

Synthesis, characterization, and X-ray structure of (1*E*,5*E*)-1,5-bis(1-hydroxypropan-2-ylidene)carbonohydrazide

Cheikh Ndoye^a, Amar Diop^a, Grégory Excoffier^b, Mamadou Sidibé^a, Ousmane Diouf^a, Mohamed Gaye^{a,*}

^aDepartment of Chemistry, University Cheikh Anta Diop, Dakar, 10700, Sénégal

^bAix Marseille Univ, CNRS, Centrale Marseille, FSCM, Spectropole, Marseille, France

Abstract: A new symmetrical carbonohydrazide derivative, (1*E*,5*E*)-1,5-bis(1-hydroxypropan-2-ylidene)carbonohydrazide (**I**), was synthesized by one-step condensation reaction. The structure of this new compound was confirmed by elemental analysis, infrared, UV-Visible and ¹H and ¹³C NMR spectroscopy techniques. The compound (**I**) crystallizes in the orthorhombic space group *Pbca* with the following unit cell parameters *a* = 11.5576 (1) Å, *b* = 12.6971 (1) Å, *c* = 13.5932(2) Å, *V* = 1994.78(4) Å³, *Z* = 8, *R*₁ = 0.043 and *wR*₂ = 0.131. In the structure numerous intermolecular hydrogen bonds which consolidate the structure into three dimensional network were observed.

Keywords: Carbohydrazide, 2-hydroxyacetone, NMR, UV-Visible, X-ray.

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I. Introduction

Carbonohydrazides and thiocarbonohydrazides are very important precursors of Schiff's base [1-6]. Indeed, they are symmetric and can make it possible to generate symmetric or asymmetric Schiff bases depending on the stoichiometry and the operating conditions of the reaction [3,7,8]. Several studies have been carried out in recent years to prepare carbono or thiocarbono ligands and their complexes. The molecular materials derived from these types of ligands exhibit original structures and important physical and / or biological properties. Indeed, these Schiff bases can be presented with several types of conformations allowing them to react with the metal ions so as to give different structures. The topologies and versatility of these ligands based on carbohydrazide or thiocarbonohydrazide have made it possible to develop mono or polynuclear complexes with very original structures, the most remarkable of which have grid structures [4, 9-13]. Several Schiff bases obtained by condensation reaction of carbono or thiocarbonohydrazide with keto-precursors exhibit biological activity and are used as antimicrobial [14], antidepressant [15], anti-inflammatory [16], anti-tumor [17] or antioxidants [18]. Many complexes of these Schiff bases show important physical properties in the fields of magnetism [19], luminescence [20] or SMMs [21].

Our research group has already studied these types of Schiff bases and them derives [3,7,8,13]. Continuing our attempt to develop new Schiff bases from carbonohydrazide, we report in this work the synthesis and structure determination of the compound (1*E*,5*E*)-1,5-bis(1-hydroxypropan-2-ylidene)carbonohydrazide (**I**) derived from 1-hydroxypropan-2-one.

II. Experimental

2.1 Starting materials and instrumentations

Carbohydrazide, and 1-hydroxypropan-2-one were of analytical reagent grade and were obtained from Sigma-Aldrich Company. All used solvents were of analytical reagent grade and were used directly without further purification. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on an FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000-400 cm⁻¹ region. The UV-Visible spectrum was recorded on a Perkin Elmer Lambda UV-Vis spectrophotometer. The ¹H and ¹³C NMR spectra were recorded in DMSO-*d*₆ on a Bruker 500 MHz spectrometer at room temperature using TMS as an internal reference.

