APPENDIX A

Supplementary data to the research article: Rukk N.S., Shamsiev R.S., Albov D.V., Mudretsova S.N. Structural characterization of hydrogen bonding for antipyrine derivatives: Single-crystal X-ray diffraction and theoretical studies. *Tonk. Khim. Tekhnol. = Fine Chem. Technol.* 2021;16(2):113–124

Table A1. Crystal data and the details of data collection and refinement for compounds **I**, **II**, and **III**

Compound	I	II	III
Empirical formula	$C_{10}H_{10}N_2O$	$C_{10}H_9ClN_2O$	$C_{11}H_{12}N_2O$
Formula weight	174.20	208.64	188.23
Crystal system, Sp. gr., Z	Monoclinic, $P2_1/c$, 8	Triclinic, P–1, 2	Monoclinic, C2/c, 8
a, *b*, *c*, Å	*a* = 10.331(3) *b* = 11.155(4) *c* = 15.880(5)	*a* = 5.8551(12) *b* = 7.674(3) *c* = 11.348(3)	*a* = 16.892(9) *b* = 7.429(3) *c* = 17.776(8)
α, β, γ, °; V, Å^3	90.00, 95.06(3), 90.00; 1822.9 (10)	106.11(2), 94.669(19), 101.51(2); 475.0(2)	90.00, 116.98(5), 90.00; 1987.9(16)
F(000)	736	216	800
D_x, Mg·m^{-3}	1.269	1.459	1.258
Radiation	Cu Kα radiation, λ = 1.5418 Å, Cell parameters from 25 reflections θ = 33°–35°	Cu Kα radiation, λ = 1.5418 Å, Cell parameters from 25 reflections θ = 30°–33°	Ag Kα radiation, λ = 0.56087 Å, Cell parameters from 25 reflections θ = 15°–16°
μ, mm^{-1}	0.68	3.28	0.05
Crystal size, mm^3	0.20 × 0.20 × 0.20 Colorless prisms	0.10 × 0.10 × 0.10 Colorless prisms	0.50 × 0.50 × 0.50 Colorless prisms
Data collection: Enraf Nonius CAD4 diffractometer[3]; radiation source: fine-focus sealed tube; monochromator: graphite; non–profiled ω scans.			
3450 measured reflections, 3450 independent reflections, 2988 reflections with $I > 2\sigma(I)$; R_{int} = 0.0000; θ_{max} = 69.9°, θ_{min} = 4.3°; $h = -12 \rightarrow 12$; $k = 0 \rightarrow 13$; $l = 0 \rightarrow 19$; 1 standard reflection every 120 min; intensity decay: 1%	1952 measured reflections, 1952 independent reflections, 1740 reflections with $I > 2\sigma(I)$; R_{int} = 0.0000; θ_{max} = 74.8°, θ_{min} = 4.1°; $h = -7 \rightarrow 7$; $k = -9 \rightarrow 9$; $l = 0 \rightarrow 14$; 2 standard reflections every 120 min; intensity decay: 2%	9715 measured reflections, 4882 independent reflections, 2820 reflections with $I > 2\sigma(I)$; R_{int} = 0.070; θ_{max} = 28.0°, θ_{min} = 2.0°; $h = -28 \rightarrow 28$; $k = -12 \rightarrow 12$; $l = -29 \rightarrow 15$; 1 standard reflection every 60 min; intensity decay: 2%	

[3] Enraf_Nonius CAD_4 Software. Version 5.0. Delft (The Netherlands): Enraf_Nonius, 1989.

Table A1. Continued

Absorption correction: multiscan [20]. Refinement on F^2. Least-squares matrix: full			
Refinement			
H-atom treatment	H atoms were treated by a mixture of independent and constrained refinement	H-atom parameters constrained	
$R[F^2 > 2\sigma(F^2)]$	0.037	0.057	0.058
w$R(F^2)$	0.102	0.158	0.175
S	1.04	1.06	1.01
Reflections/parameters/ restraints	3450/250/0	1952/128/0	4882/130/0
$\Delta\rho_{max}/\Delta\rho_{min}$, e·Å^{-3}	0.15/−0.11	0.33/−0.22	0.26/−0.30

Note: Z = the number of formula units in the unit cell;
R_{int} = merging error (measure of the precision/reproducibility);
θ_{max} = max θ angle in degrees for the reflection used for measurement of the unit cell;
θ_{min} = min θ angle in degrees for the reflection used for measurement of the unit cell;
Sp. gr. = space group;
μ = absorption coefficient;
λ = wavelength, refers to the radiation used to measure intensities;
V = unit cell volume;
a, *b*, and *c* = cell lengths; α, β, and γ = cell angles;
D_x = calculated density;
I = intensity of reflection;
R = R-factor;
w*R* = weighed R-factor;
F(000) = sum of all electrons in the unit cell;
σ = standard deviation;
S = goodness of fit;
h, *k*, and *l* = Miller indices;
$I > 2\sigma(I)$ = criterion for strong reflections.

