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CRYSTAL STRUCTURE AND NMR SPECTROSCOPIC CHARACTERIZATION OF 1,5-BIS(2-HYDROXY-3-METHOXYBENZYLIDENE)CARBONOHYDRAZIDE

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Abstract. The solid-state structure of a symmetrical carbohydrazone, namely 1,5-bis(2-hydroxy-3-methoxybenzylidene)carbonohydrazide (**1**) was determined by X-ray single crystal diffraction method. Compound **1** crystallizes in the monoclinic space group $P2_1/n$ with unit cell parameters a = 10.1198(6), b = 22.7847(11), c= 15.1738(10) Å, β = 100.458(6)°, Z = 4, V = 3440.6(3) Å^3, R_1 = 0.0540. Crystal structure of **1** is defined by two crystallographic independent molecules, which are bonded *via* N–H···O hydrogen bond. The organic molecules are as keto tautomers with respect to the carbamide fragment, and adopt the *anti* conformation. 1D and 2D NMR experiments have argued on the presence of the title compound in DMSO-d_6 solution mostly as keto tautomer in *syn* conformation, and enol-imino form when considering *o*-vanillin residue.

Keywords: carbohydrazide, *o*-vanillin, *syn-anti* isomer, X-ray diffraction, NMR spectroscopy.

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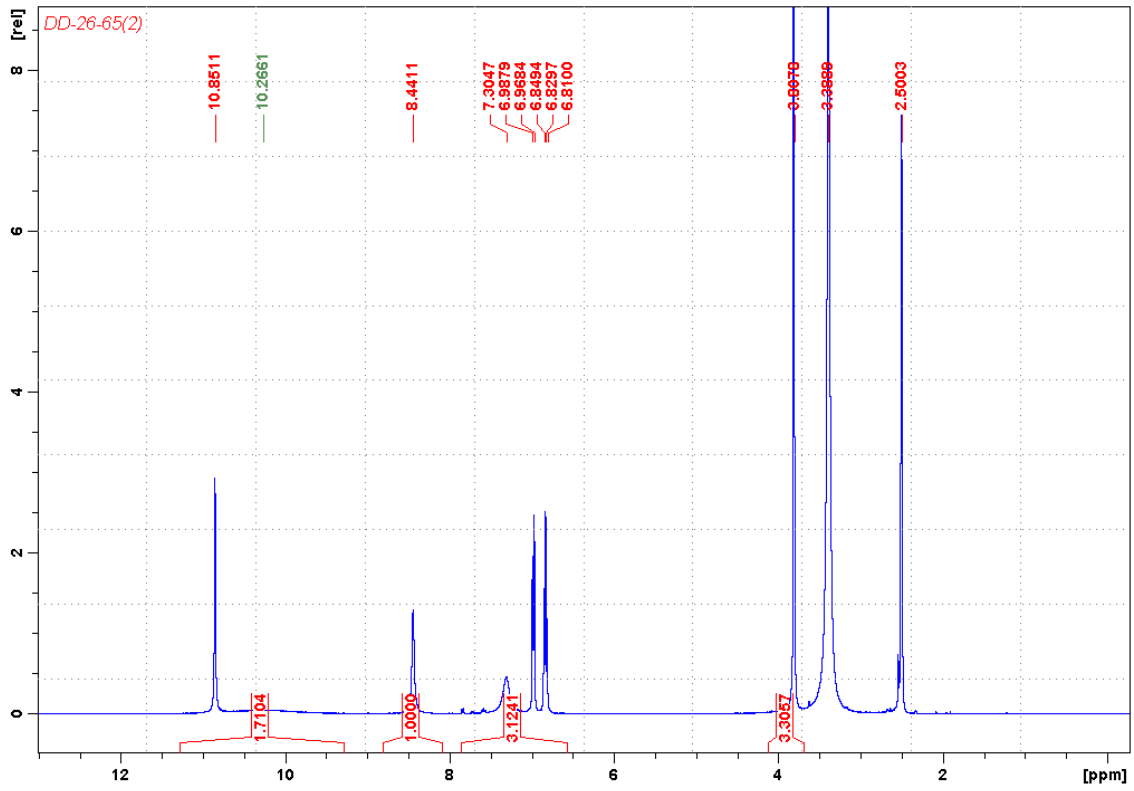

Figure S1. ^{1}H NMR spectrum of 1,5-bis(2-hydroxy-3-methoxybenzylidene)carbohydrazide 1.