* Corresponding author : mohamedl.gaye@ucad.edu.sn

2.2 Synthesis of the ligand (1E,5E)-1,5-bis(1-hydroxypropan-2-ylidene)carbonohydrazide (I)

In a round bottomed flask, carbonohydrazide (2.50 g, 27.75 mmol) was introduced with methanol (30 mL). 1-Hydroxy-propan-2-one (4.12 g, 55.50 mmol) dissolved in 10 mL of the same solvent was added. After four hours under reflux, the yellow precipitate formed after cooling to room temperature was filtered off and washed with cold methanol (2 x 20 mL) and diethyl ether (2 x 20 mL). The resulting solid was dried in the desiccator under anhydrous P₂O₅. The solid was recrystallized in hot ethanol and left at room temperature. Slow evaporation of the solvent gave yellow crystals after two days. Yield: 86 %; m.p. 605 K. Anal. Calc. for [C₇H₁₄N₄O₃] (%): C, 41.58; H, 6.98, N, 27.71. Found: C, 41.56; H, 7.01, N, 27.68. Selected IR data (cm⁻¹, KBr pellet): 3100, 2934, 1679, 1651, 1271. ¹H-NMR (DMSO-d₆) δ: 1.83 (s, 6H, -CH₃); 3.98 (s, 4H, -CH₂-); 5.02 (s, 1H, -OH); 9.37 (s, 2H, H-C=N). ¹³C-NMR (DMSO-d₆) d: 13.27 (-CH₃); 65.84 (C-OH); 152.28 (C=N); 153.08 (C=O). UV-Visible (MeOH, λ_{max}, nm): 267; 300.

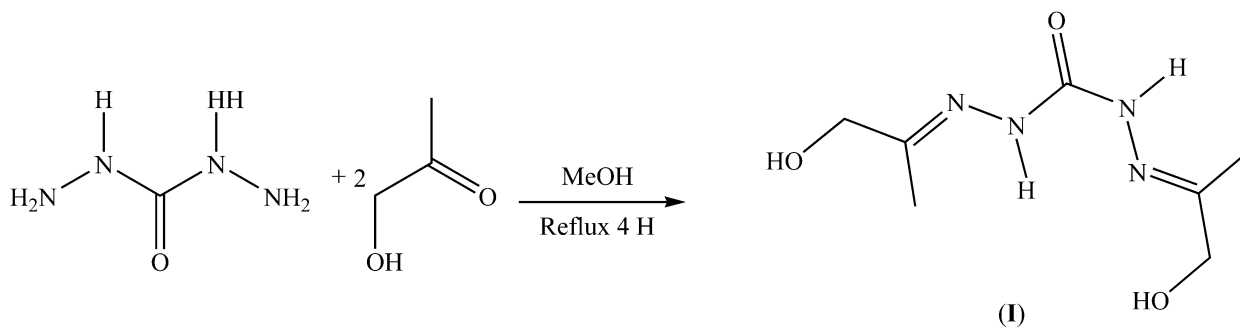
2.3 Crystal structure determination

Crystals suitable for X-diffraction, of the reported compound, were grown by slow evaporation of MeOH solution of the complex. Details of the X- rays crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an ENRAF NONIUS Kappa CCD diffractometer with graphite monochromatized CuKα radiation (λ = 1.54187 Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL [22]. The structures were solved by direct methods which revealed the position of all non- hydrogen atoms. All the structures were refined on F² by a full- matrix least- squares procedure using anisotropic displacement parameters for all non- hydrogen atoms [23]. The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH₃ groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP- 3 [24].

III. Results and discussion

3.1 General study

Recently our research group has reported the synthesis, characterization, and antioxidant activities of novels carbonohydrazone derivatives [7,8]. In this manuscript, we have discussed a simple procedure to synthesize the title compound and characterized it by elemental analysis, various spectroscopic methods, and single crystal X- ray diffraction. The presence of amidic bond in the symmetrical Schiff base dicarbonohydrazone (1E,5E)-1,5-bis(1-hydroxypropan-2-ylidene)carbonohydrazide can lead to keto-enol tautomer.



Scheme 1. Synthetic scheme for the compound (I)

The ligand H₄L was prepared by a facile condensation of carbonohydrazide and 1-hydroxypropan-2-one in methanol in 1/2 ratio (**Scheme 1**). The resulting compound is quantitatively yielded (86 %). The elemental analyses results is in accordance with the chemical formulae obtained from X-ray diffraction study. The infrared spectrum (**Figure 1**) of this ligand reveals an intense band at 1679 cm⁻¹ attributed to ν(C=O) while the medium intensity band located at 1651 cm⁻¹ is assigned the elongation vibration ν(C=N). At 3100 cm⁻¹, a band of medium intensity appears due to the elongation of the N-H bond. At 1271 cm⁻¹ there is a low intensity band which represents the O-C stretching vibration.