Table A2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for compound **I**

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.19338 (10)	0.37391 (8)	0.06745 (7)	0.0430 (2)
N2	0.27022 (11)	0.29948 (9)	0.02385 (7)	0.0498 (3)
C3	0.32777 (14)	0.37019 (11)	−0.02844 (9)	0.0518 (3)
C4	0.29077 (14)	0.48962 (11)	−0.01909 (9)	0.0530 (3)
H4	0.3209 (16)	0.5576 (15)	−0.0496 (10)	0.067 (5)*
C5	0.20504 (12)	0.48922 (10)	0.04174 (8)	0.0439 (3)
O5	0.13825 (10)	0.57503 (8)	0.07625 (7)	0.0570 (3)
H5	0.1924 (18)	0.6493 (19)	0.0784 (12)	0.087 (6)*
C6	0.11839 (13)	0.32597 (10)	0.12979 (8)	0.0455 (3)
C7	−0.00259 (14)	0.37340 (13)	0.14200 (9)	0.0555 (3)
H7	−0.0347	0.4389	0.1105	0.067*
C8	−0.07485 (17)	0.32197 (17)	0.20172 (12)	0.0747 (5)
H8	−0.1553	0.3543	0.2110	0.090*
C9	−0.0297 (2)	0.22383 (18)	0.24762 (12)	0.0854 (6)
H9	−0.0802	0.1886	0.2865	0.102*
C10	0.0912 (2)	0.17804 (15)	0.23559 (11)	0.0802 (6)
H10	0.1224	0.1122	0.2670	0.096*
C11	0.16638 (17)	0.22889 (12)	0.17740 (9)	0.0598 (4)
H11	0.2484	0.1984	0.1702	0.072*
C12	0.41644 (18)	0.31848 (16)	−0.08831 (12)	0.0780 (5)
H12A	0.5048	0.3378	−0.0694	0.117*
H12B	0.3954	0.3514	−0.1437	0.117*
H12C	0.4061	0.2330	−0.0904	0.117*
N21	0.30234 (11)	0.96602 (8)	0.07065 (7)	0.0473 (3)
N22	0.26333 (12)	1.04936 (9)	0.00912 (8)	0.0517 (3)
H22	0.2696 (16)	1.1302 (18)	0.0200 (11)	0.075 (5)*
C23	0.16953 (14)	0.99970 (12)	−0.04340 (9)	0.0546 (3)
C24	0.15371 (15)	0.88297 (12)	−0.02102 (10)	0.0564 (4)
H24	0.0945 (16)	0.8289 (15)	−0.0484 (10)	0.068 (5)*
C25	0.23756 (13)	0.85971 (10)	0.05141 (9)	0.0486 (3)
O25	0.26042 (11)	0.76589 (8)	0.09483 (7)	0.0646 (3)
C26	0.41009 (12)	0.98926 (10)	0.12963 (9)	0.0468 (3)
C27	0.42958 (16)	0.92134 (13)	0.20319 (10)	0.0606 (4)

Table A2. Continued

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H27	0.3720	0.8601	0.2137	0.073*
C28	0.53456 (18)	0.94523 (17)	0.26046 (11)	0.0752 (5)
H28	0.5485	0.8987	0.3090	0.090*
C29	0.61888 (18)	1.03716 (19)	0.24653 (13)	0.0799 (5)
H29	0.6887	1.0536	0.2859	0.096*
C30	0.59944 (16)	1.10417 (17)	0.17444 (13)	0.0750 (5)
H30	0.6563	1.1665	0.1652	0.090*
C31	0.49607 (14)	1.08054 (13)	0.11487 (11)	0.0595 (4)
H31	0.4847	1.1256	0.0655	0.071*
C32	0.10490 (19)	1.07208 (16)	−0.11336 (11)	0.0751 (5)
H32A	0.0953	1.1532	−0.0947	0.113*
H32B	0.0208	1.0389	−0.1301	0.113*
H32C	0.1568	1.0710	−0.1606	0.113*

Note: The sign * indicates the isotropic displacement parameters, Å^2.

