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Crystal structures of 5,5′-bis(hydroxymethyl)-3,3′-biisoxazole and 4,4′,5,5′-tetrakis(hydroxymethyl)-3,3′-biisoxazole

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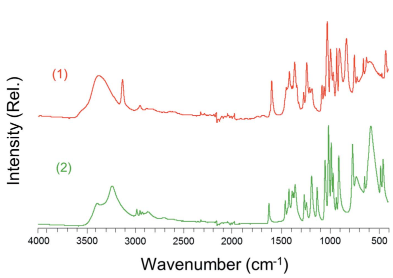
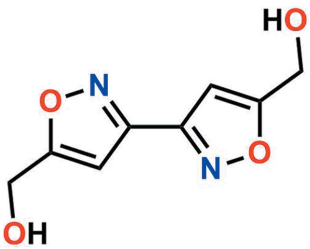
The molecular structure of 5,5′-bis(hydroxymethyl)-3,3′-biisoxazole, $C_8H_8N_2O_4$ (**1**), is composed of two *trans* planar isoxazole rings [r.m.s deviation = 0.006 (1) Å], each connected with a methyl hydroxyl group. Similarly, the structure of 4,4′,5,5′-tetrakis(hydroxymethyl)-3,3′-biisoxazole, $C_{10}H_{12}N_2O_6$ (**2**), is composed of two planar isoxazole rings [r.m.s. deviation = 0.002 (1) Å], but with four hydroxymethyl groups as substituents. Both molecules sit on a center of inversion, thus Z' = 0.5. The crystal structures are stabilized by networks of O—H···N [for (**1**)] and O—H···O hydrogen-bonding interactions [for (**2**)], giving rise to corrugated supramolecular planes. The isoxazole rings are packed in a slip-stacked fashion, with centroid-to-centroid distances of 4.0652 (1) Å for (**1**) (along the *b*-axis direction) and of 4.5379 (Å) for (**2**) (along the *a*-axis direction).

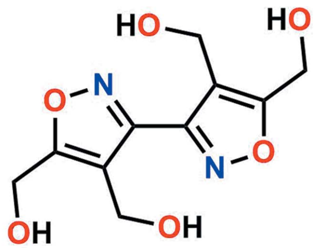


1. Chemical context

The five-membered, heterocyclic isoxazole moiety forms the basis for a number of medical and agricultural products, as well as energetic materials (Galenko *et al.*, 2015; Sausa *et al.*, 2017; Wingard *et al.*, 2017*a*,*b*; Sysak & Obmińska-Mrukowicz, 2017). Its versatility stems from the electronegative oxygen and nitrogen atoms, which provide the ring nucleophilic activity, and its three carbon atoms, which afford the addition of a variety of functional groups. The title compounds 5,5′-bis(hydroxymethyl)-3,3′-biisoxazole (**1**) and 4,4′,5,5′-tetrakis-(hydroxymethyl)-3,3′-biisoxazole (**2**) exhibit two isoxazole rings, each attached with one or two hydroxymethy groups. These compounds have been synthesized recently in our laboratory as useful precursors to a new class of energetic materials. The addition of nitric acid to the title compounds results in nitrate esterification, yielding the energetic materials biisoxazolebis(methylene dinitrate) (**3**) and biisoxazoletetrakis(methyl nitrate) (**4**), where a nitrate functional group replaces the hydrogen atom in the hydroxyl groups (Wingard *et al.*, 2017*a*,*b*). These derivative compounds are potential energetic plasticizing ingredients in nitrocellulose or melt-castable formulations because the rings present Lewis-base behavior towards electrophilic nitrocellulose and the alkyl nitric esters afford miscibility and compatibility with conventional energetic plasticizers.



(1)



(2)

2. Structural commentary

The title compounds exhibit molecular structures typical of biisoxazole derivatives. Fig. 1 reveals that the isoxazole rings of (**1**) exhibit a *trans* planar configuration [r.m.s deviation = 0.0009 (1) Å], suggesting a delocalized aromatic π system. The C4 atom is nearly coplanar with the ring (atom-to-mean plane distance = 0.006 Å), whereas the C4—O2 bond is twisted slightly out of the plane, as evidenced by the torsion angles C2—C1—C4—O2 = −13.3 (2)° and O1—C1—C4—O2 = 167.55 (11)°. Atoms C1/C4/O2 form a plane that subtends a dihedral angle of 12.72 (1)° with respect to the isoxazole ring. Similarly, the isoxazole rings of (**2**) are nearly planar [r.m.s deviation = 0.002 (1) Å]; however, the corresponding O2—C4 bond is twisted more out of plane than that of compound (**1**), as evidenced by the magnitude of the torsion angle O2—C4—C1—O1 = −54.93 (11)°. For comparison, the torsion angle formed by atoms O3—C5—C2—C1 is −110.02 (11)°. The atoms O2/C4/C1 and O3/C5/C2 form planes subtending dihedral angles of 53.78 (8) and 69.37 (7)° with respect to the isoxazole ring. Superimposition of the ring atoms of both structures (see Fig. 2) yields an r.m.s. deviation of 0.01 Å. Finally, compound (**2**) exhibits a weak intramolecular interaction involving atoms O3—H3*A* and N1^{iii} [see Table 2 for the geometrical parameters; symmetry code: (iii) = $-x + 2, -y + 1, -z + 1$.]