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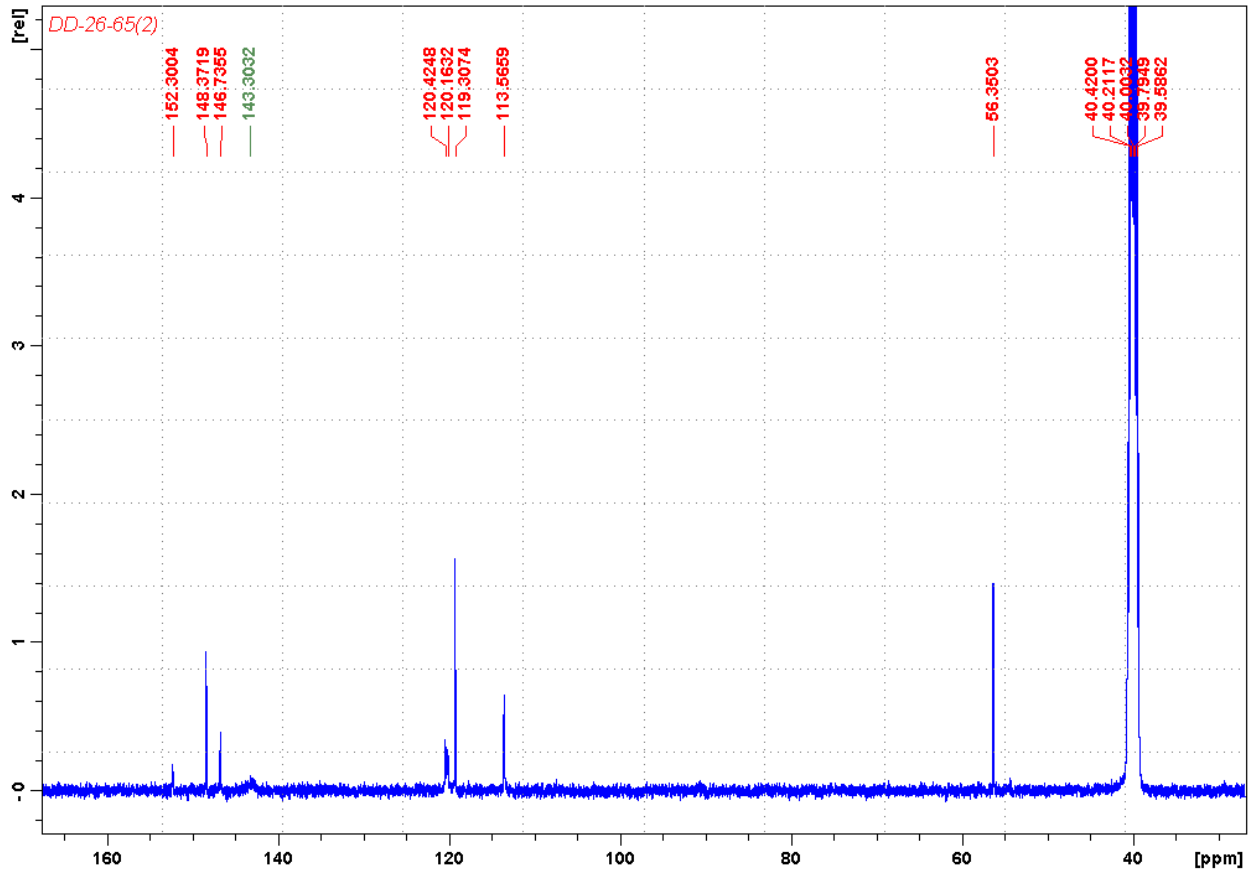

Figure S2. ^{13}C NMR spectrum of compound 1.