Table A3. Atomic displacement parameters (Å^2) for compound **I**

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0534 (6)	0.0206 (4)	0.0566 (6)	−0.0024 (4)	0.0141 (5)	−0.0016 (4)
N2	0.0611 (7)	0.0248 (5)	0.0661 (7)	0.0017 (4)	0.0207 (5)	−0.0021 (4)
C3	0.0578 (7)	0.0343 (6)	0.0656 (8)	−0.0016 (5)	0.0180 (6)	0.0004 (6)
C4	0.0654 (8)	0.0278 (6)	0.0681 (8)	−0.0062 (6)	0.0182 (7)	0.0068 (6)
C5	0.0535 (7)	0.0206 (5)	0.0582 (7)	−0.0031 (5)	0.0080 (6)	−0.0013 (5)
O5	0.0680 (6)	0.0238 (4)	0.0819 (7)	−0.0005 (4)	0.0224 (5)	−0.0055 (4)
C6	0.0581 (7)	0.0275 (6)	0.0522 (7)	−0.0118 (5)	0.0124 (6)	−0.0068 (5)
C7	0.0565 (8)	0.0460 (7)	0.0652 (8)	−0.0128 (6)	0.0128 (6)	−0.0136 (6)
C8	0.0718 (10)	0.0743 (11)	0.0825 (11)	−0.0306 (9)	0.0328 (9)	−0.0272 (9)
C9	0.1175 (16)	0.0750 (12)	0.0694 (10)	−0.0501(12)	0.0415 (11)	−0.0159 (9)
C10	0.1320 (17)	0.0452 (9)	0.0659 (10)	−0.0267(10)	0.0222 (10)	0.0051 (7)
C11	0.0854 (10)	0.0328 (6)	0.0628 (8)	−0.0065 (6)	0.0152 (7)	0.0018 (6)
C12	0.0865 (12)	0.0592 (10)	0.0951 (12)	0.0037 (8)	0.0462 (10)	−0.0027 (9)
N21	0.0558 (6)	0.0203 (4)	0.0649 (6)	−0.0015 (4)	−0.0003 (5)	−0.0005 (4)
N22	0.0660 (7)	0.0233 (5)	0.0648 (7)	−0.0013 (5)	−0.0004 (6)	−0.0012 (5)
C23	0.0625 (8)	0.0395 (7)	0.0613 (8)	0.0002 (6)	0.0019 (6)	−0.0059 (6)
C24	0.0623 (8)	0.0363 (7)	0.0699 (9)	−0.0102 (6)	0.0020 (7)	−0.0121 (6)

Table A3. Continued

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C25	0.0540 (7)	0.0220 (5)	0.0707 (8)	−0.0033 (5)	0.0099 (6)	−0.0068 (5)
O25	0.0771 (7)	0.0234 (4)	0.0925 (8)	−0.0051 (4)	0.0027 (6)	0.0034 (4)
C26	0.0497 (7)	0.0283 (6)	0.0626 (7)	0.0039 (5)	0.0064 (6)	−0.0092 (5)
C27	0.0675 (9)	0.0459 (8)	0.0683 (9)	0.0036 (7)	0.0058 (7)	0.0002 (7)
C28	0.0793 (11)	0.0766 (11)	0.0676 (10)	0.0117 (9)	−0.0046 (8)	−0.0029 (8)
C29	0.0662 (10)	0.0902 (13)	0.0802 (11)	0.0004 (10)	−0.0099 (9)	−0.0195(10)
C30	0.0607 (9)	0.0674 (10)	0.0966 (13)	−0.0145 (8)	0.0047 (9)	−0.0190(10)
C31	0.0587 (8)	0.0446 (7)	0.0749 (9)	−0.0085 (6)	0.0050 (7)	−0.0059 (7)
C32	0.0907 (12)	0.0627 (10)	0.0691 (10)	0.0040 (9)	−0.0079 (9)	0.0032 (8)

Table A4. Geometric parameters (Å, º) for compound **I**

Atom–Atom	Bond length, Å	Atom–Atom	Bond length, Å
N1–C5	1.3581 (15)	N21–C25	1.3826 (15)
N1–N2	1.3768 (14)	N21–N22	1.3827 (15)
N1–C6	1.4147 (16)	N21–C26	1.4140 (18)
N2–C3	1.3237 (17)	N22–C23	1.3413 (19)
C3–C4	1.3976 (18)	N22–H22	0.92 (2)
C3–C12	1.493 (2)	C23–C24	1.363 (2)
C4–C5	1.367 (2)	C23–C32	1.484 (2)
C4–H4	0.966 (17)	C24–C25	1.402 (2)
C5–O5	1.3265 (15)	C24–H24	0.938 (17)
O5–H5	1.00 (2)	C25–O25	1.2640 (16)
C6–C7	1.386 (2)	C26–C31	1.3848 (19)
C6–C11	1.3870 (19)	C26–C27	1.392 (2)
C7–C8	1.382 (2)	C27–C28	1.379 (2)
C7–H7	0.9300	C27–H27	0.9300
C8–C9	1.374 (3)	C28–C29	1.376 (3)
C8–H8	0.9300	C28–H28	0.9300
C9–C10	1.378 (3)	C29–C30	1.367 (3)
C9–H9	0.9300	C29–H29	0.9300
C10–C11	1.381 (2)	C30–C31	1.388 (2)
C10–H10	0.9300	C30–H30	0.9300
C11–H11	0.9300	C31–H31	0.9300
C12–H12A	0.9600	C32–H32A	0.9600
C12–H12B	0.9600	C32–H32B	0.9600
C12–H12C	0.9600	C32–H32C	0.9600