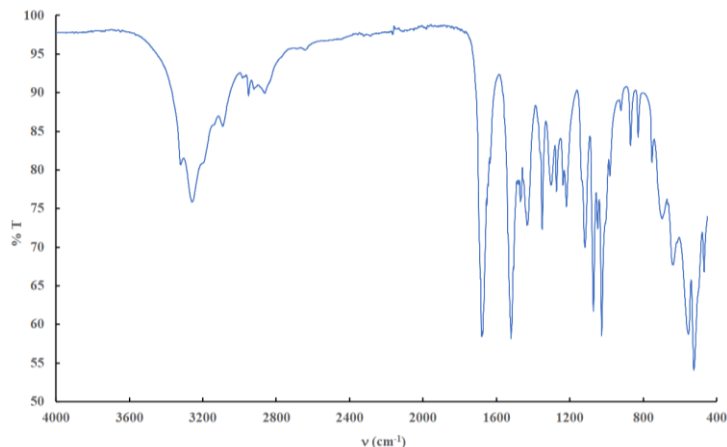


Figure 1. Infrared spectrum of the compound (**I**).

The ^1H and ^{13}C NMR (**Figure 2** and **3**) spectra of the H_4L ligand was recorded in DMSO ($\text{dms}\text{-d}_6$). The signals at 9.37 ppm representing two protons, is due to the two NH of the hydrazone moieties. The -OH protons of the alcohol moieties are pointed at 5.02 ppm. Signals at 1.83 ppm and 3.98 ppm are respectively assigned to the protons of the methyl groups ($\text{CH}_3\text{-C=N}$) and the protons of the methylene groups ($\text{-CH}_2\text{-O}$). The ^{13}C NMR spectrum of the H_4L ligand shows signals at 155.08 ppm (C=O), 152.28 ppm (C=N), 65.84 ppm ($\text{-CH}_2\text{-O}$) and 13.27 ppm ($\text{CH}_3\text{-C=N}$). The signal $\delta_{\text{C=O}}$ at 155.08 ppm combined to the signal δ_{HN} at 9.37 ppm are indicative of the presence of the compound (**I**) in its keto-form.

The electronic spectrum of the compound (**I**) was recorded in methanol over the range 200–1000 nm (**Figure 4**). The Schiff base exhibits two bands centred around 267 and 300 nm in the UV–visible region. The first band corresponds to intra ligand $\pi\rightarrow\pi^*$ transitions and the second band is associated with $n\rightarrow\pi^*$ transitions of the azomethine functional groups [25].

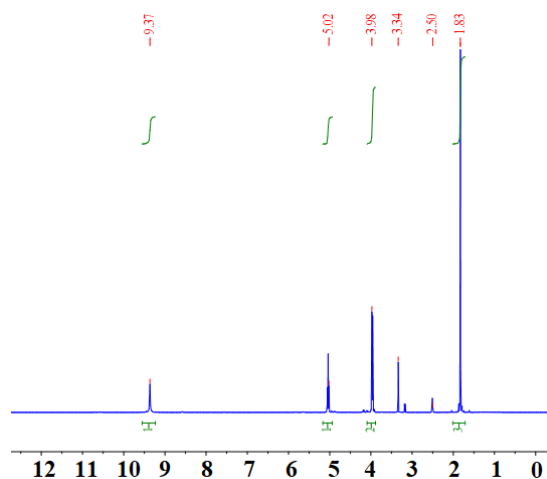


Figure 2. ^1H NMR of compound (**I**).

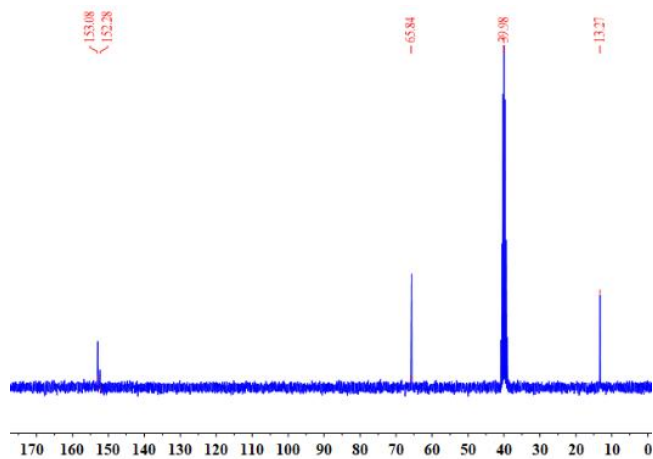


Figure 3. ^{13}C NMR of compound (I).

