Supplementary Materials For:

4,5-Dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one

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Supplementary Information

IR spectra were registered on a Shimadzu IR Prestige-21 spectrometers with samples in KBr pellets. ^{1}H, $^{13}C\{^{1}H\}$ NMR spectra, ^{1}H–^{13}C HMQC, ^{1}H–^{13}C HMBC, experiments were acquired on a Jeol ECX400A spectrometer (400 MHz for ^{1}H nuclei and 100 MHz for ^{13}C nuclei) in DMSO-d_6. The residual signals of the solvent (DMSO-d_6: 2.50 ppm for ^{1}H nuclei and 39.6 ppm for ^{13}C nuclei) were used as internal standard.

Spectral studies were performed using the equipment of the Center for Collective Use «Physico-chemical methods for the study of nitro compounds, coordination compounds, biologically active substances, and nanostructured materials» of the Interdisciplinary Resource Center for Collective Use «Modern physico-chemical methods of formation and research of materials for the needs of industry, science, and education» of the Herzen State Pedagogical University of Russia.

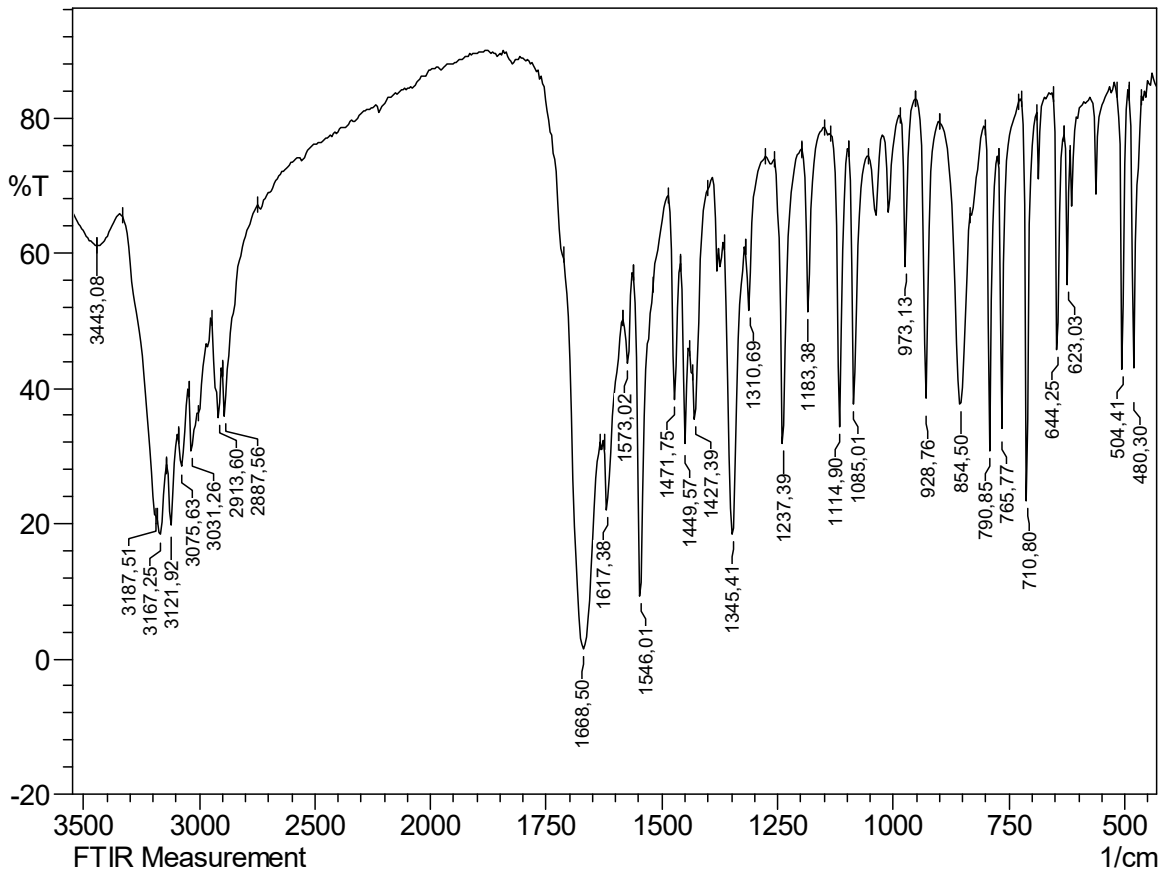

Figure S1. IR spectrum of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one in KBr.