Table A4. Continued

Angle	Angle value, °	Angle	Angle value, °
C5–N1–N2	110.46 (10)	C25–N21–C26	129.87 (10)
C5–N1–C6	129.57 (10)	N22–N21–C26	120.54 (10)
N2–N1–C6	119.97 (9)	C23–N22–N21	108.14 (11)
C3–N2–N1	105.48 (10)	C23–N22–H22	123.9 (11)
N2–C3–C4	111.05 (12)	N21–N22–H22	121.0 (11)
N2–C3–C12	120.26 (12)	N22–C23–C24	109.21 (13)
C4–C3–C12	128.67 (13)	N22–C23–C32	119.58 (13)
C5–C4–C3	105.80 (11)	C24–C23–C32	131.20 (14)
C5–C4–H4	127.9 (10)	C23–C24–C25	108.13 (12)
C3–C4–H4	126.3 (10)	C23–C24–H24	125.5 (10)
O5–C5–N1	119.74 (11)	C25–C24–H24	126.3 (10)
O5–C5–C4	133.05 (11)	O25–C25–N21	121.75 (13)
N1–C5–C4	107.21 (10)	O25–C25–C24	132.31 (12)
C5–O5–H5	107.6 (11)	N21–C25–C24	105.94 (11)
C7–C6–C11	120.33 (13)	C31–C26–C27	119.65 (14)
C7–C6–N1	120.64 (12)	C31–C26–N21	120.13 (13)
C11–C6–N1	119.01 (12)	C27–C26–N21	120.21 (12)
C8–C7–C6	119.05 (16)	C28–C27–C26	119.71 (15)
C8–C7–H7	120.5	C28–C27–H27	120.1
C6–C7–H7	120.5	C26–C27–H27	120.1
C9–C8–C7	121.06 (17)	C29–C28–C27	120.69 (17)
C9–C8–H8	119.5	C29–C28–H28	119.7
C7–C8–H8	119.5	C27–C28–H28	119.7
C8–C9–C10	119.44 (15)	C30–C29–C28	119.59 (16)
C8–C9–H9	120.3	C30–C29–H29	120.2
C10–C9–H9	120.3	C28–C29–H29	120.2
C9–C10–C11	120.72 (18)	C29–C30–C31	120.95 (17)
C9–C10–H10	119.6	C29–C30–H30	119.5
C11–C10–H10	119.6	C31–C30–H30	119.5
C10–C11–C6	119.37 (16)	C26–C31–C30	119.39 (16)
C10–C11–H11	120.3	C26–C31–H31	120.3
C6–C11–H11	120.3	C30–C31–H31	120.3
C3–C12–H12A	109.5	C23–C32–H32A	109.5
C3–C12–H12B	109.5	C23–C32–H32B	109.5
H12A–C12–H12B	109.5	H32A–C32–H32B	109.5

Table A4. Continued

C3–C12–H12C	109.5	C23–C32–H32C	109.5
H12A–C12–H12C	109.5	H32A–C32–H32C	109.5
H12B–C12–H12C	109.5	H32B–C32–H32C	109.5
C25–N21–N22	108.38 (11)	–	–
Torsion angle	**Angle value, º**	**Torsion angle**	**Angle value, º**
C5–N1–N2–C3	−0.26 (15)	C25–N21–N22–C23	4.61 (15)
C6–N1–N2–C3	−179.50 (12)	C26–N21–N22–C23	173.22 (12)
N1–N2–C3–C4	0.60 (16)	N21–N22–C23–C24	−4.54 (16)
N1–N2–C3–C12	−178.10 (14)	N21–N22–C23–C32	176.90 (14)
N2–C3–C4–C5	−0.72 (18)	N22–C23–C24–C25	2.76 (17)
C12–C3–C4–C5	177.84 (16)	C32–C23–C24–C25	−178.91 (16)
N2–N1–C5–O5	179.90 (11)	N22–N21–C25–O25	175.97 (13)
C6–N1–C5–O5	−0.9 (2)	C26–N21–C25–O25	8.8 (2)
N2–N1–C5–C4	−0.18 (15)	N22–N21–C25–C24	−2.85 (15)
C6–N1–C5–C4	178.97 (13)	C26–N21–C25–C24	−170.05 (13)
C3–C4–C5–O5	−179.57 (15)	C23–C24–C25–O25	−178.53 (16)
C3–C4–C5–N1	0.52 (16)	C23–C24–C25–N21	0.12 (16)
C5–N1–C6–C7	35.6 (2)	C25–N21–C26–C31	150.33 (14)
N2–N1–C6–C7	−145.28 (12)	N22–N21–C26–C31	−15.54 (18)
C5–N1–C6–C11	−145.78 (14)	C25–N21–C26–C27	−30.3 (2)
N2–N1–C6–C11	33.30 (17)	N22–N21–C26–C27	163.82 (12)
C11–C6–C7–C8	−0.5 (2)	C31–C26–C27–C28	−0.3 (2)
N1–C6–C7–C8	178.05 (12)	N21–C26–C27–C28	−179.65 (13)
C6–C7–C8–C9	−1.2 (2)	C26–C27–C28–C29	1.3 (2)
C7–C8–C9–C10	1.8 (3)	C27–C28–C29–C30	−1.1 (3)
C8–C9–C10–C11	−0.7 (3)	C28–C29–C30–C31	−0.3 (3)
C9–C10–C11–C6	−1.0 (2)	C27–C26–C31–C30	−1.0 (2)
C7–C6–C11–C10	1.6 (2)	N21–C26–C31–C30	178.35 (13)
N1–C6–C11–C10	−176.98 (13)	C29–C30–C31–C26	1.3 (3)