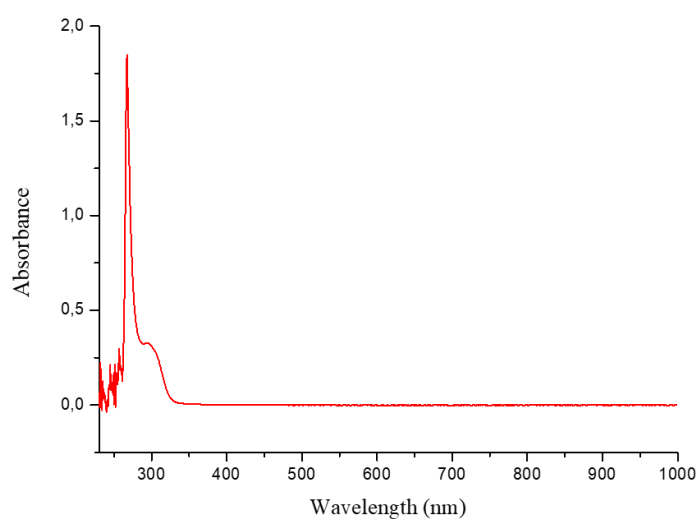


Figure 4. UV-Visible spectrum of compound (I).

Table-1. Crystallographic data and refinement parameter for the compound (I).

| | |
|---|---|
| Chemical formula | $\text{C}_7\text{H}_{14}\text{N}_4\text{O}_3$ |
| M_r | 202.22 |
| Crystal shape/color | Prismatic/colorless |
| Crystal system, space group | Orthorhombic, <i>Pbca</i> |
| Crystal size (mm) | $0.26 \times 0.15 \times 0.15$ |
| a (Å) | 11.5576 (1) |
| b (Å) | 12.6971 (1) |
| c (Å) | 13.5932 (2) |
| V (Å ³) | 1994.78 (4) |
| Z | 8 |
| D_{calc} (g.cm ⁻³) | 1.347 |
| λ (Cu $K\alpha$) (Å) | 1.54187 |
| T (K) | 295 |
| μ (mm ⁻¹) | 0.90 |

| | |
|---|--------------|
| $F(000)$ | 864 |
| θ range ($^{\circ}$) | 6.116-72.766 |
| No. of measured reflections | 17348 |
| No. of independent reflections | 1971 |
| No. of observed [$I > 2\sigma(I)$] reflections | 1804 |
| R_{int} | 0.028 |
| $R[F^2 > 2\sigma(F^2)]$ | 0.043 |
| $wR(F^2)$ | 0.131 |
| Goodness-of-fitt (Gof) on F^2 | 1.08 |
| No. of parameters | 131 |
| No. of restraints | 0 |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ($e \text{ \AA}^{-3}$) | 0.24, -0.18 |

3.2 Structure description of the compound (I)

The title compound crystallizes in the orthorhombic system in the space group $Pbca$. The asymmetric unit of the title compound (I), $C_7H_{14}N_4O_3$, contains one neutral molecule (Figure 5). The selected bond lengths and angles are collected in Table 2. The O1 and N4 atoms are in a *syn* conformation with respect to the C1–N3 link [$O1-C1-N3-N4 = -0.8 (2)^{\circ}$], while the O1 and N2 are in *trans* position with respect to the C1–N1 link [$O1-C1-N1-N2 = -175.5 (1)^{\circ}$]. The carbono fragments O1/N1/N2/C1/N3 (rms 0.0082 \AA) and O1/N3/N4/C1/N1 (rms 0.0327 \AA) are planar, with a maximum deviation from the least-squares plane of 0.0107 (1) \AA for the N3 and 0.0327 \AA for N1 atoms, respectively. Their means planes are almost coplanar with a dihedral angle of 1.70 (3°). The O1–C1 [1.2309 (16) \AA], N2–C2 [1.2717 (18) \AA] and N4–C5 [1.2813 (18) \AA] distances indicate that these correspond to double bonds and are in the normal range comparable to those observed for 1,5-Bis(2-chlorobenzylidene)carbonohydrazide [26]. The C1–N1 [1.3551 (18) \AA] and C1–N3 [1.3576 (16) \AA] are in the normal range expected for single C–N bond lengths [27]. The distances values of C2–O4 [1.4121 (17) \AA] and C7–O3 [1.4262 (19) \AA] are also indicative of single bond character [28].

