

# Hydrogen Bonding in 2-Hydroxy-benzhydrazide and 2-Hydroxy-thiobenzhydrazide. Structural and Spectroscopic Study

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**Summary.** X-ray structural data are reported for 2-hydroxy-benzhydrazide (monoclinic;  $P2_1/n - C_{2h}^5$ ;  $a = 10.569(2) \text{ \AA}$ ,  $b = 11.264(2) \text{ \AA}$ ,  $c = 6.074(1) \text{ \AA}$ ,  $\beta = 98.80(1)^\circ$ ;  $Z = 4$ ;  $R = 0.054$ ) and 2-hydroxy-thiobenzhydrazide (monoclinic;  $P2_1/n - C_{2h}^5$ ;  $a = 14.598(6) \text{ \AA}$ ,  $b = 5.975(2) \text{ \AA}$ ,  $c = 9.286(4) \text{ \AA}$ ,  $\beta = 107.80(3)^\circ$ ;  $Z = 4$ ;  $R = 0.044$ ). In both compounds the phenolic OH groups are intramolecularly hydrogen bonded to the (thio)carbonyl oxygens and sulfurs, respectively; the basic intermolecular association patterns consist of closed six-membered hydrogen bonded dimers which are connected via  $N-H \cdots NH_2$  hydrogen bonds. The spectral characteristics of the intramolecular  $O-H \cdots O/S$  hydrogen bonds (solid state  $\bar{\nu}(OH) = 2800/2700 \text{ cm}^{-1}$ ,  $CCl_4$  solution  $\bar{\nu}(OH) = 3035/3100 \text{ cm}^{-1}$ ,  $CDCl_3$  solution  $\delta(OH) = 11.62/10.56 \text{ ppm}$ ) are similar to those of corresponding primary and secondary amides. The solid state IR spectra are consistent with hydrogen bonded NH groups and free  $NH_2$  groups, the solution IR spectra indicate free  $NH_2$  as well as free NH groups.

**Keywords.** 2-Hydroxy-benzhydrazide; 2-Hydroxy-thiobenzhydrazide; Hydrogen bonding; X-ray structure; IR-NMR-spectra.

## Wasserstoffbrückenbindung in 2-Hydroxy-benzhydrazid und 2-Hydroxy-thiobenzhydrazid. Strukturelle und spektroskopische Untersuchungen

**Zusammenfassung.** Es werden Röntgenstrukturdaten für 2-Hydroxy-benzhydrazid (monoklin;  $P2_1/n - C_{2h}^5$ ;  $a = 10.569(2) \text{ \AA}$ ,  $b = 11.264(2) \text{ \AA}$ ,  $c = 6.074(1) \text{ \AA}$ ,  $\beta = 98.80(1)^\circ$ ;  $Z = 4$ ;  $R = 0.054$ ) und 2-Hydroxy-thiobenzhydrazid (monoklin;  $P2_1/n - C_{2h}^5$ ;  $a = 14.598(6) \text{ \AA}$ ,  $b = 5.975(2) \text{ \AA}$ ,  $c = 9.286(4) \text{ \AA}$ ,  $\beta = 107.80(3)^\circ$ ;  $Z = 4$ ;  $R = 0.044$ ) berichtet. In beiden Verbindungen bilden die phenolischen OH-Gruppen intramolekulare Wasserstoffbrücken mit den Carbonyl-Sauerstoff-, bzw. den Thiocarbonyl-Schwefel-Atomen. Das intermolekulare Assoziationsmuster wird durch  $N-H \cdots NH_2$  Wasserstoffbrücken bestimmt, die jeweils zwei Moleküle über sechszählige Ringe zu Dimeren verbinden. Die IR- und NMR-spektroskopischen Charakteristika der intramolekularen  $O-H \cdots O/S$  Wasserstoffbrücken (kristallin:  $\bar{\nu}(OH) = 2800/2700 \text{ cm}^{-1}$ ,  $CCl_4$ -Lösungen:  $\bar{\nu}(OH) = 3035/3100 \text{ cm}^{-1}$ ,  $CDCl_3$ -Lösungen:  $\delta(OH) = 11.62/10.56 \text{ ppm}$ ) sind sehr ähnlich zu denen entsprechender primärer und sekundärer Amide. Die Festkörper-IR-Spektren stehen in Einklang mit assoziierten NH- und freien  $NH_2$ -Gruppen, während die Lösungs-IR-Spektren auch auf das Vorliegen freier NH-Gruppen schließen lassen.