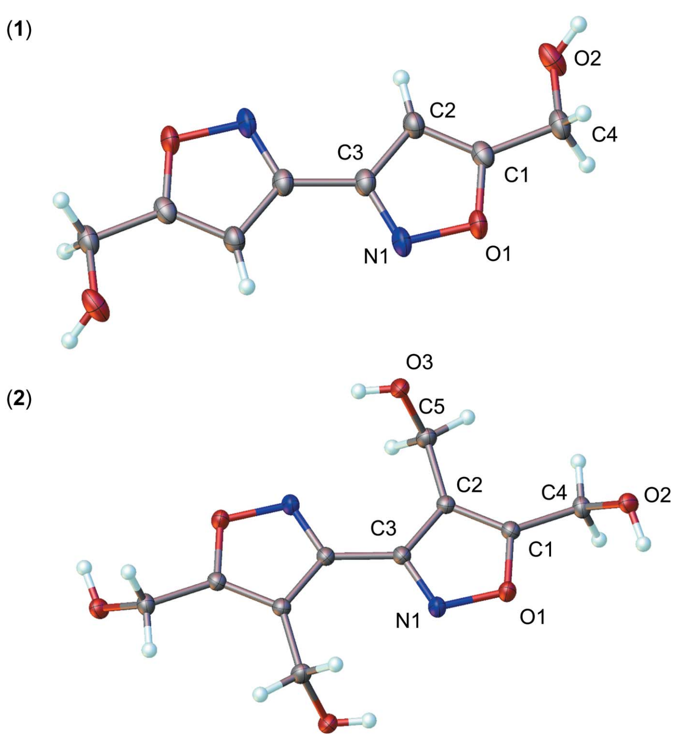


Figure 1
Molecular conformation and atom-numbering scheme of compounds (**1**) and (**2**). Non-labeled atoms of both structures are generated by inversion ($-x + 2$, $-y + 1$, $-z + 1$). Non-hydrogen atoms are shown as 50% probability displacement ellipsoids.

Table 1
Hydrogen-bond geometry (Å, °) for (**I**).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N1^{i}	0.82	2.03	2.8461 (15)	171

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °) for (**II**).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3^{i}	0.849 (18)	1.849 (18)	2.6936 (11)	172.8 (16)
O3—H3*A*···O2^{ii}	0.792 (19)	2.085 (19)	2.7898 (11)	148.3 (18)
O3—H3*A*···N1^{iii}	0.792 (19)	2.550 (19)	3.0728 (12)	125.0 (16)

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x + 1, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x + 2, -y + 1, -z + 1$.

3. Supramolecular features

Intermolecular hydrogen bonding plays a key role in the stabilization of the crystal structures of the title compounds. Figs. 3 and 4 show the packing of (**1**) and (**2**), respectively, and Tables 1 and 2 list their hydrogen-bonding geometries. Compound (**1**) displays hydrogen bonding between the oxygen atoms O2, belonging to the hydroxy groups, and the N1 atoms of the isoxazole rings of adjacent molecules, generating a supramolecular framework parallel to ($\overline{2}01$) [O2···N1^{i} = 2.8461 (15) Å; symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$]. In contrast, compound (**2**) forms a network of hydrogen bonds involving the hydroxy groups O2—H2*A* and O3—H3*A* of adjacent molecules, so that each OH group acts both as

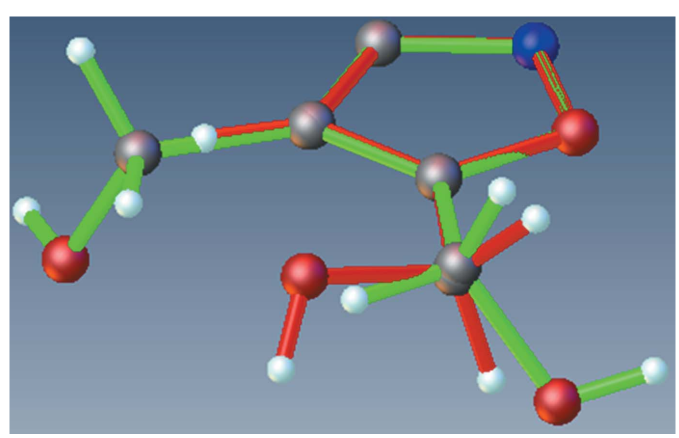


Figure 2
An overlay of the asymmetric units of compounds (**1**) and (**2**), depicted in red and green, respectively.