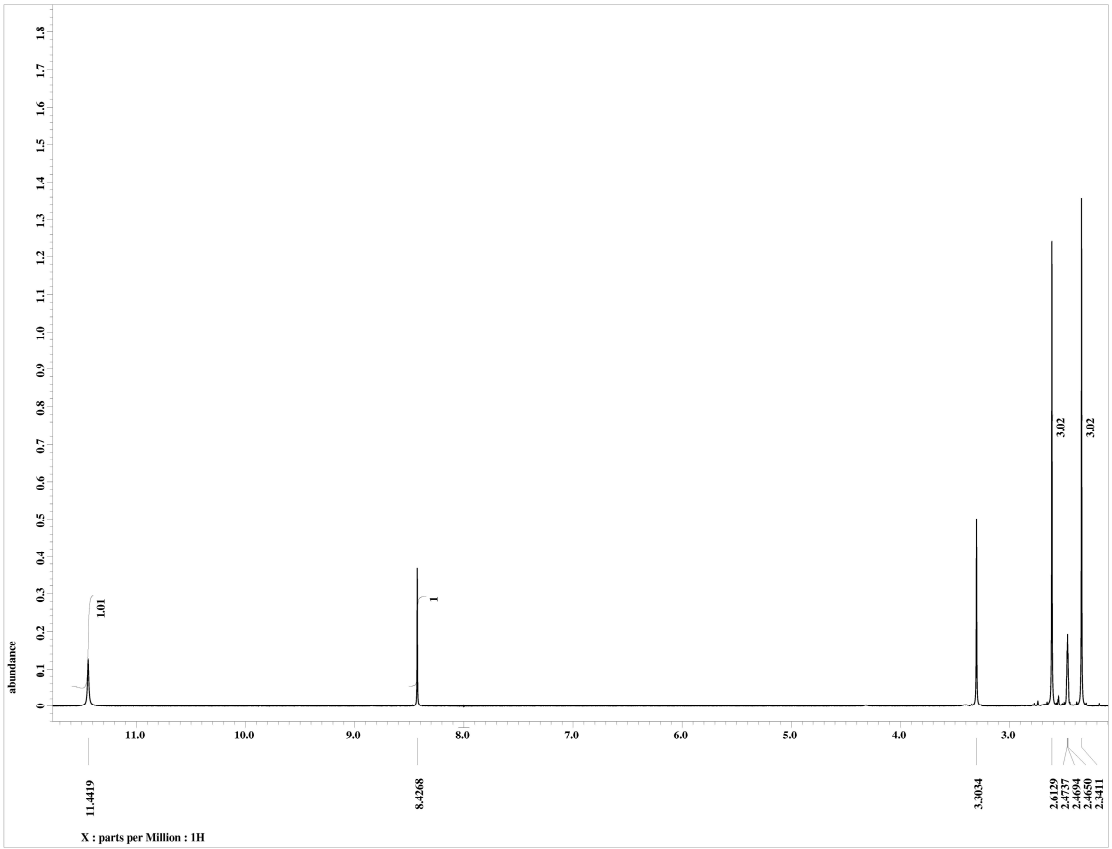

Figure S2. ^{1}H NMR spectrum of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one in DMSO-d_6.