Hydrogen-bond geometry (Å, º)

D–H---A	D–H	H---A	D---A	D–H---A
O5–H5---O25	1.00 (2)	1.49 (2)	2.4794 (14)	169.8 (18)
N22–H22---N2^{i}	0.92 (2)	1.89 (2)	2.8004 (17)	170.5 (16)

Symmetry code: (i) x, $y + 1$, z.

Table A5. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for compound **II**

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.03028 (10)	0.54630 (7)	0.35235 (4)	0.0752 (3)
N1	−0.0218 (2)	0.11695 (18)	−0.17303 (11)	0.0448 (3)
N2	−0.2362 (2)	0.00890 (18)	−0.23953 (12)	0.0491 (3)
H2	−0.3709	0.0045	−0.2140	0.059*
C3	−0.1961 (3)	−0.0883 (2)	−0.35162 (15)	0.0510 (4)
C4	0.0361 (3)	−0.0560 (2)	−0.35717 (15)	0.0510 (4)
H4	0.1068	−0.1099	−0.4241	0.061*
C5	0.1557 (2)	0.0752 (2)	−0.24282 (14)	0.0487 (4)
O5	0.36892 (19)	0.14357 (19)	−0.20424 (12)	0.0616 (4)
C12	−0.3968 (3)	−0.2094 (3)	−0.44703 (18)	0.0706 (5)
H12A	−0.5131	−0.2709	−0.4079	0.106*
H12B	−0.3411	−0.3009	−0.5067	0.106*
H12C	−0.4657	−0.1344	−0.4881	0.106*
C6	−0.0052 (2)	0.21556 (19)	−0.04676 (13)	0.0431 (3)
C7	0.1996 (3)	0.3438 (2)	0.01551 (16)	0.0532 (4)
H7	0.3290	0.3629	−0.0256	0.064*
C8	0.2129 (3)	0.4441 (3)	0.13892 (17)	0.0579 (4)
H8	0.3511	0.5290	0.1816	0.070*
C9	0.0174 (3)	0.4155 (2)	0.19710 (15)	0.0544 (4)
C10	−0.1861 (3)	0.2891 (3)	0.13769 (15)	0.0565 (4)
H10	−0.3152	0.2717	0.1793	0.068*
C11	−0.1989 (3)	0.1865 (2)	0.01432 (14)	0.0520 (4)
H11	−0.3361	0.0990	−0.0270	0.062*

Note: The sign * indicates the isotropic displacement parameters, Å^2.

Table A6. Atomic displacement parameters (Å^2) for compound **II**

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0948 (5)	0.0763 (4)	0.0413 (3)	0.0189 (3)	0.0047 (3)	−0.0013 (2)
N1	0.0331 (6)	0.0553 (7)	0.0374 (6)	0.0073 (5)	0.0003 (5)	0.0038 (5)
N2	0.0356 (6)	0.0574 (7)	0.0438 (7)	0.0084 (5)	−0.0002 (5)	0.0019 (6)
C3	0.0473 (8)	0.0519 (8)	0.0433 (8)	0.0116 (6)	−0.0036 (6)	0.0002 (6)
C4	0.0460 (8)	0.0585 (8)	0.0416 (8)	0.0166 (6)	0.0047 (6)	0.0015 (6)
C5	0.0395 (7)	0.0617 (8)	0.0426 (8)	0.0147 (6)	0.0049 (6)	0.0104 (7)