The crystal packing of compound (I) (Figure 6a) is stabilized by intermolecular hydrogen bonds N(carbohydrazide)–H \cdots O(alcohol), O(alcohol)–H \cdots O(carbohydrazide) and O(alcohol)–H \cdots O(alcohol) (Figure 2a). Intermolecular hydrogen bonds, N3(hydrazinyl)–H3A \cdots O3ⁱ(alcohol), O2(alcohol)–H2 \cdots O3ⁱ(alcohol) (i: $x-1/2, y, -z+3/2$), O3(alcohol)H3 \cdots O1ⁱⁱ(carbonyl) (ii: $-x+3/2, y-1/2, z$), N1(hydrazinyl)–H1 \cdots O2ⁱⁱⁱ(alcohol) (iii: $-x+1/2, y+1/2, z$), lead to the formation of layers parallel to b axis (Figure 6b, Table 3). Additional C–H \cdots O1^{iv}(carbonyl) (iv: $x-1/2, y+1/2, 1-z$) and C4–H4B \cdots O1(carbonyl) connect the layers and consolidate the structure into a three-dimensional network (Figure 2b).

Table-2. Selected bond distances [\AA] and angles [deg] for the compound (I).

| | | | |
|-------|-------------|----------|-------------|
| O1–C1 | 1.2309 (16) | N2–C2–C3 | 126.94 (11) |
| N2–C2 | 1.2717 (18) | N2–C2–C4 | 116.28 (11) |
| N2–N1 | 1.3817 (14) | C3–C2–C4 | 116.71 (12) |
| N3–N4 | 1.3799 (16) | O1–C1–N1 | 120.45 (12) |
| N1–C1 | 1.3551 (18) | O1–C1–N3 | 124.38 (13) |
| N4–C5 | 1.2813 (18) | N1–C1–N3 | 115.16 (11) |
| N3–C1 | 1.3576 (16) | N4–C5–C6 | 127.39 (14) |
| O2–C4 | 1.4121 (17) | N4–C5–C7 | 114.27 (12) |
| O3–C7 | 1.4262 (19) | C6–C5–C7 | 118.31 (12) |

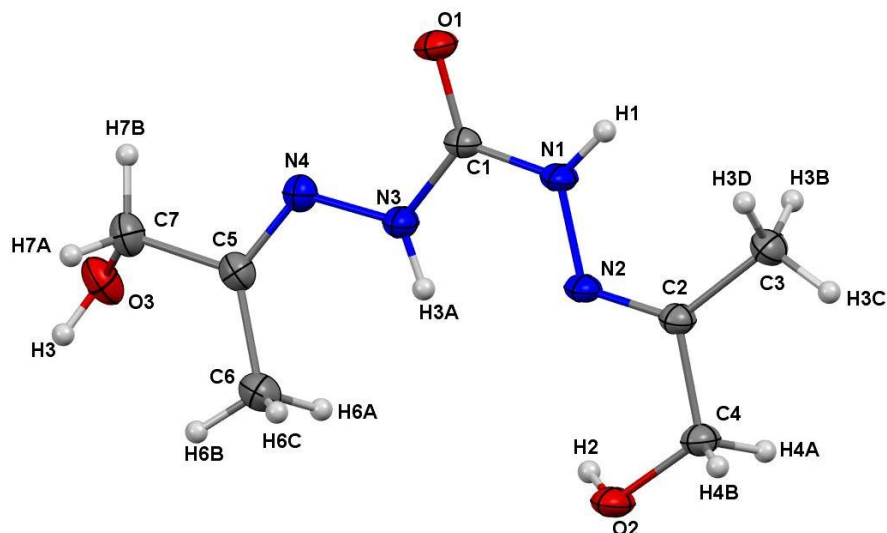


Fig 5. Crystal structure of the compound (I).