## Introduction

In the course of a systematic investigation of hydrogen bonding in 2-hydroxy-(thio)carbonyl compounds, 2-hydroxy-benzhydrazide (*HBH*) and 2-hydroxy-thio-benzhydrazide (*HTBH*) have been studied by structural and spectroscopic methods. An X-ray structural study of *HTBH* has already been published in the Ref. [1], however, the quality was very poor and from IR spectroscopic data (see below) the proposed thioimin form,  $-\text{C}(\text{SH})=\text{N}-\text{NH}_2$ , seemed highly questionable.

In the present paper X-ray structures, solid state and solution IR spectra, and solution NMR spectra are reported for *HBH* and *HTBH*. Both, structural and spectroscopic data are briefly discussed mainly with respect to the intermolecular and intramolecular hydrogen bond patterns.

## Experimental Part

### 2-Hydroxy-benzhydrazide (*HBH*)

Commercially available material (Aldrich Chemie); recrystallization from  $\text{CHCl}_3$ ; m.p. 147–149 °C.  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ): 4.09 (s,  $\text{NH}_2$ ), 6.86 (t, 1H), 7.03 (d, 1H), 7.35 (d, 1H), 7.42 (d, 1H), 7.59 (broad, NH), 11.77 (s, OH).  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ ): 112.9, 118.7, 124.9, 134.6, 161.1, 170.6.

**Table 1.** Summary of crystal data and experimental details for intensity measurements and crystal structure refinements

	<i>HBH</i>	<i>HTBH</i>
Formula	$\text{C}_7\text{H}_8\text{N}_2\text{O}_2$	$\text{C}_7\text{H}_8\text{N}_2\text{OS}$
Crystal size [mm]	0.7·0.4·0.8	0.1·0.6·0.2
Space group	$\text{P}2_1/\text{n} - \text{C}_{2\text{h}}^5$	$\text{P}2_1/\text{n} - \text{C}_{2\text{h}}^5$
$a$ [Å]	10.569(2)	14.598(6)
$b$ [Å]	11.264(2)	5.975(2)
$c$ [Å]	6.074(1)	9.286(4)
$\beta$ [°]	98.80(1)	107.01(3)
$Z$	4	4
$V$ [Å <sup>3</sup> ]	714.6	774.5
$\rho_{\text{calc}}$ [g·cm <sup>-3</sup> ]	1.424	1.443
$\mu(\text{MoK}\alpha)$ [cm <sup>-1</sup> ]	0.66	3.04
Step width [°]	0.03	0.03
$2\Theta_{\text{max}}$ [°]	55	55
Range of $h, k, l$	$\pm h, k, \pm l$	$\pm h, k, \pm l$
Measured reflections	4360	4958
Unique reflections	2080	2280
$R_{\text{int}}$ [ $\sum  I - \langle I \rangle  / \sum I$ ]	0.058	0.064
Reflect. with $F_0 > 4\sigma(F_0)$	1450	1415
Number of variables	133	133
$R$	0.054	0.044
$R_w$	0.048	0.044
$(\Delta/\sigma)_{\text{max}}$	<0.001	<0.001
$(\Delta\rho)_{\text{max/min}}$	0.20/0.29	0.27/0.23

*2-Hydroxy-thiobenzhydrazide (HTBH)*

Preparation according to Ref. [2]; recrystallization from benzene/ligroine; yellow crystals; m.p. 100–101 °C. <sup>1</sup>H-NMR (CDCl<sub>3</sub>): 4.69 (s, NH<sub>2</sub>), 6.85 (t, 1H), 7.02 (d, 1H), 7.24 (d, 1H), 7.36 (t, 1H), 8.64 (broad, NH), 10.55 (OH). <sup>13</sup>C-NMR (CDCl<sub>3</sub>): 119.3, 119.7, 120.9, 124.1, 133.4, 157.9, 191.7 (broad; halfwidth ≈ 100 Hz).