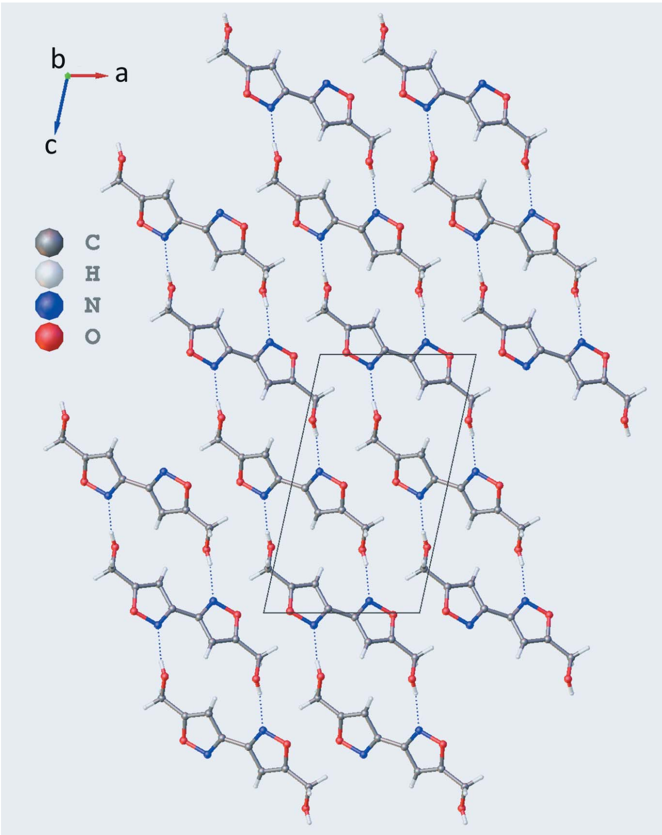


Figure 3
Crystal packing of (**1**) viewed along the *a*-axis direction. Dashed lines represent O2—H2*A*···N1[i] hydrogen bonds; symmetry code: (i) $x - \frac{1}{2}$, $-y + \frac{1}{2}$, $z - \frac{1}{2}$.

donor and acceptor [see Table 2 and Fig. 4; O2···O3[i] = 2.694 (1) Å; symmetry code: (i) $-x + 1$, $y + \frac{1}{2}$, $-z + \frac{1}{2}$; O3···O2[ii] = 2.790 (1) Å; symmetry code: (ii) $x + 1$, $-y + \frac{1}{2}$, $z + \frac{1}{2}$]. In this way, each molecule forms eight hydrogen bonds with the four closest surrounding analogues, giving rise to corrugated planes parallel to ($\overline{1}02$).

The crystal structure of (**1**) reveals a slip-stacked geometry of the rings in the *b*-axis direction, with centroid-to-centroid distances of 4.0652 (1) Å and plane-to-plane shifts of 2.256 (2) Å. In contrast, in compound (**2**) the rings are stacked along the *a*-axis direction, with centroid-to-centroid distances of 4.5379 (4) Å and plane-to-plane shifts of 2.683 (2) Å.

4. Database survey

A search of the Cambridge Structural Database (CSD web interface, December 2017; Groom *et al.*, 2016) and the Crystallography Open Database (Gražulis *et al.*, 2009) yielded the crystal structures of several compounds containing the biisoxazole moiety. For examples, see Cannas & Marongiu (1967) (CCDC 1111317, BIOXZL); van der Peet *et al.* (2013) (CCDC 935274, LIRLEF); Sausa *et al.* (2017) (CCDC 1540757, TAXDUU); Wingard *et al.* (2017*b*) (CCDC 1529260, WANVEP). Compounds (**3**) (Sausa *et al.*, 2017) and (**4**) (Wingard *et al.* 2017*b*) are noteworthy because they are nitrate derivatives of the title compounds (**1**) and (**2**), respectively, with the hydrogen atoms in the OH groups replaced by NO_2 moieties. A superimposition of the respective isoxazole rings of compound (**1**) and (**3**) yields an r.m.s. deviation of 0.004 Å (Fig. 5A). In both molecules, the rings adopt a *trans* conformations; however, in (**1**) the O1 and O2 atoms are in a *trans* conformation with respect to the C1—C4 bond, whereas in (**3**) the corresponding O atoms are in a *cis* conformation. In (**1**), the plane encompassing the atoms O2, C4, and C1 forms a dihedral angle of 12.72 (1)° with respect to the mean plane of the isoxazole ring, in contrast to a value of 66.8 (2)° in (**3**) for the corresponding atoms. A similar comparison between (**2**) and (**4**) yields an r.m.s. deviation of 0.01 Å for the superimposition of the isoxazole rings, and dihedral angles of 53.78 (8) and 69.37 (7)° for (**2**) (planes formed by the atoms O2/C4/C1 and O3/C5/C2, respectively) compared to those of 84.54 (14) and 84.81 (18)° or 79.19 (15) and 82.32 (17)° for (**4**) (Fig. 5B). The most striking supramolecular difference between the title compounds and (**3**) and (**4**) is that the former exhibit hydrogen bonding, which contributes to the stability of their crystal structure.