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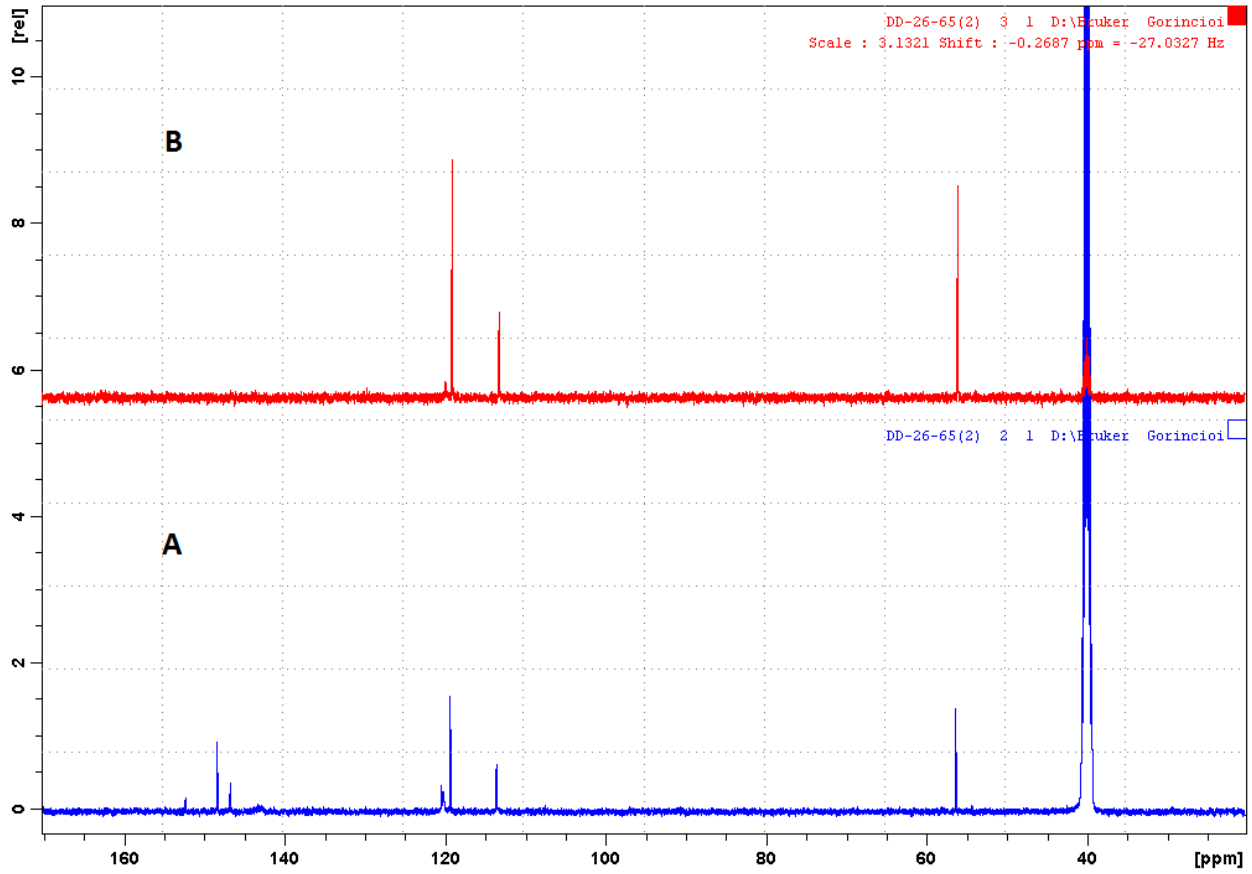

Figure S3. ^{13}C (A) and DEPT-135 (B) NMR spectra of compound 1.