*Spectra*

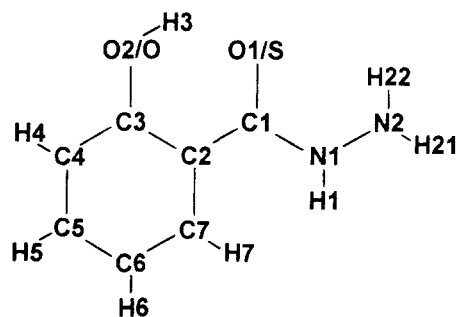
NMR-spectra were measured with a Bruker WM-250 or a Bruker AM-400 spectrometer. IR-spectra (KCl pellets, nujol and fluorolube mulls, CCl<sub>4</sub> solutions) were measured with a Perkin Elmer 1740 spectrometer.

*X-Ray Diffraction*

Well developed single crystals of *HBH* and *HTBH* were used for the determination of lattice constants, Laue symmetries and extinction rules by oscillation and Weissenberg film methods. Three dimensional single crystal data were collected at room temperature with a Stoe four circle diffractometer, AED2 (program system STRUCSY [3]), and corrected for Lorentz and polarisation effects by standard methods. The positions of the non-hydrogen atoms were determined by direct method strategies, the hydrogen atoms were located from difference Fourier maps. The atomic coordinates and the displacement factors (isotropic as well as anisotropic for non-hydrogen atoms) were refined by least squares techniques, in which the complex scattering factors were taken from standard tables [4]. A summary of crystal data and of experimental details concerning the intensity measurements and the crystal structure refinements are given in Table 1.

**Results and Discussion***Structures*

The atom numberings used in this paper are shown in Scheme 1. Atomic coordinates and temperature factors are given in Tables 2 and 3 for *HBH* and *HTBH* respectively\*. Selected bond distances and angles are summarised in Table 4. In order to account for the uncertainties and systematic errors concerning the location of hydrogen atoms by X-ray diffraction methods, “corrected” H···Y bond distance [5]



**Scheme 1.** Atom numbering of the title compounds *HBH/HTBH*

\* Structure factor tables are available from the authors upon request

**Table 2.** 2-Hydroxy-benzhydrazide: atomic coordinates and temperature factors with esd's in parentheses

	$x/a$	$y/b$	$z/c$	$U_{11}/U_{iso}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
C1	0.3421(1)	0.0515(1)	0.8485(2)	0.0452(7)	0.0373(6)	0.0410(6)	0.0047(6)	0.0049(5)	0.0043(6)
C2	0.3825(1)	0.1586(1)	0.9834(2)	0.0453(6)	0.0369(6)	0.0387(6)	0.0059(6)	0.0066(5)	0.0025(6)
C3	0.3205(1)	0.1836(1)	1.1682(2)	0.0458(7)	0.0466(7)	0.0432(7)	0.0110(7)	0.0097(6)	0.0044(6)
C4	0.3589(2)	0.2805(2)	1.3038(2)	0.0601(8)	0.0564(9)	0.0463(8)	0.0178(8)	0.0107(7)	-0.0058(7)
C5	0.4559(2)	0.3528(1)	1.2569(3)	0.0674(9)	0.0464(8)	0.0570(9)	0.0064(9)	0.0013(7)	-0.0164(8)
C6	0.5177(2)	0.3307(1)	1.0739(3)	0.0622(10)	0.0483(6)	0.0618(9)	-0.0074(9)	0.0113(8)	-0.0074(8)
C7	0.4809(1)	0.2339(1)	0.9412(2)	0.0569(8)	0.0450(7)	0.0471(8)	-0.0022(7)	0.0142(7)	-0.0038(7)
O1	0.2237(1)	0.1152(1)	1.2185(2)	0.0624(6)	0.0711(7)	0.0634(7)	-0.0051(6)	0.0286(8)	-0.0034(6)
O2	0.2513(1)	-0.0114(1)	0.8859(2)	0.0638(6)	0.0506(6)	0.0679(7)	-0.0141(6)	0.0206(6)	-0.0066(6)
N1	0.4072(1)	0.0231(1)	0.6842(2)	0.0565(6)	0.0428(6)	0.0458(6)	-0.0047(6)	0.0108(5)	-0.0110(5)
N2	0.3752(1)	-0.0781(1)	0.5490(2)	0.0652(8)	0.0444(6)	0.0532(7)	-0.0030(7)	0.0060(6)	-0.0132(6)
H1	0.4748(17)	0.0645(15)	0.6515(24)	0.0575(47)					
H21	0.2933(17)	-0.0669(16)	0.4826(29)	0.0718(50)					
H22	0.3676(19)	-0.1401(18)	0.6390(31)	0.0852(61)					
H3	0.2123(20)	0.0574(19)	1.1169(33)	0.0878(72)					
H4	0.3188(15)	0.2930(15)	1.4339(25)	0.0597(46)					
H5	0.4814(20)	0.4160(21)	1.3578(34)	0.0882(62)					
H6	0.5842(17)	0.3815(15)	1.0421(27)	0.0615(45)					
H7	0.5267(16)	0.2175(15)	0.8122(27)	0.0648(47)					