5. Synthesis and crystallization

The synthesis of the title compounds has been reported recently (Wingard *et al.*, 2017*a,b*). Briefly, they were prepared

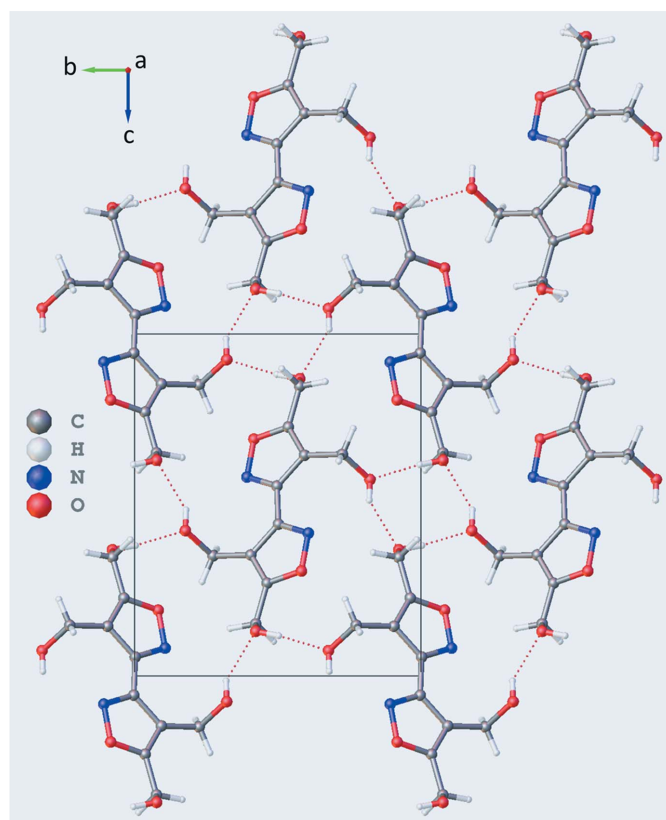


Figure 4
Crystal packing of (**2**) viewed along the *a*-axis direction. Dashed lines represent O2—H2*A*···O3[i] and O3—H3*A*···O2[ii] hydrogen bonds; symmetry codes: (i) $-x + 1$, $y + \frac{1}{2}$, $-z + \frac{1}{2}$; (ii) $x + 1$, $-y + \frac{1}{2}$, $z + \frac{1}{2}$.

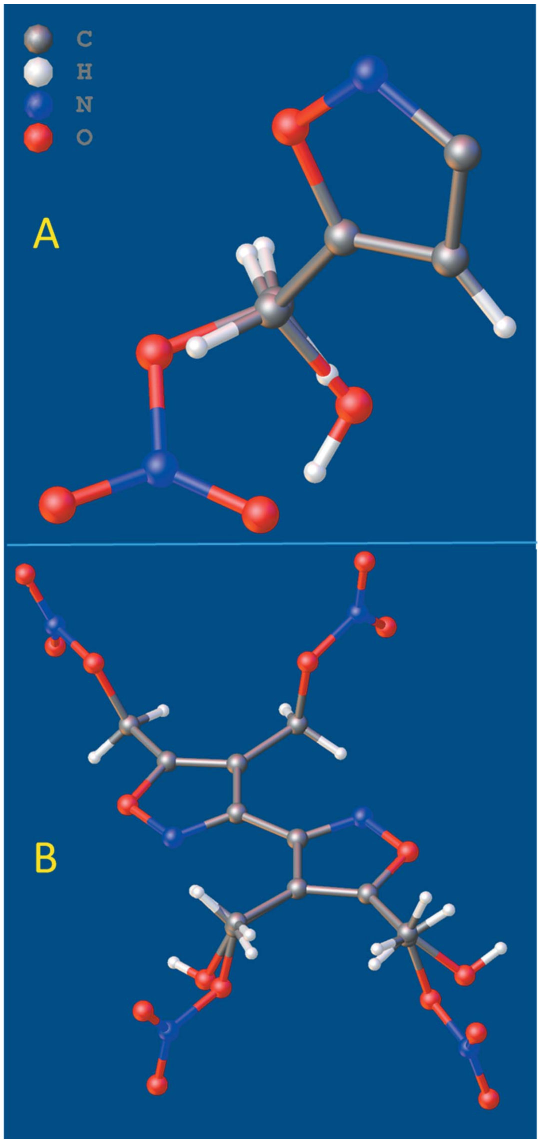


Figure 5
Overlays of the asymmetric units of (**1**) and (**3**) (A) and (**2**) and (**4**) (B).