Table A6. Continued

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.0345 (5)	0.0772 (8)	0.0577 (7)	0.0102 (5)	0.0046 (5)	−0.0020 (6)
C12	0.0497 (9)	0.0786 (11)	0.0591 (11)	0.0118 (9)	−0.0054 (8)	−0.0125 (9)
C6	0.0418 (8)	0.0480 (7)	0.0350 (7)	0.0106 (6)	−0.0007 (5)	0.0072 (6)
C7	0.0411 (7)	0.0565 (8)	0.0490 (8)	0.0044 (6)	0.0035 (6)	0.0010 (7)
C8	0.0499 (9)	0.0589 (9)	0.0505 (9)	0.0031 (7)	−0.0035 (7)	0.0028 (7)
C9	0.0633 (10)	0.0550 (8)	0.0410 (8)	0.0165 (7)	−0.0005 (7)	0.0085 (7)
C10	0.0531 (9)	0.0723 (10)	0.0408 (8)	0.0107 (7)	0.0099 (6)	0.0137 (7)
C11	0.0432 (8)	0.0629 (9)	0.0401 (8)	0.0023 (6)	0.0006 (6)	0.0084 (7)

Table A7. Geometric parameters (Å, º) for compound **II**

Atom–Atom	Bond length, Å	Atom–Atom	Bond length, Å
Cl1–C9	1.7562 (18)	C12–H12B	0.9600
N1–N2	1.3843 (17)	C12–H12C	0.9600
N1–C5	1.3914 (17)	C6–C7	1.382 (2)
N1–C6	1.4077 (18)	C6–C11	1.389 (2)
N2–C3	1.349 (2)	C7–C8	1.385 (2)
N2–H2	0.8600	C7–H7	0.9300
C3–C4	1.343 (2)	C8–C9	1.377 (2)
C3–C12	1.488 (2)	C8–H8	0.9300
C4–C5	1.426 (2)	C9–C10	1.364 (3)
C4–H4	0.9300	C10–C11	1.391 (2)
C5–O5	1.2440 (19)	C10–H10	0.9300
C12–H12A	0.9600	C11—H11	0.9300
Angle	**Angle value, º**	**Angle**	**Angle value, º**
N2–N1–C5	108.65 (12)	H12B–C12–H12C	109.5
N2–N1–C6	120.36 (12)	C7–C6–C11	120.05 (14)
C5–N1–C6	129.59 (12)	C7–C6–N1	120.55 (13)
C3–N2–N1	107.82 (12)	C11–C6–N1	119.38 (13)
C3–N2–H2	126.1	C6–C7–C8	120.31 (15)
N1–N2–H2	126.1	C6–C7–H7	119.8
C4–C3–N2	110.05 (15)	C8–C7–H7	119.8
C4–C3–C12	129.74 (16)	C9–C8–C7	118.70 (16)
N2–C3–C12	120.20 (15)	C9–C8–H8	120.6

Table A7. Continued

C3–C4–C5	108.29 (14)	C7–C8–H8	120.6
C3–C4–H4	125.9	C10–C9–C8	122.01 (16)
C5–C4–H4	125.9	C10–C9–Cl1	118.99 (14)
O5–C5–N1	123.33 (14)	C8–C9–Cl1	119.01 (14)
O5–C5–C4	131.64 (14)	C9–C10–C11	119.41 (15)
N1–C5–C4	105.03 (12)	C9–C10–H10	120.3
C3–C12–H12A	109.5	C11–C10–H10	120.3
C3–C12–H12B	109.5	C6–C11–C10	119.51 (15)
H12A–C12–H12B	109.5	C6–C11–H11	120.2
C3–C12–H12C	109.5	C10–C11–H11	120.2
H12A–C12–H12C	109.5	–	–
Torsion angle	**Angle value, º**	**Torsion angle**	**Angle value, º**
C5–N1–N2–C3	−4.19 (17)	C5–N1–C6–C7	24.4 (2)
C6–N1–N2–C3	−171.92 (12)	N2–N1–C6–C11	7.6 (2)
N1–N2–C3–C4	3.76 (19)	C5–N1–C6–C11	−157.28 (15)
N1–N2–C3–C12	−176.41 (16)	C11–C6–C7–C8	0.0 (3)
N2–C3–C4–C5	−1.88 (19)	N1–C6–C7–C8	178.29 (13)
C12–C3–C4–C5	178.31 (19)	C6–C7–C8–C9	−1.1 (3)
N2–N1–C5–O5	−176.12 (15)	C7–C8–C9–C10	1.4 (3)
C6–N1–C5–O5	−9.9 (3)	C7–C8–C9–Cl1	−178.39 (12)
N2–N1–C5–C4	2.98 (16)	C8–C9–C10–C11	−0.6 (3)
C6–N1–C5–C4	169.22 (14)	Cl1–C9–C10–C11	179.25 (12)
C3–C4–C5–O5	178.28 (18)	C7–C6–C11–C10	0.9 (2)
C3–C4–C5–N1	−0.72 (17)	N1–C6–C11–C10	−177.44 (14)
N2–N1–C6–C7	−170.78 (13)	C9–C10–C11–C6	−0.6 (3)

