0.056(2)	0.522(2)	0.338(2)	0.055(5)
H(9a)	-0.238(2)	0.316(2)	0.615(2)	0.075(7)					

b)	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	0.1813(2)	0.8833(2)	0.9798(4)	0.0480(19)	0.0336(17)	0.0465(21)	-0.0020(16)	-0.0033(18)	-0.0030(16)
C(2)	0.2520(2)	0.8161(2)	0.9646(3)	0.0429(17)	0.0375(17)	0.0361(16)	-0.0003(15)	0.0044(16)	0.0032(16)
C(3)	0.2337(2)	0.7547(2)	0.8679(3)	0.0406(17)	0.0478(18)	0.0354(17)	-0.0021(16)	0.0004(16)	-0.0015(16)
C(4)	0.2985(3)	0.6915(2)	0.8657(4)	0.0532(21)	0.0477(21)	0.0493(21)	0.0063(18)	0.0050(20)	-0.0111(19)
C(5)	0.3824(3)	0.6885(2)	0.9581(4)	0.0556(22)	0.0529(21)	0.0522(20)	0.0144(19)	0.0041(21)	0.0000(20)
C(6)	0.4027(3)	0.7484(2)	1.0535(4)	0.0546(22)	0.0654(24)	0.0487(21)	0.0055(21)	-0.0100(20)	0.0040(21)
C(7)	0.3368(3)	0.8108(2)	1.0583(4)	0.0596(21)	0.0433(19)	0.0423(19)	0.0011(18)	-0.0058(18)	-0.0036(16)
C(8)	0.0950(3)	0.9957(2)	0.8734(5)	0.0738(27)	0.0438(20)	0.0690(27)	0.0167(21)	-0.0051(26)	0.0027(21)
C(9)	0.0318(3)	0.9933(3)	0.7355(5)	0.0695(28)	0.0671(27)	0.0843(31)	0.0164(25)	-0.0102(28)	0.0144(27)
C(10)	0.1614(3)	0.9330(2)	0.6023(4)	0.0819(30)	0.0673(27)	0.0430(23)	0.0000(24)	0.0023(24)	0.0091(21)
C(11)	0.2327(3)	0.9324(2)	0.7328(4)	0.0582(23)	0.0621(25)	0.0472(23)	-0.0040(21)	0.0060(21)	0.0117(21)
N	0.1710(2)	0.9333(1)	0.8680(3)	0.0580(17)	0.0418(14)	0.0443(15)	0.0067(14)	0.0036(16)	0.0050(14)
O(1)	0.1334(2)	0.8937(1)	1.0966(2)	0.0758(17)	0.0466(12)	0.0456(14)	0.0092(13)	0.0156(14)	0.0017(12)
O(2)	0.1497(2)	0.7604(1)	0.7791(3)	0.0549(14)	0.0549(15)	0.0553(15)	0.0072(13)	-0.0090(13)	-0.0174(13)
O(3)	0.0939(2)	0.9981(1)	0.6077(3)	0.0807(17)	0.0690(15)	0.0640(16)	0.0047(15)	-0.0148(16)	0.0192(14)

	x/a	y/b	z/c	U_{iso}		x/a	y/b	z/c	U_{iso}
H(1)	0.140(3)	0.716(2)	0.728(4)	0.099(15)	H(9a)	-0.011(2)	0.942(2)	0.733(4)	0.064(11)
H(4)	0.283(2)	0.646(2)	0.807(3)	0.040(8)	H(9b)	-0.013(3)	1.041(2)	0.733(4)	0.085(12)
H(5)	0.427(2)	0.639(2)	0.954(4)	0.074(11)	H(10a)	0.196(3)	0.940(2)	0.511(4)	0.101(15)
H(6)	0.460(2)	0.746(2)	1.119(4)	0.069(11)	H(10b)	0.127(2)	0.882(2)	0.610(3)	0.043(9)
H(7)	0.346(2)	0.853(2)	1.123(3)	0.044(8)	H(11a)	0.281(2)	0.882(2)	0.730(4)	0.062(10)
H(8a)	0.130(2)	1.050(2)	0.875(4)	0.071(11)	H(11b)	0.276(3)	0.984(2)	0.725(4)	0.091(14)
H(8b)	0.049(2)	0.990(2)	0.965(4)	0.071(12)					