Table 3. 2-Hydroxy-thiobenzhydrazide: atomic coordinates and temperature factors with esd's in parentheses

	$x/a$	$y/b$	$z/c$	$U_{11}/U_{iso}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
C1	0.9482(1)	0.3511(3)	0.7701(2)	0.0460(8)	0.0561(10)	0.0448(11)	0.0086(9)	0.0071(7)	-0.0001(8)
C2	1.0290(1)	0.5053(3)	0.7825(2)	0.0472(9)	0.0496(9)	0.0453(10)	0.0065(7)	0.0092(7)	-0.0005(8)
C3	1.0251(1)	0.6880(3)	0.6864(2)	0.0624(10)	0.0589(11)	0.0517(11)	0.0087(9)	0.0055(8)	0.0081(9)
C4	1.1029(2)	0.8294(4)	0.7039(3)	0.0828(14)	0.0639(13)	0.0603(13)	-0.0074(11)	0.0106(11)	0.0142(11)
C5	1.1866(1)	0.7913(3)	0.8162(2)	0.0678(12)	0.0651(12)	0.0609(13)	-0.0141(10)	0.0167(10)	0.0005(11)
C6	1.1936(1)	0.6115(3)	0.9115(2)	0.0576(10)	0.0708(13)	0.0592(13)	-0.0079(9)	0.0043(9)	0.0068(10)
C7	1.1165(1)	0.4737(3)	0.8955(2)	0.0548(10)	0.0615(11)	0.0555(12)	-0.0001(8)	0.0031(8)	0.0148(9)
O	0.9462(1)	0.7426(3)	0.5729(2)	0.0827(10)	0.0905(10)	0.0903(11)	-0.0060(8)	-0.0216(8)	0.0459(9)
S	0.8372(1)	0.3853(1)	0.6528(1)	0.0493(3)	0.0935(4)	0.0786(5)	0.0024(2)	-0.0096(3)	0.0039(2)
N1	0.9652(1)	0.1745(3)	0.8598(2)	0.0412(7)	0.0648(9)	0.0676(11)	-0.0023(7)	-0.0010(7)	0.0157(8)
N2	0.8961(1)	0.0133(3)	0.8689(3)	0.0508(9)	0.0674(11)	0.0760(13)	-0.0084(8)	0.0071(8)	0.0142(10)
H1	1.0208(16)	0.1525(34)	0.9255(25)	0.0925(74)					
H21	0.8836(18)	-0.0607(45)	0.7833(29)	0.1185(115)					
H22	0.8354(21)	0.0899(40)	0.8680(29)	0.1368(109)					
H3	0.9028(19)	0.6397(40)	0.5751(30)	0.1094(100)					
H4	1.0960(14)	0.9496(35)	0.6275(23)	0.0846(65)					
H5	1.2359(14)	0.8798(32)	0.8242(24)	0.0788(68)					
H6	1.2532(15)	0.5785(32)	0.9964(26)	0.0973(70)					
H7	1.1211(15)	0.3560(34)	0.9629(26)	0.0890(73)					

**Table 4.** Selected intra- and intermolecular bond distances [ $\text{\AA}$ ] and angles [ $^\circ$ ] with esd's in parentheses (corrected hydrogen bond distances and angles, see text, are given in brackets)