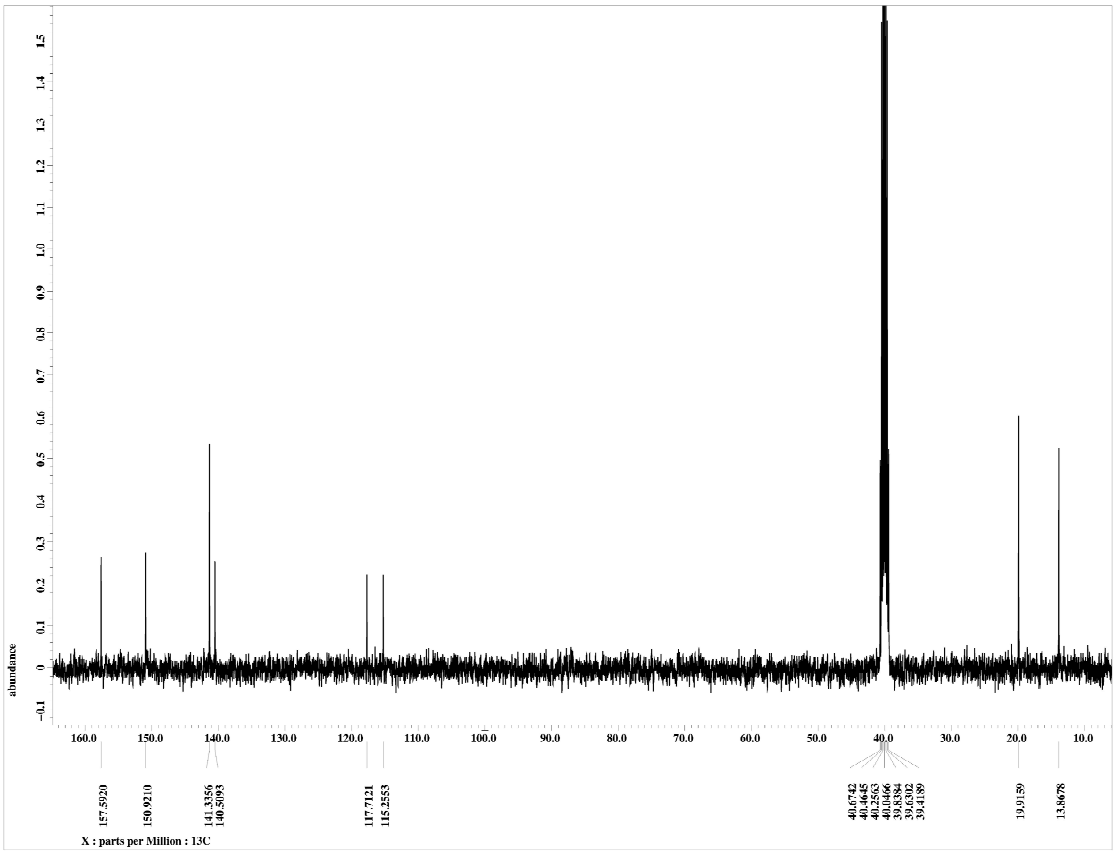

Figure S3. ^{13}C-{^{1}H} NMR spectrum of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one in DMSO-*d*$_6$.