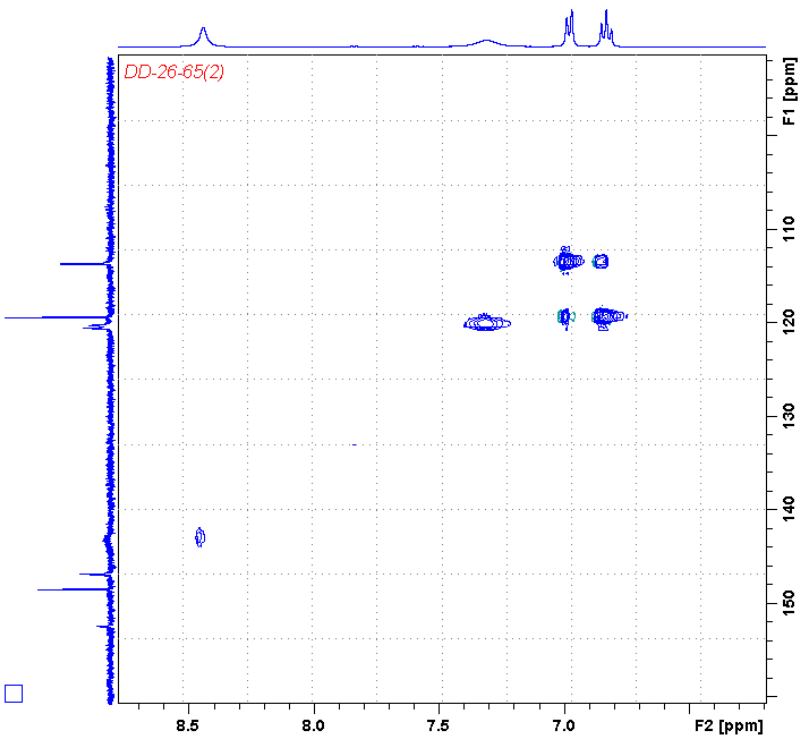

Figure S4. ^{1}H/ ^{13}C qHSQC NMR spectrum of compound 1.

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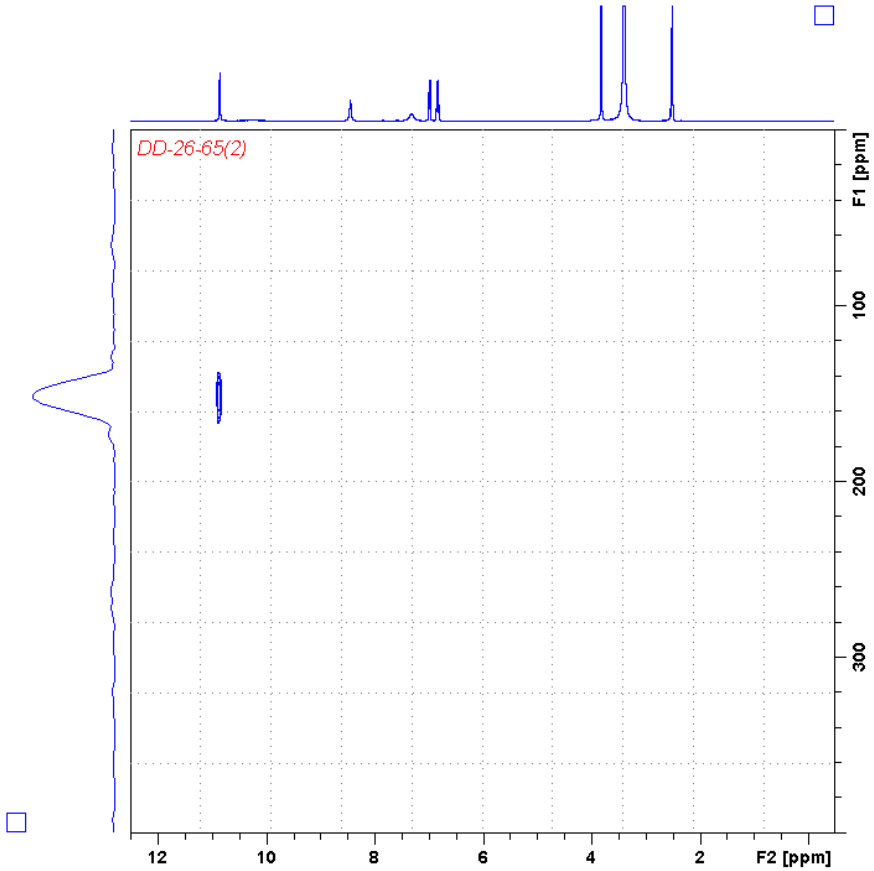

Figure S5. $^{1}H/^{15}N$ HMQC NMR spectrum of compound 1.