	<i>HBH</i>	<i>HTBH</i>
C1–O2/S	1.242(1)	1.679(1)
C1–N1	1.335(1)	1.322(2)
C1–C2	1.484(1)	1.474(2)
N1–N2	1.415(1)	1.415(2)
N1–H1	0.901(17) [1.03]	0.871(23) [1.03]
H1 $\cdots$ N2'	2.147(16) [2.04]	2.178(23) [2.05]
N1 $\cdots$ N2'	2.945(1)	2.953(2)
C2–C3	1.412(1)	1.401(2)
C7–C2	1.396(1)	1.409(2)
C3–O1/O	1.352(1)	1.354(2)
O1/O–H3	0.893(21) [1.00]	0.887(26) [1.00]
H3 $\cdots$ O2/S	1.708(20) [1.61]	2.037(25) [1.93]
O1 $\cdots$ O2/S	2.526(1)	2.886(1)
O2/S–C1–N1	120.1(1)	118.3(1)
O2/S–C1–C2	121.9(1)	118.3(1)
N1–C1–C2	118.1(1)	116.8(1)
C1–N1–N2	121.5(1)	125.1(2)
N1–H1 $\cdots$ N2'	147.3(13) [145]	148.0(20) [146]
C1–C2–C3	118.2(1)	123.7(2)
C1–C2–C7	118.3(1)	116.0(2)
C2–C3–O1/O	121.7(1)	123.4(2)
O1/O–H3 $\cdots$ O2/S	151.1(16) [149]	159.6(30) [159]
O2/S–C1–C2–C3	$\pm 4.4$	$\pm 6.7$
O2/S–C1–C2–C7	$\mp 177.5$	$\mp 173.3$
N1–C1–C2–C3	$\mp 175.1$	$\mp 174.0$
N1–C1–C2–C7	$\pm 3.0$	$\pm 6.0$

are also given in Table 4, which have been calculated by adopting O–H distances to 1.00  $\text{\AA}$  and N–H distances to 1.03  $\text{\AA}$ . Stereoviews of the unit cells are shown in Figs. 1 and 2.

The two title compounds, *HBH* and *HTHB*, exhibit a number of common structural features. Both, the hydrazide and the thiohydrazide molecules assume the *Z*-configuration which is usually preferred with hydrazides and with secondary amides. Except of the terminal  $\text{NH}_2$  groups, the molecules are fairly planar with dihedral angles of less than  $\pm 7^\circ$ . Besides the C–O/S bond distances, distances and angles within the hydrazide and thiohydrazide moieties, C2–C1(O2/S)–N1–N2, are very similar (Table 4) and inspection of the Cambridge Crystallographic Data Base (some 40 entries for hydrazide and 1 entry for thiohydrazide, including organometallic complexes) shows, that the values closely correspond to those reported for other (thio)hydrazides.

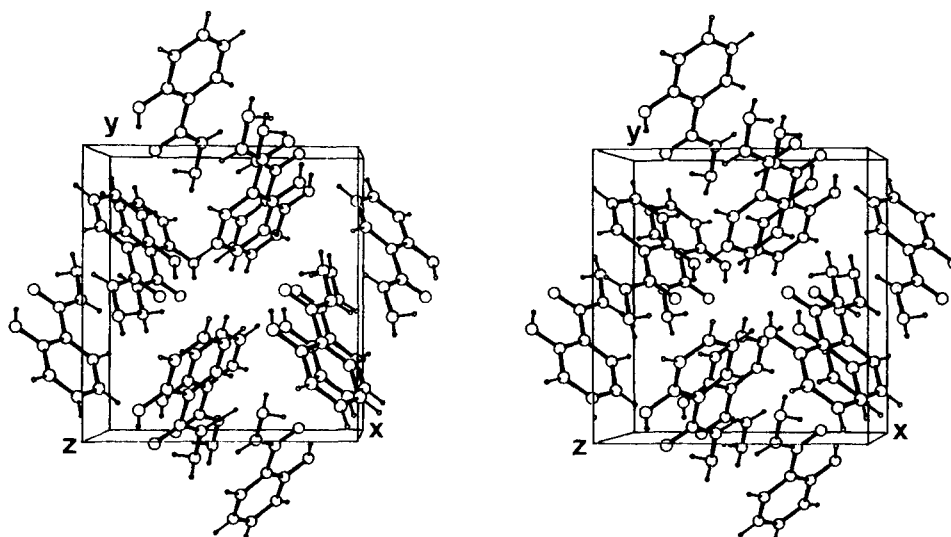


Fig. 1. Stereoview of the unit cell of 2-hydroxy-benzhydrazide (*HBH*)