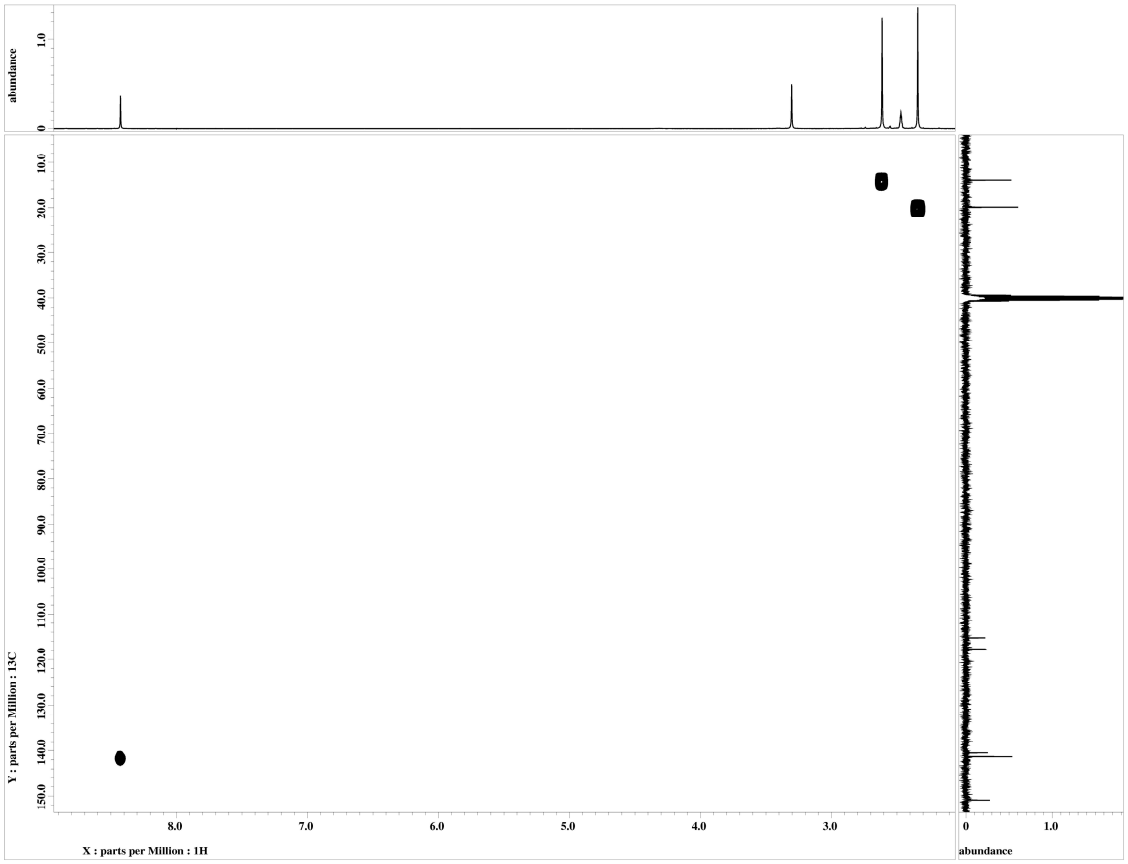

Figure S4. ^{1}H-^{13}C HMQC spectrum of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one in DMSO-*d*$_6$.

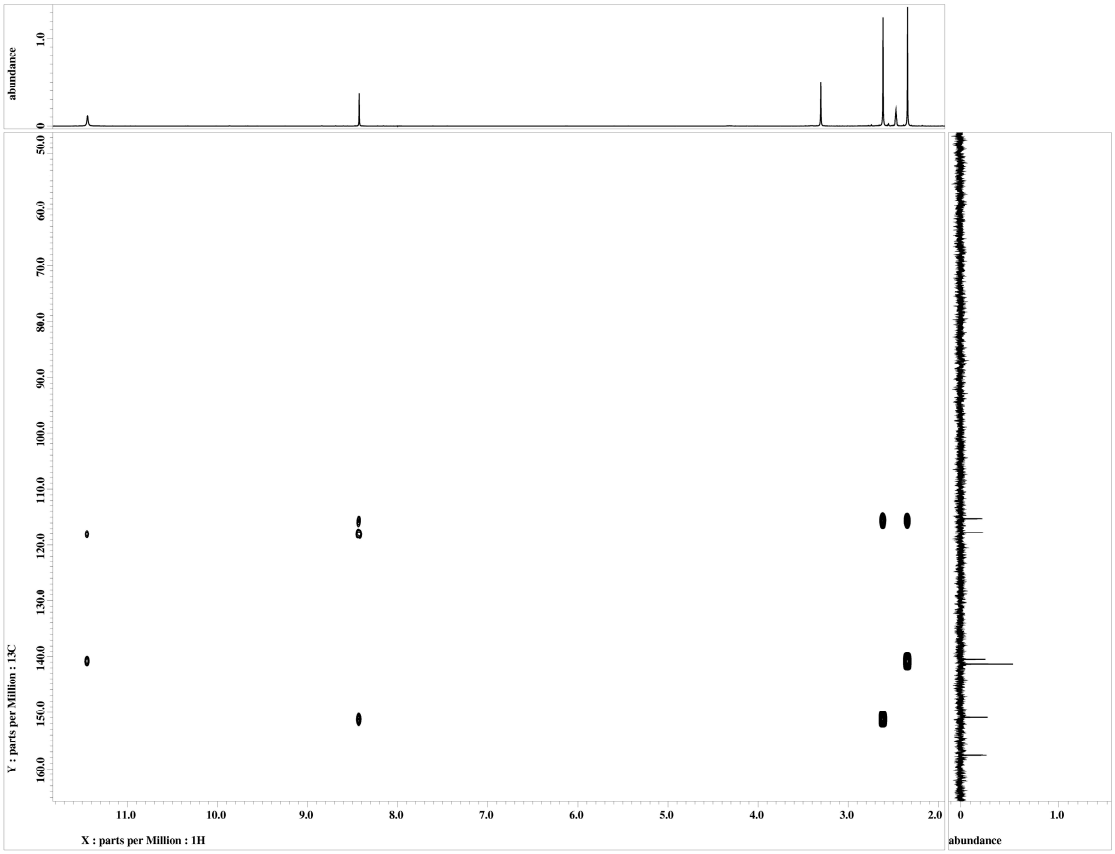

Figure S5. ^{1}H-^{13}C HMBC spectrum of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one in DMSO-d_6.