by [3 + 2] cycloaddition of dichloroglyoxime and alcohol. In the case of compound (**1**), a saturated solution of sodium bicarbonate was added to a solution of dichloroglyoxime (30 g), propargyl alcohol (55.2 ml), and methanol (1900 ml) over 6 h. Once the reaction was complete, the product was stirred for an additional 10 h and the remaining solvent evaporated. A yield of 75% was obtained after the product was washed with distilled water, collected by Büchner filtration, and then dried. Compound (**2**) was prepared by adding dropwise a dichloroglyoxime and butyl alcohol solution (0.8 *M*) to a refluxing solution comprising $NaHCO_3$ (6.7 g), 2-butyne-1,4-diol (13.72 g), and butyl alcohol (200 ml). Once the reaction was complete, the product was cooled to room temperature and the remaining solvent evaporated. Then, the product was washed with distilled water, filtered, and dried, resulting in a yield of 68%. Slow solvent evaporation of the title compounds in methanol yielded suitable single crystals for the X-ray diffraction experiments at 150K. We note the title compounds have nearly the same density (1.596 *vs* 1.597 Mg m^{-3}), given that their molecular mass and cell constants are quite different.

Fig. 6 shows the FTIR spectra of (**1**) and (**2**) recorded with a Nicolet iS50 spectrophotometer, using attenuated total reflectance. The intense peak frequencies (cm^{-1}) are listed as follows: Compound (**1**): 3371.83, 3126.65, 1596.96, 1415.14, 1360.62, 1268.13, 1237.16, 1080.70, 1058.61, 1026.40, 993.24, 929.53, 901.95, 828.87, 746.83, 653.69, 621.96, and 424.11. Compound (**2**): 3234.89, 1623.59, 1456.55, 1418.41, 1354.66, 1261.30, 1185.44, 1128.41, 1046.52, 1011.82, 984.07, 964.14, 931.24, 906.80, 764.50, 725.86, 641.00, 576.90, 475.85, and 449.97.

6. Refinement

Crystal data, data collection, structure solution and refinement details are summarized in Table 3. The hydrogen atoms for compound (**1**) were refined using a riding model with C—H = 0.93 or 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ and O—H = 0.74–0.85 Å and $U_{iso}(H) = 1.5U_{eq}(O)$, whereas for compound (**2**) all the hydrogen atoms were refined independently including isotropic displacement parameters.

Acknowledgements

We thank Dr D. Taylor of the US Army Research Laboratory and Dr Eric Reinheimer of Rigaku for helpful suggestions regarding this work.

References

Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* A**71**, 59–75.
Bruker (2010). *APEX2*, *SAINT* and *XSHELL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cannas, M. & Marongiu, G. (1967). *Z. Kristallogr.* **124**, 143–151.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

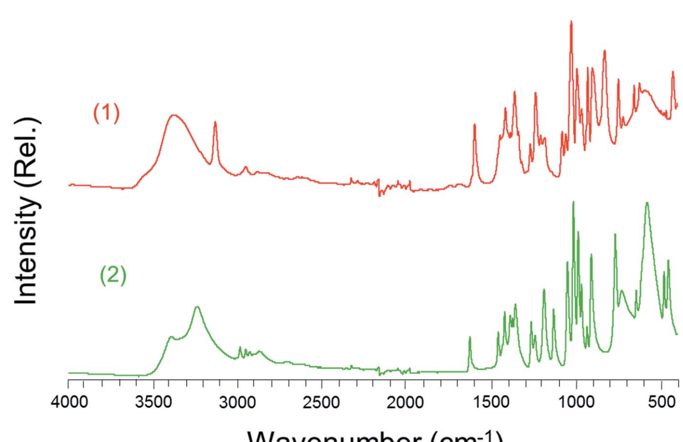


Figure 6
FTIR spectra of the title compounds.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_8H_8N_2O_4$	$C_{10}H_{12}N_2O_6$
M_r	196.16	256.22
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	150	150
a, b, c (Å)	7.7824 (3), 4.0652 (1), 13.2109 (5)	4.5379 (4), 9.9195 (8), 12.0177 (9)
β (°)	102.334 (4)	99.9312 (11)
V (Å^3)	408.31 (2)	532.86 (8)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.13	0.13
Crystal size (mm)	0.35 × 0.25 × 0.05	0.49 × 0.20 × 0.11
Data collection		
Diffractometer	Rigaku Oxford DiffractionSuperNova, Dualflex, EosS2	Bruker SMART APEXII CCD
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015; Bourhis *et al.*, 2015)	Multi-scan (*SADABS*; Sheldrick, 2008)
T_{min}, T_{max}	0.207, 1.000	0.904, 0.985
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3474, 823, 754	7638, 1737, 1570
R_{int}	0.027	0.019
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.624	0.730
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.086, 1.04	0.034, 0.072, 1.00
No. of reflections	823	1737
No. of parameters	66	106
H-atom treatment	H-atom parameters constrained	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.15	0.44, −0.22