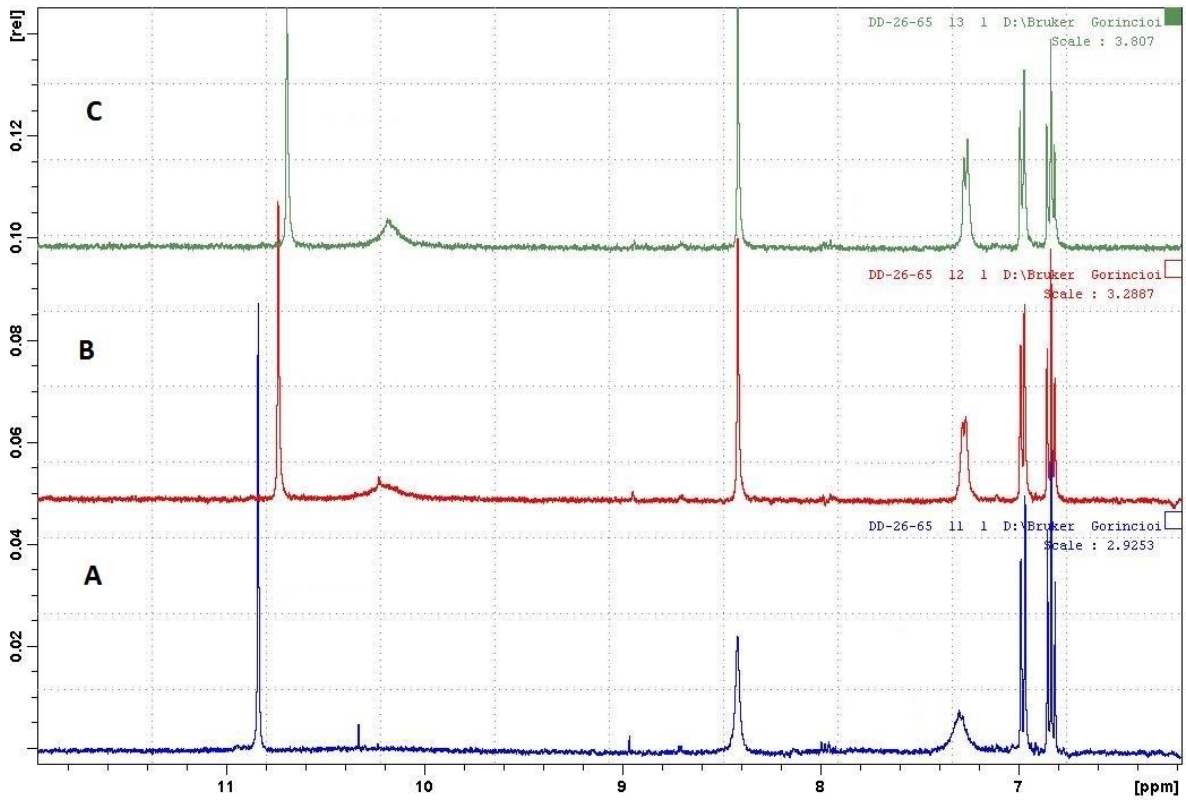

Figure S6. ^{1}H spectra of compound 1 recorded at three temperatures: A- 298K, B- 318K, C- 328K.

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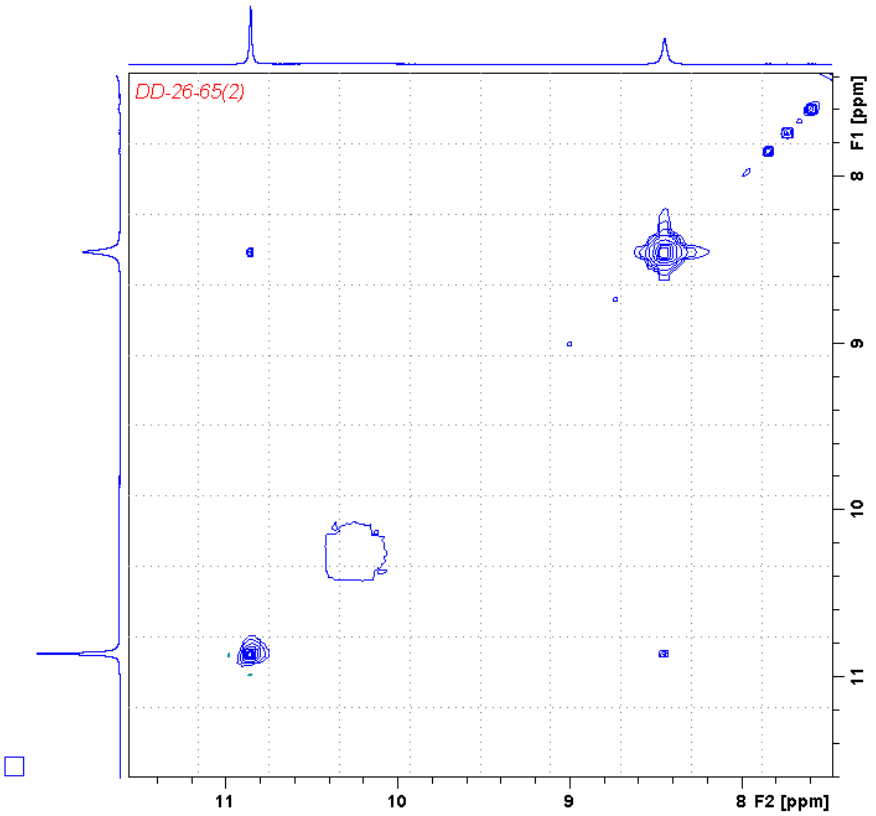