c)	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	0.0796(2)	0.6508(2)	0.4742(3)	0.0393(16)	0.0342(16)	0.0389(17)	0.0001(14)	0.0028(16)	0.0013(15)
C(2)	0.0142(2)	0.7219(2)	0.4631(3)	0.0411(17)	0.0357(16)	0.0337(16)	0.0051(15)	0.0001(16)	-0.0016(15)
C(3)	0.0423(2)	0.7857(2)	0.3795(3)	0.0440(17)	0.0399(16)	0.0320(16)	0.0050(15)	-0.0067(15)	-0.0017(15)
C(4)	-0.0160(3)	0.8526(2)	0.3852(4)	0.0550(20)	0.0377(18)	0.0422(20)	0.0078(17)	-0.0043(19)	0.0041(17)
C(5)	-0.1022(3)	0.8560(2)	0.4716(4)	0.0613(23)	0.0481(21)	0.0511(20)	0.0186(20)	-0.0083(21)	-0.0043(20)
C(6)	-0.1332(3)	0.7924(2)	0.5526(4)	0.0544(23)	0.0655(26)	0.0591(24)	0.0143(22)	0.0125(22)	-0.0049(21)
C(7)	-0.0745(3)	0.7262(2)	0.5492(4)	0.0597(23)	0.0476(21)	0.0506(21)	0.0005(20)	0.0121(20)	0.0031(19)
C(8)	0.1531(3)	0.5357(2)	0.3584(4)	0.0594(23)	0.0367(19)	0.0517(22)	0.0073(19)	-0.0025(20)	-0.0024(17)
C(9)	0.2232(3)	0.5384(2)	0.2278(4)	0.0530(21)	0.0451(21)	0.0742(27)	0.0091(18)	0.0016(22)	-0.0141(20)
C(10)	0.1065(3)	0.6128(2)	0.0943(4)	0.0742(27)	0.0572(23)	0.0374(20)	0.0125(22)	-0.0014(21)	-0.0089(19)
C(11)	0.0307(3)	0.6127(2)	0.2192(4)	0.0471(20)	0.0471(20)	0.0477(20)	0.0064(19)	-0.0096(18)	-0.0065(19)
N	0.0857(2)	0.6032(1)	0.3582(3)	0.0458(15)	0.0373(13)	0.0378(14)	0.0049(13)	-0.0041(13)	-0.0014(12)
O(1)	0.1261(2)	0.6371(1)	0.5905(2)	0.0662(14)	0.0444(12)	0.0375(12)	0.0106(12)	-0.0101(12)	-0.0003(10)
O(2)	0.1287(2)	0.7793(1)	0.2966(2)	0.0534(13)	0.0493(12)	0.0622(15)	0.0088(11)	0.0189(12)	0.0138(12)
O(3)	0.1671(2)	0.5441(1)	0.0949(2)	0.0690(15)	0.0603(14)	0.0494(14)	0.0083(13)	0.0034(13)	-0.0187(12)

	x/a	y/b	z/c	U_{iso}		x/a	y/b	z/c	U_{iso}
H(1)	0.134(2)	0.824(2)	1.233(4)	0.102(12)	H(9a)	0.271(2)	0.584(2)	0.239(3)	0.051(9)
H(4)	0.012(2)	0.897(2)	0.336(3)	0.046(9)	H(9b)	0.260(2)	0.488(2)	0.222(3)	0.056(9)
H(5)	-0.138(2)	0.905(2)	0.465(3)	0.042(9)	H(10a)	0.070(2)	0.612(2)	0.003(3)	0.065(11)
H(6)	-0.194(2)	0.798(2)	0.600(4)	0.069(12)	H(10b)	0.154(2)	0.661(2)	0.103(4)	0.083(13)
H(7)	-0.088(2)	0.683(2)	0.606(3)	0.065(11)	H(11a)	-0.007(2)	0.664(2)	0.224(3)	0.040(8)
H(8a)	0.113(2)	0.490(1)	0.355(3)	0.029(7)	H(11b)	-0.016(2)	0.569(2)	0.208(3)	0.050(10)
H(8b)	0.196(2)	0.535(2)	0.462(4)	0.064(10)					

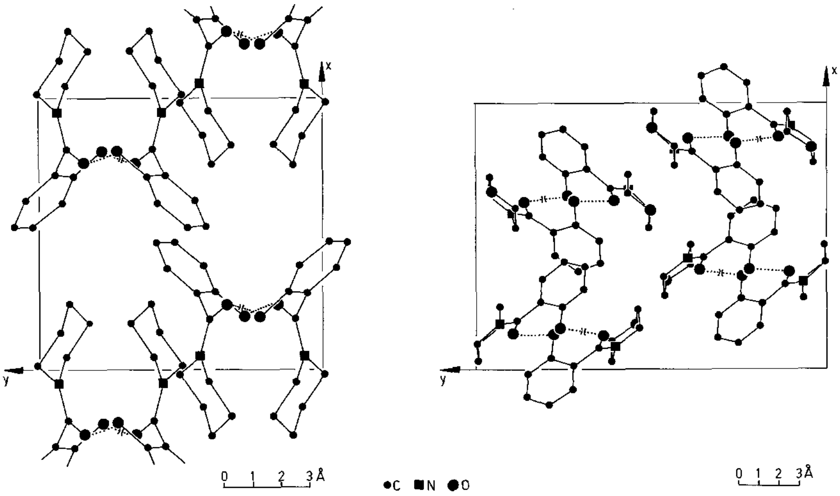

Fig. 2. The stacking of the molecules in projections parallel [001]. The intermolecular hydrogen bonding in 1-(2′-hydroxybenzoyl)piperidine (*a*) and 4-(2′-hydroxybenzoyl)morpholine (*b*) O(2)−H(1) . . . O(1) is dotted