X-ray diffraction analysis

X-ray diffraction analysis was performed at 108 K on a Bruker D8 QUEST automatic three-circle diffractometer (graphite monochromator, λMoK$_\alpha$ = 0.71073 Å, ω- and φ-scan with a step of 0.5°) at the Distributed Spectral-Analytical Center of Shared Facilities for Study of Structure, Composition and Properties of Substances and Materials of FRC Kazan Scientific Center of RAS.

An X-ray diffraction analysis of **2** was performed on a Bruker D8 QUEST automatic three-circle diffractometer with a PHOTON III two-dimensional detector and an I_S DIAMOND microfocus X-ray tube (Mo Kα radiation, λ = 0.71073 Å) at cooling conditions. Data collection and the processing of diffraction data were performed using APEX3 software package. Structure **2** was solved by the direct method using the SHELXT program. It was refined by the full-matrix least squares method over F^2 using the SHELXL program. Crystal **2** was found to be two-component twin, twin rot matrix (-0.694 0 -0.424)/ (0 -1 0)/ (-1.223 0 0.694), Estimated BASF Line = 0.46. Final model was refined using a combined diffraction data set (HKLF 5), refined parameter BASF 0.33285. All calculations were performed in the WinGX software package. The calculations of the geometry of molecules and intermolecular interactions in crystals were carried out using the PLATON program. The drawings of molecule was performed using the MERCURY programs. Non-hydrogen atoms were refined in the anisotropic approximation. The position of the hydrogen atom H(N2) were determined using difference Fourier map. The remaining hydrogen atoms were placed in geometrically calculated positions, and all hydrogen atoms included in the refinement in the "riding" model.

Crystal **2** monoclinic, at 108(2) K a =7.024(4), b = 7.659(4), c= 13.714(7) Å, β = 98.312(17)°, V = 730.1(6) Å^3, Z = 4 ρ_{calc} 1.493 g/cm^3, μ 0.110 mm^{1-}. Reflections collected 19508, Independent reflections 1464, Observed reflections (I≥ 2σ(I) 831. Final R-factors: *R* factors [I>=2σ (I)] R_1 = 0.0802, wR_2 = 0.1895, *R* factors [all reflections] R_1 = 0.1442, wR_2 = 0.2182, GOOF on F^2 1.003

Crystallographic data, experimental parameters, and refinement of the condensed biheterocyclic structure are shown in Table S1 and was deposited at the Cambridge Crystallographic Data Center; registration numbers and the most important characteristics are provided in Tables S1–S3.

Table S1. Crystallographic data, experimental parameters, and refinement of the 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one.

Parameter	**Value**
Empirical formula	$C_8H_8N_2O_2$
Molecular weight	164.16
Temperature/K	108(2)
Crystal system	monoclinic
Space group	*P* $2_1/c$
a/Å	7.024(4)
b/Å	7.659(4)
c/Å	13.714(7)
α/°	90
β/°	98.312(17)
γ/°	90
Volume/Å^3	730.1(6)
Z	4
ρ_{calc}/ g/cm^3	1.493
μ/mm^{-1}	0.110
F(000)	344.0
Crystal size/mm	0.20 × 0.10 × 0.03
Radiation/ λ/Å	MoKα 0.71073)
Θ range for data collection/°	2.93 - 26.304
Index ranges	$-8 \le h \le 8$, $-9 \le k \le 9$, $-9 \le l \le 17$
Reflections collected	19508
Independent reflections	1464
Observed reflections (I≥ 2σ(I))	831
GOOF on F^2	1.003
R factors [I>=2σ (I)]	$R_1 = 0.0802$, $wR_2 = 0.1895$
R factors [all reflections]	$R_1 = 0.1442$, $wR_2 = 0.2182$
Largest diff. peak/hole, e Å^{-3}	0.70/-0.32
CCDC No.	2321540