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *APEX2*, *XSHELL* and *SAINT* (Bruker, 2010), *SHELXT* (Sheldrick, 2015*a*), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

Galenko, A. V., Khlebnikov, A. F., Novikov, M. F., Pakalnis, V. V. & Rostovskii, N. V. (2015). *Russ. Chem. Rev.* **84**, 335–377.
Gražulis, S., Chateigner, D., Downs, R. T., Yokochi, A. F. T., Quirós, M., Lutterotti, L., Manakova, E., Butkus, J., Moeck, P. & Le Bail, A. (2009). *J. Appl. Cryst.* **42**, 726–729.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Peet, P. L. van der, Connell, T., Gunawan, C., White, J., Donnelly, P. & Williams, S. (2013). *J. Org. Chem.* **78**, 7298–7304.
Rigaku OD (2015). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, England.
Sausa, R. C., Pesce-Rodriguez, R. A., Wingard, L. A., Guzmán, P. E. & Sabatini, J. J. (2017). *Acta Cryst.* E**73**, 644–646.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Sysak, A. & Obmińska-Mrukowicz, B. (2017). *Eur. J. Med. Chem.* **137**, 292–309.
Wingard, L. A., Guzmán, P. E., Johnson, E. C., Sabatini, J. J., Drake, G. W. & Byrd, E. F. C. (2017*a*). *ChemPlusChem*, **82**, 195-198.
Wingard, L. A., Johnson, E. C., Guzmán, P. E., Sabatini, J. J., Drake, G. W., Byrd, E. F. C. & Sausa, R. C. (2017*b*). *Eur. J. Org. Chem.* pp. 1765–1768.

supporting information

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Crystal structures of 5,5′-bis(hydroxymethyl)-3,3′-biisoxazole and 4,4′,5,5′-tetrakis(hydroxymethyl)-3,3′-biisoxazole

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015) for (1); *APEX2* (Bruker, 2010) for (2). Cell refinement: *CrysAlis PRO* (Rigaku OD, 2015) for (1); *SAINT* (Bruker, 2010) for (2). Data reduction: *CrysAlis PRO* (Rigaku OD, 2015) for (1); *SAINT* (Bruker, 2010) for (2). Program(s) used to solve structure: SHELXT (Sheldrick, 2015a) for (1); *SHELXS97* (Sheldrick, 2008) for (2). Program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b) for (1); *SHELXL2014* (Sheldrick, 2015b) for (2). Molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) for (1); *XSHELL* (Bruker, 2010) for (2). Software used to prepare material for publication: *Mercury* (Macrae *et al.*, 2008) for (1); *PLATON* (Spek, 2009) for (2).

5,5′-Dihydroxymethyl-3,3′-biisoxazole (1)

Crystal data

$C_8H_8N_2O_4$
M_r = 196.16
Monoclinic, $P2_1/n$
a = 7.7824 (3) Å
b = 4.0652 (1) Å
c = 13.2109 (5) Å
β = 102.334 (4)°
V = 408.31 (2) Å^3
Z = 2
$F(000)$ = 204
D_x = 1.596 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1980 reflections
θ = 2.8–26.2°
μ = 0.13 mm^{-1}
T = 150 K
Block, colorless
0.35 × 0.25 × 0.05 mm

Data collection

Rigaku Oxford DiffractionSuperNova, Dualflex, EosS2 diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.0945 pixels mm^{-1}
ω scans
Absorption correction: multi-scan CrysAlisPro (Rigaku OD, 2015; Bourhis *et al.*, 2015)
T_{min} = 0.207, T_{max} = 1.000
3474 measured reflections
823 independent reflections
754 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.3°, θ_{min} = 2.8°
h = −9→9
k = −5→5
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.086$
$S = 1.03$
823 reflections
66 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2 + 0.183P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: SHELXL-2016/4 (Sheldrick 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.041 (8)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.64217 (17)	0.3456 (3)	0.39844 (9)	0.0193 (3)
C2	0.78918 (17)	0.5023 (3)	0.38810 (10)	0.0207 (3)
H2	0.808128	0.621413	0.331370	0.025*
C3	0.90869 (16)	0.4449 (3)	0.48325 (9)	0.0188 (3)
C4	0.46651 (17)	0.3002 (3)	0.32830 (10)	0.0230 (3)
H4A	0.453788	0.076229	0.302446	0.028*
H4B	0.373826	0.343734	0.365286	0.028*
N1	0.83872 (14)	0.2665 (3)	0.54646 (8)	0.0238 (3)
O1	0.66636 (12)	0.2010 (2)	0.49285 (7)	0.0240 (3)
O2	0.45577 (14)	0.5241 (3)	0.24526 (7)	0.0304 (3)
H2A	0.412548	0.431590	0.190584	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0199 (7)	0.0198 (7)	0.0165 (6)	0.0029 (5)	0.0004 (5)	−0.0032 (5)
C2	0.0194 (7)	0.0238 (7)	0.0174 (6)	0.0007 (5)	0.0009 (5)	−0.0005 (5)
C3	0.0173 (7)	0.0209 (7)	0.0174 (6)	0.0021 (5)	0.0019 (5)	−0.0029 (5)
C4	0.0190 (7)	0.0249 (7)	0.0226 (7)	−0.0005 (5)	−0.0009 (5)	−0.0054 (5)
N1	0.0173 (6)	0.0315 (7)	0.0198 (6)	−0.0029 (5)	−0.0019 (4)	−0.0002 (5)
O1	0.0180 (5)	0.0319 (6)	0.0199 (5)	−0.0052 (4)	−0.0008 (4)	0.0003 (4)
O2	0.0342 (6)	0.0275 (6)	0.0230 (5)	−0.0011 (4)	−0.0087 (4)	−0.0024 (4)