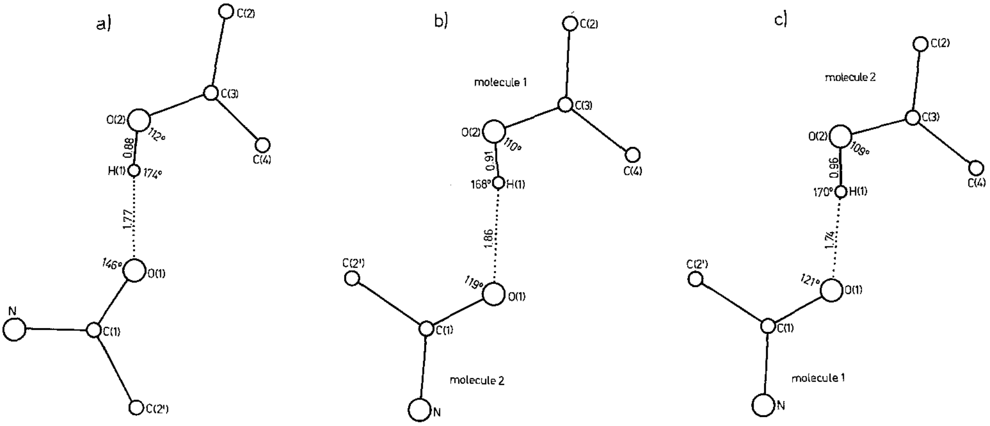

Fig. 3. Geometry of the O(2)−H(1) . . . O(1) bond in 1-(2′-hydroxybenzoyl)piperidine (*a*) and in 4-(2′-hydroxybenzoyl)morpholine (*b* molecule 1; *c* molecule 2). Interatomic distances are given in [Å], bond angles in [°]; the view is in all three cases perpendicular to the plane C(2), O(1) and N

tances are somewhat longer. For the three molecules they are equal to each other and amount to 1.02 Å. (All the e.s.d.'s for single C−H distances are $\leqslant$ 0.03 Å.) The differences of the C−H distances at the benzene ring resp. the piperidine and morpholine rings are caused by the different

Table 3. *Torsional angles (°). The e.s.d.'s are* $\leq 0.3°$. a) *HBP*; b) *HBM, molecule 1*; c) *HBM, molecule 2*

	a)	b)	c)
N(1) − C(1) − C(2) − C(3)	78.4	67.7	72.4
C(7)	105.6	117.1	112.5
O(1) − C(1) − C(2) − C(3)	105.1	113.5	108.1
C(7)	71.0	61.7	66.9
C(2) − C(1) − N(1) − C(8)	174.9	174.0	176.1
C(11)	−	6.7	2.2
C(12)	3.8	−	−
O(1) − C(1) − N(1) − C(8)	1.6	7.2	4.5
C(11)	−	172.1	177.3
C(12)	179.8	−	−
C(1) − C(2) − C(3) − C(4)	176.1	175.7	173.5
O(2)	3.2	3.8	5.4
C(7) − C(2) − C(3) − O(2)	179.3	179.0	179.7

chemical binding relationships at the C atoms. The values are in accordance with crystal chemical experience.

For *HBP* and *HBM* the question about the connection of the individual molecules was of special interest. As it was shown by the crystal structure analyses (Fig. 2), intermolecular O − H . . . O bonds connect the molecules in both compounds to extend chains parallel to [001]. Within the structure of *HBM* these hydrogen bonds are formed between the two crystallographically different molecules and in *HBP*, of course, between identic ones. The single chains are only combined by *van der Waals* contacts. The stackings of the molecules in *HBP* as well as in *HBM* are comparable with some respect to those determined for *HTBP* and *HTBM* [1, 2].

In detail the compound *HBM* is isotypic to the analogues sulphur derivate. The structure determined for *HBP* differs from that of *HTBP*: The sulphur compound *HTBP* has two molecules in the asymmetric unit and the unit cell therefore is doubled in one direction. The connection of the molecules by intermoleculare hydrogen bonds is almost the same in *HTBP* and *HBP*.

For a comparison of the conformation of the molecules in *HBP* and *HBM* some important torsional angles are given in Table 3. In all three molecules these angles are of the same magnitude. It can be presumed that the differences are predominantly caused by the stacking of the molecules.

The three crystallographic different bridging hydrogen bonds are all labelled as O(2) − H(1) . . . O(1). Their geometries are compiled in Fig. 3. Comparing the O(2) . . . O(1) distances with *van der Waals* radii suggests, that these hydrogen bonds are of intermediate bond strength [cf. 6]. The hydrogen bonds are weakly bent (up to 12°). For the angles H(1) . . . O(1) − C(1) there is a clear distinction between the two title compounds: In *HBP* this angle measures 146°, in *HBM* 119° resp. 121°. These differences are obviously caused by the stacking of the molecules. As a further result of this stacking the volume claimed by these molecules is 277.6 $Å^3$ for *HBP* but 256.0 $Å^3$ for *HBM*.

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