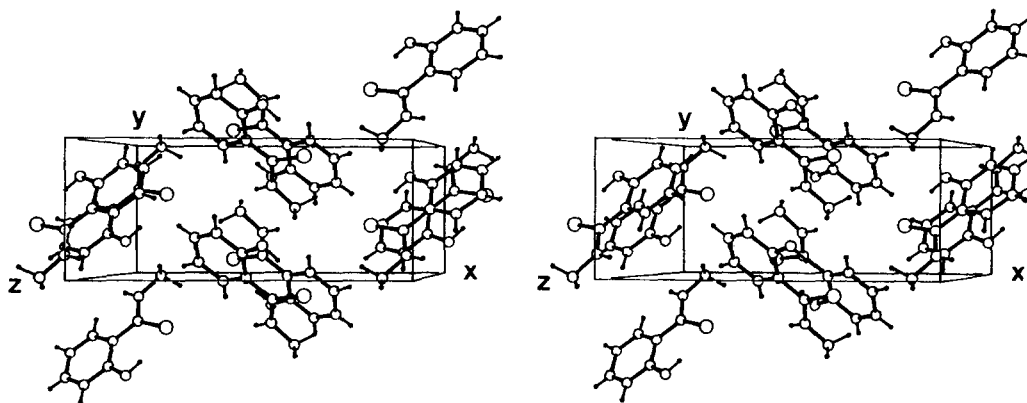


Fig. 2. Stereoview of the unit cell of 2-hydroxy-thiobenzhydrazide (*HTBH*)

The structure of *HTBH* was found to be quite different to that reported in Ref. [1], not only with regard to the proposed thioimin form, but also to the molecular packing arrangement. Polymorphism could be a possible reason for the apparent differences, although our melting point agrees with the reported one and we failed in obtaining a second modification.

In both title compounds the OH groups are intramolecularly hydrogen bonded to the carbonyl oxygens and the thiocarbonyl sulfurs, respectively. The O $\cdots$ O/S distances (2.53/2.89 Å) are similar to those of primary (2.51/2.90–2.92 Å [6]) and secondary (2.51/2.94 Å [7]) 2-hydroxy-(thio)benzamides. As is quite generally observed with hydrazides, the strongest intermolecular interaction arises from hydrogen bonding of the N1–H1 groups. In both title compounds each two alternating enantiomers are connected via two intermolecular N1–H1 $\cdots$ N2' bonds (N1 $\cdots$ N2' = 2.95 Å); centrosymmetric closed six-membered hydrogen bonded dimers

result. In both title compounds neither the OH groups (closest intermolecular contacts:  $\text{H3}\cdots\text{N2}' = 3.29 \text{ \AA}$ ;  $\text{H3}\cdots\text{O}' = 3.71 \text{ \AA}$ ) nor the terminal  $\text{NH}_2$  groups (closest intermolecular contacts:  $\text{H21}\cdots\text{O1}' = 2.61 \text{ \AA}$  in *HBH*;  $\text{H21}\cdots\text{O}' = 2.66 \text{ \AA}$  and  $\text{H22}\cdots\text{S}' = 2.76 \text{ \AA}$  in *HTBH*) are engaged in intermolecular hydrogen bonds. Among the hydrazide structures available in the reference, a similar hydrogen bond pattern (closed six-membered dimers) was found to be realized only in isonicotine-hydrazide ( $\text{N1}\cdots\text{N2}' = 2.97 \text{ \AA}$  [8]).

### Spectra

Survey IR spectra are shown in Fig. 3. In agreement with the structural data reported above, the thioimino form proposed for *HTBH* in Ref. [1] can confidently be ruled out from the spectral data for both, the solid state and the solution, e.g. from the lack of a corresponding  $\nu(\text{SH})$  band.