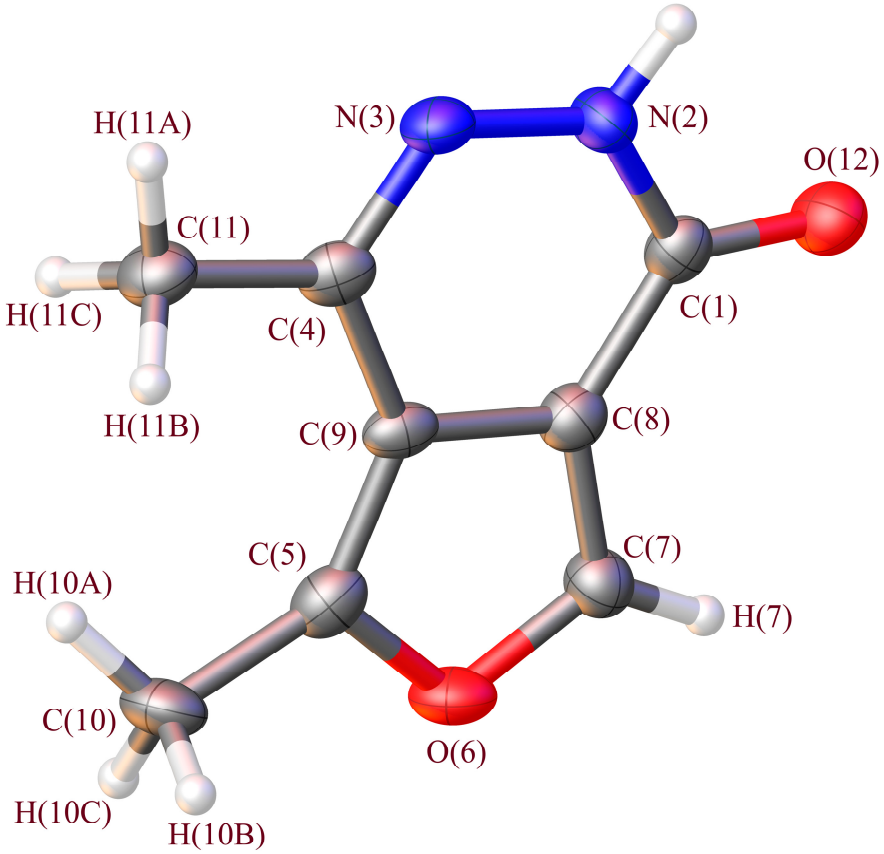

Figure S6. Geometry of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one in the crystal. Ellipsoids of anisotropic displacements are shown with the 50% probability.

Table S2. Bond lengths (*d*) in the molecule of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one.

Bond	***d*, Å**	**Bond**	***d*, Å**
O(6)–C(5)	1.390(5)	O(6)–C(7)	1.354(5)
O(12)–C(1)	1.243(5)	N(2)–N(3)	1.384(4)
N(2)–C(1)	1.348(5)	N(3)–C(4)	1.298(5)
C(1)–C(8)	1.432(5)	C(4)–C(9)	1.453(4)
C(4)–C(11)	1.476(5)	C(5)–C(9)	1.352(5)
C(5)–C(10)	1.468(6)	C(7)–C(8)	1.351(5)
C(8)–C(9)	1.422(5)		

Table S3. Angles (τ) in the molecule of 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one.

Angle	**τ/deg**	**Angle**	**τ/deg**
C(8)–C(1)–N(2)	113.5(3)	C(5)–C(10)–H(10A)	109.4
C(8)–C(1)–O(12)	126.5(4)	C(5)–C(10)–H(10B)	109.5
N(2)–C(1)–O(12)	120.0(3)	C(5)–C(10)–H(10C)	109.4
C(9)–C(4)–C(11)	122.6(3)	H(10A)–C(10)–H(10B)	109.5
C(9)–C(4)–N(3)	119.8(3)	H(10A)–C(10)–H(10C)	109.4
C(11)–C(4)–N(3)	117.6(3)	H(10B)–C(10)–H(10C)	109.5
C(9)–C(5)–C(10)	137.8(4)	C(4)–C(11)–H(11A)	109.4
C(9)–C(5)–O(6)	107.4(3)	C(4)–C(11)–H(11B)	109.5

C(10)–C(5)–O(6)	114.8(3)	C(4)–C(11)–H(11C)	109.4
H(7)–C(7)–C(8)	125.3	H(11A)–C(11)–H(11B)	109.5
H(7)–C(7)–O(6)	125.3	H(11A)–C(11)–H(11C)	109.4
C(8)–C(7)–O(6)	109.4(3)	H(11B)–C(11)–H(11C)	109.5
C(1)–C(8)–C(7)	133.5(4)	C(1)–N(2)–N(3)	129.0(3)
C(1)–C(8)–C(9)	119.9(3)	C(1)–N(2)–H(2)	111(2)
C(7)–C(8)–C(9)	106.6(3)	N(3)–N(2)–H(2)	120(2)
C(4)–C(9)–C(5)	132.6(4)	C(4)–N(3)–N(2)	118.3(3)
C(4)–C(9)–C(8)	119.3(3)	C(5)–O(6)–C(7)	108.5(3)
C(5)–C(9)–C(8)	108.1(3)		