Hydrogen-bond geometry (Å, º)

D–H---A	D–H	H---A	D---A	D–H---A
N2–H2---O5^{i}	0.86	2.02	2.7293 (18)	139

Symmetry code: (i) *x*–1, *y*, *z*.

Table A8. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for compound **III**

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.48507 (6)	0.37211 (12)	0.10131 (6)	0.0388 (2)
N2	0.47599 (6)	0.54096 (11)	0.13277 (6)	0.0393 (2)

Table A8. Continued

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C3	0.38667 (7)	0.58089 (15)	0.09304 (7)	0.0399 (2)
C4	0.33980 (7)	0.43974 (16)	0.04683 (7)	0.0432 (3)
H4	0.2783	0.4342	0.0164	0.052*
O4	0.38798 (6)	0.14436 (13)	0.02542 (6)	0.0562 (3)
C5	0.40039 (7)	0.29940 (16)	0.05233 (7)	0.0397 (2)
C6	0.56111 (6)	0.26616 (14)	0.15091 (7)	0.0357 (2)
C7	0.59414 (8)	0.14975 (16)	0.11065 (8)	0.0445 (3)
H7	0.5680	0.1449	0.0520	0.053*
C8	0.66604 (8)	0.04131 (17)	0.15833 (9)	0.0490 (3)
H8	0.6882	−0.0366	0.1315	0.059*
C9	0.70529 (8)	0.04735 (17)	0.24529 (8)	0.0475 (3)
H9	0.7532	−0.0271	0.2769	0.057*
C10	0.67305 (8)	0.16467 (17)	0.28522 (8)	0.0451 (3)
H10	0.6998	0.1699	0.3439	0.054*
C11	0.60106 (7)	0.27459 (16)	0.23843 (7)	0.0403 (2)
H11	0.5796	0.3536	0.2655	0.048*
C12	0.35505 (11)	0.75872 (19)	0.10678 (10)	0.0607 (4)
H12A	0.3672	0.8489	0.0747	0.091*
H12B	0.3853	0.7889	0.1657	0.091*
H12C	0.2923	0.7533	0.0888	0.091*
C13	0.54237 (9)	0.67445 (18)	0.14061 (10)	0.0563 (3)
H13A	0.5435	0.6874	0.0874	0.085*
H13B	0.5997	0.6361	0.1827	0.085*
H13C	0.5277	0.7879	0.1569	0.085*

Note: The sign * indicates the isotropic displacement parameters, Å^2.

Table A9. Atomic displacement parameters (Å^2) for compound **III**

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0333 (4)	0.0354 (4)	0.0417 (4)	−0.0002 (3)	0.0118 (3)	−0.0046 (3)
N2	0.0368 (4)	0.0299 (4)	0.0447 (5)	−0.0007 (3)	0.0129 (4)	−0.0017 (3)
C3	0.0398 (5)	0.0369 (5)	0.0389 (5)	0.0063 (4)	0.0142 (4)	0.0074 (4)
C4	0.0340 (5)	0.0439 (6)	0.0433 (5)	0.0023 (4)	0.0102 (4)	0.0036 (4)
O4	0.0471 (5)	0.0474 (5)	0.0614 (5)	−0.0062 (4)	0.0135 (4)	−0.0189 (4)
C5	0.0352 (5)	0.0420 (5)	0.0360 (5)	−0.0028 (4)	0.0109 (4)	−0.0027 (4)

Table A9. Continued

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0303 (4)	0.0341 (4)	0.0421 (5)	−0.0017 (3)	0.0159 (4)	−0.0018 (4)
C7	0.0435 (6)	0.0450 (6)	0.0456 (6)	0.0011 (5)	0.0209 (5)	−0.0071 (5)
C8	0.0429 (6)	0.0436 (6)	0.0635 (7)	0.0030 (5)	0.0268 (5)	−0.0089 (5)
C9	0.0347 (5)	0.0413 (6)	0.0628 (7)	0.0033 (4)	0.0190 (5)	0.0029 (5)
C10	0.0384 (5)	0.0489 (6)	0.0437 (5)	0.0019 (5)	0.0148 (4)	0.0031 (5)
C11	0.0364 (5)	0.0442 (5)	0.0415 (5)	0.0019 (4)	0.0186 (4)	−0.0027 (4)
C12	0.0625 (8)	0.0443 (7)	0.0662 (8)	0.0174 (6)	0.0212 (7)	0.0044 (6)
C13	0.0495 (7)	0.0408 (6)	0.0707 (8)	−0.0107 (5)	0.0202 (6)	0.0001 (6)