Expectedly, the spectral data characteristic of the intramolecular  $\text{O}-\text{H}\cdots\text{O}/\text{S}$  hydrogen bonds are close to those of the corresponding primary and secondary

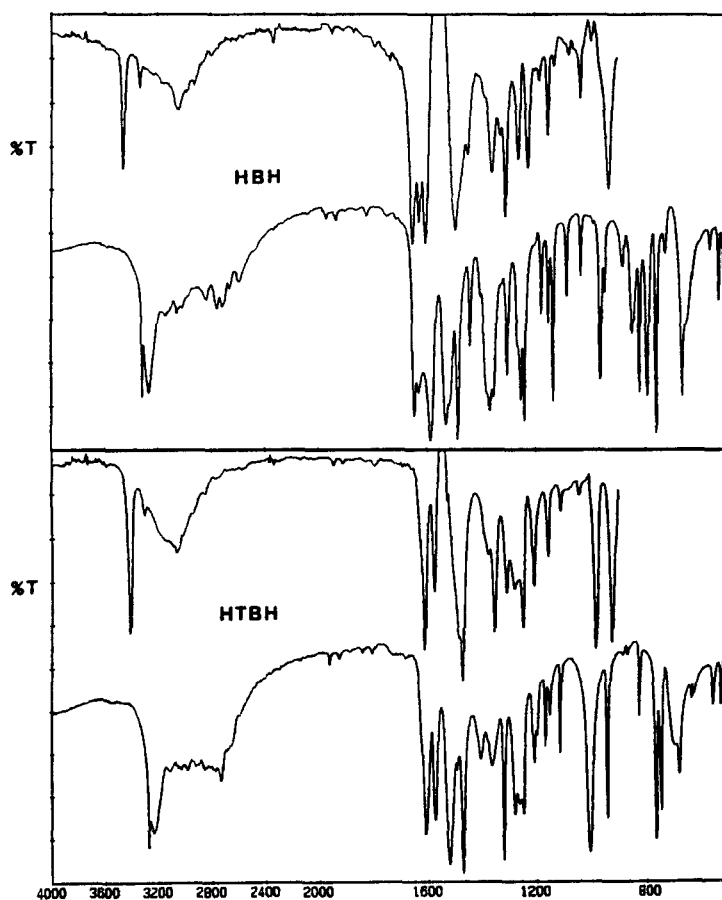


Fig. 3. Survey IR spectra of 2-hydroxy-benzhydrazide (*HBH*) and 2-hydroxy-thiobenzhydrazide (*HTBH*) (lower spectra: KCl pellets; upper spectra:  $\text{CCl}_4$  solutions, 1 mm pathlength)



(thio)amides [7]. The  $\bar{\nu}(\text{OH})$  frequencies measure about  $2800\text{ cm}^{-1}$  (*HBH*) and  $2750\text{ cm}^{-1}$  (*HTBH*) for the solids, and  $3035\text{ cm}^{-1}$  (*HBH*) and  $3100\text{ cm}^{-1}$  (*HTBH*) for  $\text{CCl}_4$  solutions. The  $\text{CDCl}_3$  solution NMR shifts of the phenolic OH groups measure 11.6 and 10.5 ppm, respectively, and fit well to the  $\bar{\nu}(\text{OH})$  vrs.  $\delta(\text{OH})$  correlations reported in Ref. [9]. A more detailed comparison between spectral and structural data of  $\text{O}-\text{H}\cdots\text{O}/\text{S}$  bonds in various 2-hydroxy-(thio)carbonyl compounds will be given elsewhere [10].

The frequencies of the  $\nu(\text{NH})$  bands increase by about  $200\text{ cm}^{-1}$  when going from solids [ $3262\text{ cm}^{-1}$  (*HBH*),  $3232\text{ cm}^{-1}$  (*HTBH*)] to solutions [ $3467\text{ cm}^{-1}$  (*HBH*),  $3414\text{ cm}^{-1}$  (*HTBH*)] and the halfwidths become significantly reduced, which is consistent with a change from the  $\text{N1}-\text{H1}\cdots\text{N2}$  type associated dimers in the solids to non-associated monomers in solution. Only one rather weak IR band due to the terminal  $\text{NH}_2$  group is observed for both compounds, as it is also known from other (thio)hydrazides, e.g. from (thio)benzhydrazide. On dissolving the bands become shifted only by about  $25\text{ cm}^{-1}$  [solids:  $3319\text{ cm}^{-1}$  (*HBH*),  $3275\text{ cm}^{-1}$  (*HTBH*); solutions:  $3340\text{ cm}^{-1}$  (*HBH*),  $3305\text{ cm}^{-1}$  (*HTBH*)] in agreement with free  $\text{NH}_2$  in both, in the solids and in the solutions.

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