Figure S7. $^{1}H/^{1}H$ NOESY NMR spectrum of compound 1.

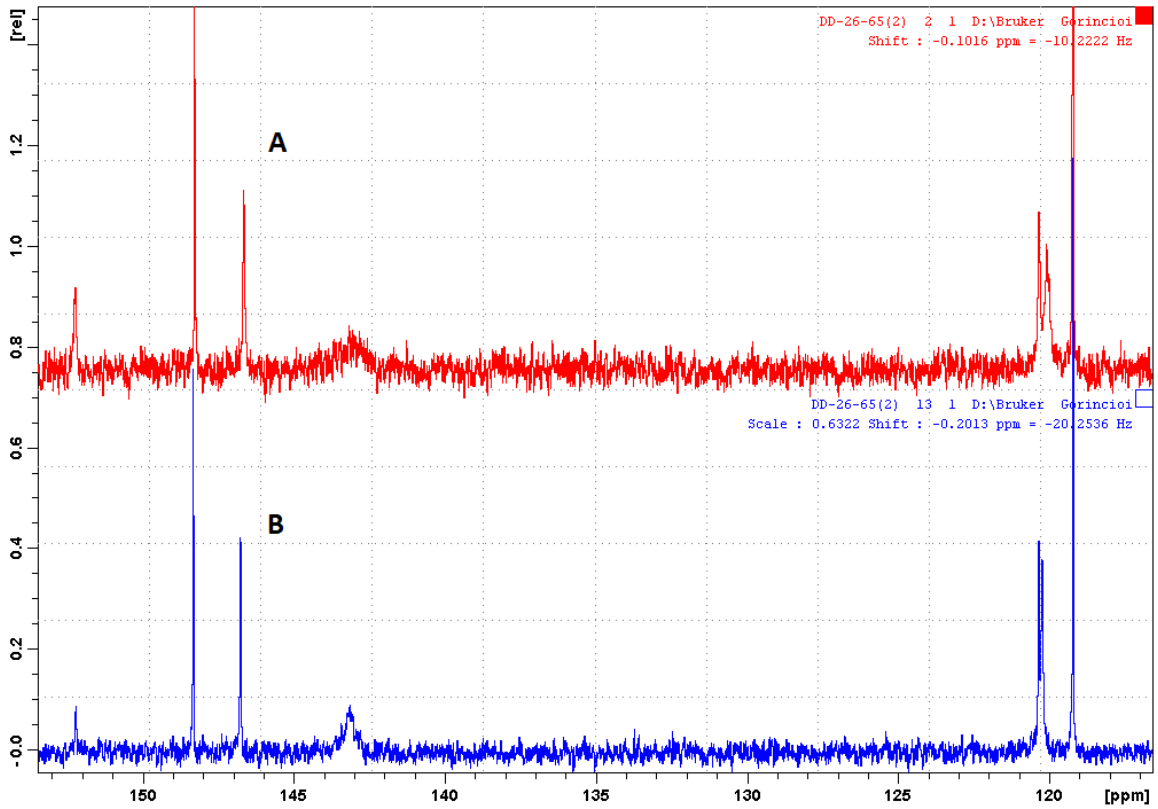

Figure S8. Fragment of ^{13}C NMR spectra of compound 1 recorded at two temperatures: A- 298K, B- 348K.