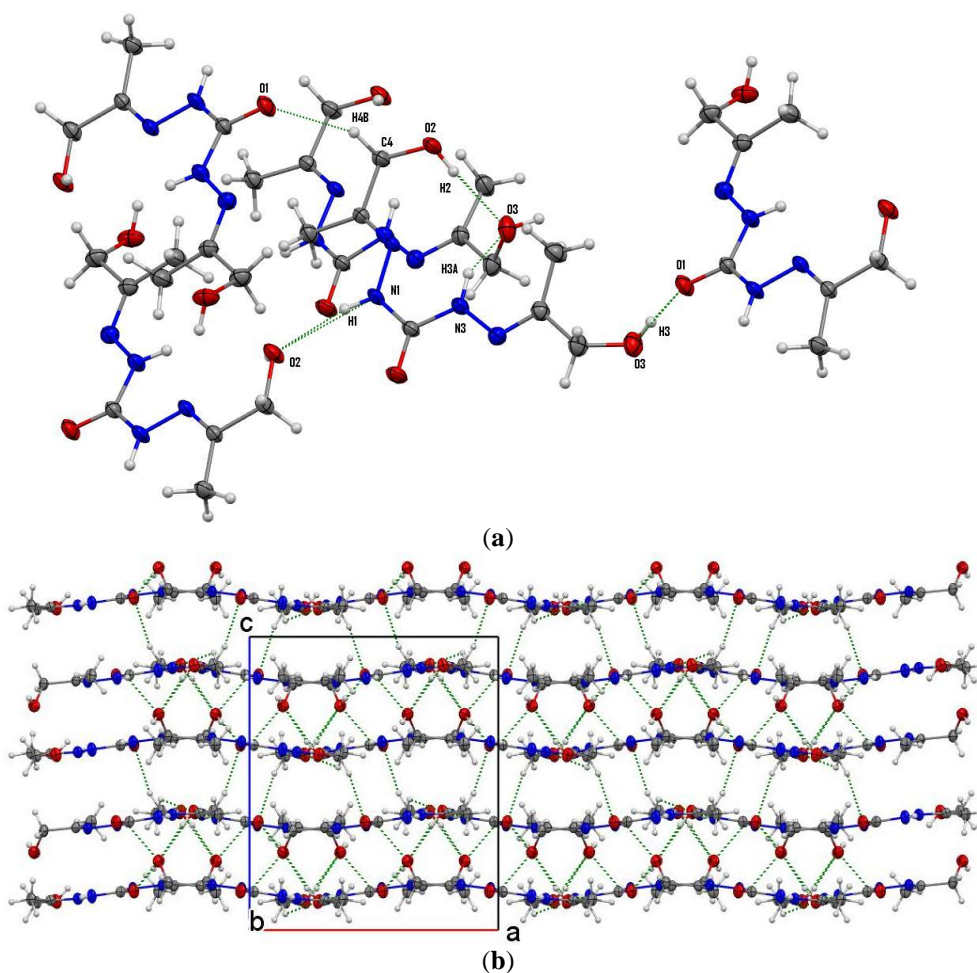


Fig 6. (a) Hydrogen bond of the structure (I) viewed, (b) packing diagram of (I) viewed along the *b* axis.

Table-3. Hydrogen-bond geometry (Å, °)

| <i>D</i> — <i>H</i> ... <i>A</i> | <i>D</i> — <i>H</i> | <i>H</i> ... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> — <i>H</i> ... <i>A</i> |
|----------------------------------|---------------------|-----------------------|-----------------------|----------------------------------|
| O2—H2...O3 ⁱ | 0.82 | 2.01 | 2.8203 (17) | 170.9 |
| O3—H3...O1 ⁱⁱ | 0.82 | 1.86 | 2.6477 (14) | 161.9 |
| N3—H3A...O3 ³ | 0.86 | 2.65 | 3.3310 (17) | 137.5 |
| N1—H1...O2 ⁱⁱⁱ | 0.86 | 2.06 | 2.8992 (15) | 165.6 |
| C4—H4B...O1 ^{iv} | 0.97 | 2.56 | 3.4477 (18) | 152.4 |

Symmetry codes : (i) $x-1/2, y, -z+3/2$; (ii) $-x+3/2, y-1/2, z$; (iii) $-x+1/2, y+1/2, z$; (iv) $x-1/2, -y+3/2, -z+1$.

IV. Concluding remarks

In this present work we reported the synthesis of a symmetrical carbonylhydrazone derivative (1E,5E)-1,5-bis(1-hydroxypropan-2-ylidene)carbonohydrazide (I). The structure of the new Schiff base was discussed by elemental analysis and spectroscopic techniques (FT-IR, UV-visible, ^1H and ^{13}C NMR). X-rays diffraction technique was used to determine the molecular structure of (I).

V. Supporting information

CCDC-2084845 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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