Geometric parameters (Å, º)

C1—C2	1.3419 (19)	C3—N1	1.3093 (17)
C1—C4	1.4901 (17)	C4—H4A	0.9700
C1—O1	1.3550 (15)	C4—H4B	0.9700

C2—H2	0.9300	C4—O2	1.4142 (17)
C2—C3	1.4143 (18)	N1—O1	1.4021 (14)
C3—C3^{i}	1.465 (2)	O2—H2A	0.8200
C2—C1—C4	133.19 (12)	C1—C4—H4A	110.3
C2—C1—O1	110.24 (11)	C1—C4—H4B	110.3
O1—C1—C4	116.57 (11)	H4A—C4—H4B	108.5
C1—C2—H2	127.9	O2—C4—C1	107.28 (11)
C1—C2—C3	104.14 (11)	O2—C4—H4A	110.3
C3—C2—H2	127.9	O2—C4—H4B	110.3
C2—C3—C3^{i}	129.00 (15)	C3—N1—O1	105.45 (10)
N1—C3—C2	111.97 (11)	C1—O1—N1	108.21 (10)
N1—C3—C3^{i}	119.03 (14)	C4—O2—H2A	109.5
C1—C2—C3—C3^{i}	179.69 (17)	C3—N1—O1—C1	0.23 (14)
C1—C2—C3—N1	−0.03 (15)	C4—C1—C2—C3	−179.05 (14)
C2—C1—C4—O2	−13.3 (2)	C4—C1—O1—N1	179.11 (11)
C2—C1—O1—N1	−0.26 (14)	O1—C1—C2—C3	0.18 (14)
C2—C3—N1—O1	−0.12 (15)	O1—C1—C4—O2	167.55 (11)
C3^{i}—C3—N1—O1	−179.87 (14)		

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N1^{ii}	0.82	2.03	2.8461 (15)	171

Symmetry code: (ii) $x-1/2, -y+1/2, z-1/2$.

4,4′,5,5′-Tetrahydroxymethyl-3,3′-biisoxazole (2)