Table A10. Geometric parameters (Å, º) for compound **III**

Atom–Atom	Bond length, Å	Atom–Atom	Bond length, Å
N1–C5	1.4021 (16)	C8–C9	1.379 (2)
N1–N2	1.4098 (13)	C8–H8	0.9300
N1–C6	1.4210 (16)	C9–C10	1.3823 (18)
N2–C3	1.3770 (17)	C9–H9	0.9300
N2–C13	1.4554 (16)	C10–C11	1.3854 (18)
C3–C4	1.3457 (17)	C10–H10	0.9300
C3–C12	1.4860 (18)	C11–H11	0.9300
C4–C5	1.4329 (17)	C12–H12A	0.9600
C4–H4	0.9300	C12–H12B	0.9600
O4–C5	1.2281 (15)	C12–H12C	0.9600
C6–C11	1.3883 (17)	C13–H13A	0.9600
C6–C7	1.3904 (15)	C13–H13B	0.9600
C6–C7	1.3904 (15)	C13–H13B	0.9600
C7–H7	0.9300	–	–
Angle	**Angle value, º**	**Angle**	**Angle value, º**
C5–N1–N2	108.98 (9)	C7–C8–H8	119.6
C5–N1–C6	123.53 (10)	C8–C9–C10	119.68 (11)
N2–N1–C6	118.50 (9)	C8–C9–H9	120.2
C3–N2–N1	106.22 (9)	C10–C9–H9	120.2
C3–N2–C13	121.31 (10)	C9–C10–C11	120.42 (12)
N1–N2–C13	115.25 (10)	C9–C10–H10	119.8
C4–C3–N2	110.46 (11)	C11–C10–H10	119.8

Table A10. Continued

C4–C3–C12	129.58 (12)	C10–C11–C6	119.61 (11)
N2–C3–C12	119.94 (11)	C10–C11–H11	120.2
C3–C4–C5	108.73 (11)	C6–C11–H11	120.2
C3–C4–H4	125.6	C3–C12–H12A	109.5
C5–C4–H4	125.6	C3–C12–H12B	109.5
O4–C5–N1	123.29 (11)	H12A–C12–H12B	109.5
O4–C5–C4	131.71 (11)	C3–C12–H12C	109.5
N1–C5–C4	104.94 (10)	H12A–C12–H12C	109.5
C11–C6–C7	120.02 (10)	H12B–C12–H12C	109.5
C11–C6–N1	120.88 (10)	N2–C13–H13A	109.5
C7–C6–N1	119.08 (10)	N2–C13–H13B	109.5
C8–C7–C6	119.56 (12)	H13A–C13–H13B	109.5
C8–C7–H7	120.2	N2–C13–H13C	109.5
C6–C7–H7	120.2	H13A–C13–H13C	109.5
C9–C8–C7	120.70 (11)	H13B–C13–H13C	109.5
C9–C8–H8	119.6	–	–
Torsion angle	**Angle value, º**	**Torsion angle**	**Angle value, º**
C5–N1–N2–C3	−8.50 (11)	C3–C4–C5–O4	174.74 (13)
C6–N1–N2–C3	−156.86 (9)	C3–C4–C5–N1	−2.69 (13)
C5–N1–N2–C13	−145.85 (11)	C5–N1–C6–C11	−112.19 (12)
C6–N1–N2–C13	65.80 (14)	N2–N1–C6–C11	31.29 (14)
N1–N2–C3–C4	6.83 (12)	C5–N1–C6–C7	66.27 (14)
C13–N2–C3–C4	141.00 (12)	N2–N1–C6–C7	−150.25 (10)
N1–N2–C3–C12	−174.78 (10)	C11–C6–C7–C8	0.83 (17)
C13–N2–C3–C12	−40.61 (17)	N1–C6–C7–C8	−177.64 (11)
N2–C3–C4–C5	−2.62 (13)	C6–C7–C8–C9	0.02 (18)
C12–C3–C4–C5	179.20 (12)	C7–C8–C9–C10	−0.78 (19)
N2–N1–C5–O4	−170.83 (11)	C8–C9–C10–C11	0.70 (18)
C6–N1–C5–O4	−24.42 (17)	C9–C10–C11–C6	0.15 (18)
N2–N1–C5–C4	6.87 (11)	C7–C6–C11–C10	−0.91 (17)
C6–N1–C5–C4	153.29 (10)	N1–C6–C11–C10	177.53 (10)