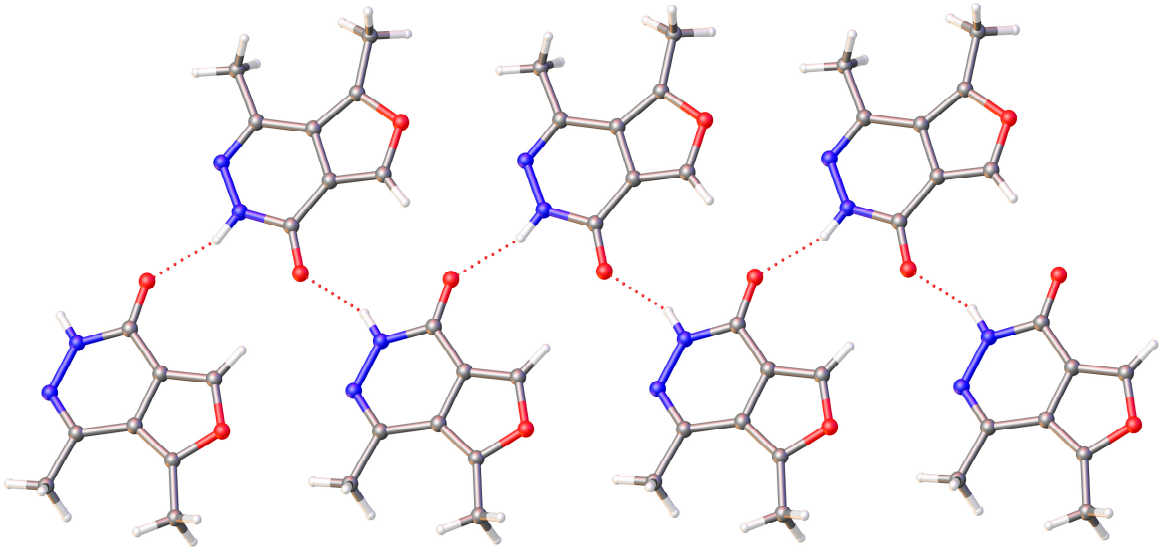

Figure S7. Hydrogen bonding in the crystal 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one. H-bonds are shown by dotted lines.

Figure S7 shows a fragment of the packaging of crystal **2**, which demonstrates interplanar interactions, due to which the formation of double layers is observed. Interplanar distances are 3.19(1) Å, the angle between the planes of molecules is 0.16(12)°.

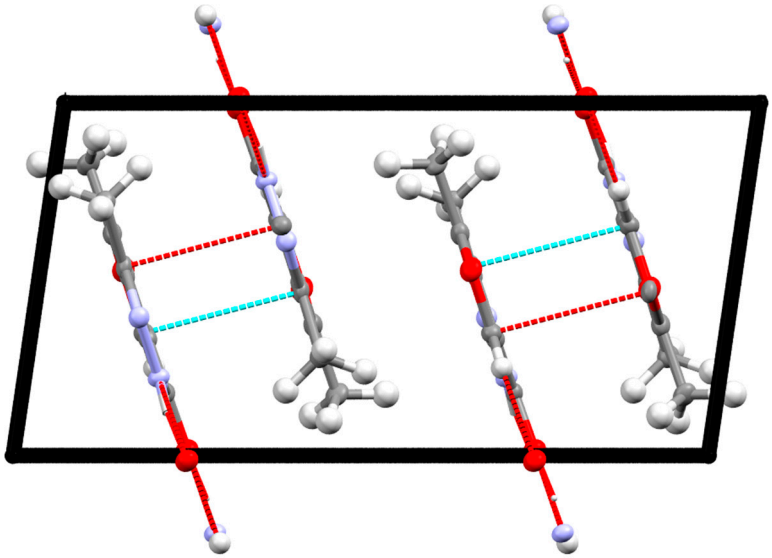

Figure S8. Stacking interaction in the crystal 4,5-dimethylfuro[3,4-*d*]pyridazin-1(2*H*)-one. Short contacts are shown by dotted lines. Projection along the b axe.