Crystal data

$C_{10}H_{12}N_2O_6$
M_r = 256.22
Monoclinic, $P2_1/c$
a = 4.5379 (4) Å
b = 9.9195 (8) Å
c = 12.0177 (9) Å
β = 99.9312 (11)°
V = 532.86 (8) Å^3
Z = 2
F(000) = 268
D_x = 1.597 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3988 reflections
θ = 2.7–32.1°
μ = 0.13 mm^{-1}
T = 150 K
Needle, colourless
0.49 × 0.20 × 0.11 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2008)
T_{min} = 0.904, T_{max} = 0.985
7638 measured reflections
1737 independent reflections
1570 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 31.3°, θ_{min} = 2.7°
h = −6→6
k = −14→14
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.072$
$S = 1.00$
1737 reflections
106 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.010P)^2 + 0.3955P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.58044 (17)	0.58291 (7)	0.30718 (6)	0.01981 (16)
O2	0.23448 (16)	0.42300 (8)	0.13078 (6)	0.01890 (15)
H2A	0.174 (4)	0.5035 (18)	0.1182 (14)	0.039 (4)*
O3	0.93528 (19)	0.17777 (8)	0.42532 (7)	0.02252 (17)
H3A	1.037 (4)	0.1801 (18)	0.4858 (16)	0.045 (5)*
N1	0.7277 (2)	0.60707 (9)	0.41811 (7)	0.02010 (18)
C1	0.6765 (2)	0.46292 (10)	0.27170 (8)	0.01576 (17)
C2	0.8810 (2)	0.40630 (9)	0.35428 (8)	0.01513 (17)
C3	0.9048 (2)	0.50244 (10)	0.44419 (8)	0.01585 (18)
C4	0.5528 (2)	0.42337 (10)	0.15275 (8)	0.01753 (18)
H4A	0.631 (3)	0.4856 (14)	0.1016 (12)	0.022 (3)*
H4B	0.622 (3)	0.3337 (14)	0.1391 (11)	0.021 (3)*
C5	1.0376 (2)	0.27405 (10)	0.35134 (9)	0.01875 (19)
H5A	1.255 (3)	0.2866 (14)	0.3717 (12)	0.024 (3)*
H5B	0.992 (3)	0.2362 (14)	0.2760 (11)	0.021 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0240 (4)	0.0166 (3)	0.0163 (3)	0.0036 (3)	−0.0036 (3)	−0.0010 (3)
O2	0.0163 (3)	0.0169 (3)	0.0216 (3)	−0.0003 (3)	−0.0019 (3)	−0.0014 (3)
O3	0.0302 (4)	0.0154 (3)	0.0187 (4)	−0.0019 (3)	−0.0050 (3)	0.0026 (3)
N1	0.0253 (4)	0.0171 (4)	0.0153 (4)	0.0022 (3)	−0.0038 (3)	−0.0019 (3)
C1	0.0171 (4)	0.0143 (4)	0.0154 (4)	−0.0012 (3)	0.0015 (3)	−0.0003 (3)
C2	0.0167 (4)	0.0136 (4)	0.0148 (4)	−0.0008 (3)	0.0017 (3)	0.0001 (3)
C3	0.0183 (4)	0.0139 (4)	0.0145 (4)	−0.0012 (3)	0.0003 (3)	0.0001 (3)
C4	0.0171 (4)	0.0199 (4)	0.0147 (4)	−0.0007 (3)	0.0001 (3)	−0.0006 (3)
C5	0.0219 (4)	0.0161 (4)	0.0176 (4)	0.0030 (3)	0.0015 (3)	−0.0006 (3)

Geometric parameters (Å, º)

O1—N1	1.4050 (11)	C3—C2	1.4312 (13)
O2—H2A	0.849 (18)	C3—C3^{i}	1.4657 (18)
O2—C4	1.4229 (12)	C4—C1	1.4952 (13)
O3—H3A	0.792 (19)	C4—H4A	0.980 (14)
N1—C3	1.3171 (12)	C4—H4B	0.966 (14)
C1—O1	1.3612 (12)	C5—O3	1.4354 (13)
C1—C2	1.3577 (13)	C5—H5A	0.982 (14)
C2—C5	1.4953 (13)	C5—H5B	0.969 (14)
C1—O1—N1	108.71 (7)	O2—C4—C1	112.33 (8)
C4—O2—H2A	108.4 (11)	O2—C4—H4A	110.6 (8)
C5—O3—H3A	110.4 (13)	O2—C4—H4B	108.3 (8)
C3—N1—O1	105.20 (8)	C1—C4—H4A	108.5 (8)
O1—C1—C4	116.17 (8)	C1—C4—H4B	108.9 (8)
C2—C1—O1	110.38 (8)	H4A—C4—H4B	108.1 (11)
C2—C1—C4	133.38 (9)	O3—C5—C2	111.30 (8)
C1—C2—C3	103.26 (8)	O3—C5—H5A	110.3 (8)
C1—C2—C5	127.83 (9)	O3—C5—H5B	106.3 (8)
C3—C2—C5	128.91 (9)	C2—C5—H5A	110.1 (8)
N1—C3—C2	112.45 (8)	C2—C5—H5B	109.7 (8)
N1—C3—C3^{i}	118.88 (11)	H5A—C5—H5B	109.0 (11)
C2—C3—C3^{i}	128.67 (11)		
O1—N1—C3—C2	−0.53 (11)	C1—C2—C5—O3	−110.02 (11)
O1—N1—C3—C3^{i}	179.26 (10)	C2—C1—O1—N1	0.05 (11)
O1—C1—C2—C3	−0.35 (10)	C3—C2—C5—O3	68.82 (13)
O1—C1—C2—C5	178.72 (9)	C3^{i}—C3—C2—C1	−179.21 (12)
O2—C4—C1—O1	−54.93 (11)	C3^{i}—C3—C2—C5	1.74 (19)
O2—C4—C1—C2	128.39 (11)	C4—C1—O1—N1	−177.38 (8)
N1—C3—C2—C1	0.56 (11)	C4—C1—C2—C3	176.48 (10)
N1—C3—C2—C5	−178.49 (10)	C4—C1—C2—C5	−4.46 (18)
C1—O1—N1—C3	0.30 (10)		

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3^{ii}	0.849 (18)	1.849 (18)	2.6936 (11)	172.8 (16)
O3—H3*A*···O2^{iii}	0.792 (19)	2.085 (19)	2.7898 (11)	148.3 (18)
O3—H3*A*···N1^{i}	0.792 (19)	2.550 (19)	3.0728 (12)	125.0 (16)

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