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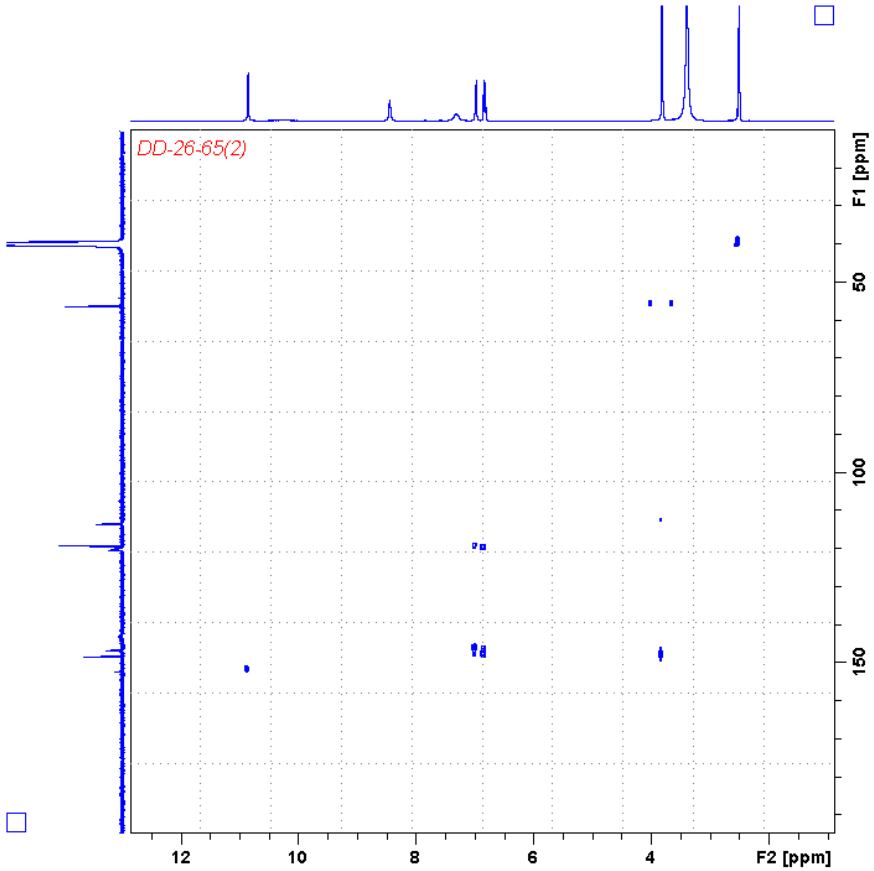

Figure S9. $^{1}H/^{13}C$ HMBC NMR spectrum of compound 1.

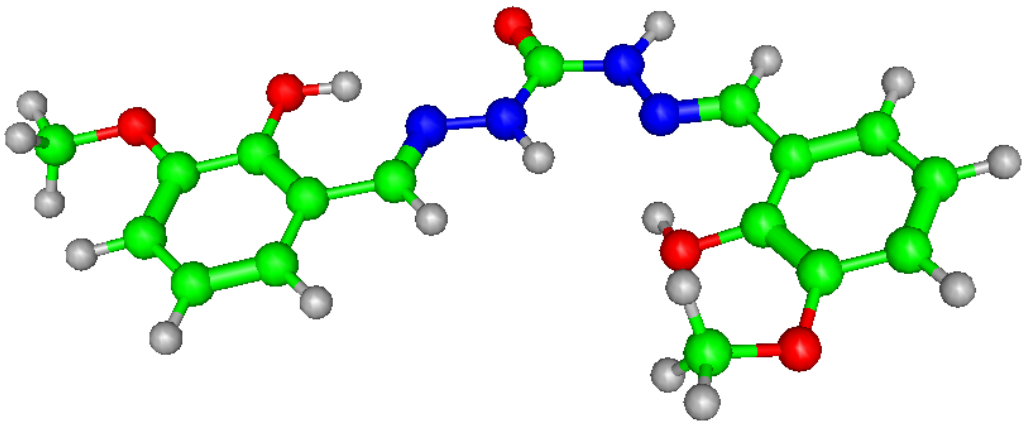

Figure S10. Minimum-energy stereo structure for an *anti*- conformer of compound 1 calculated by using PERCH NMR TOOLS (version 2014.1).