STRUCTURAL CHEMISTRY

ISSN 2053-2296

Effect of molecular perturbation on cocrystal formation: theophylline and its 8-halo analogues with flavonoids

Weijian Ye, Herman H-Y. Sung and Ian D. Williams*

Department of Chemistry, The Hong Kong University of Science and Technology, Clear Water Bay, Hong Kong SAR, People's Republic of China. *Correspondence e-mail: chwill@ust.hk

Received 10 March 2026
Accepted 23 June 2026

Edited by T. Ohhara, J-PARC Center, Japan Atomic Energy Agency, Japan

Keywords: crystal structure; cocrystal; alkaloid; flavonoid; theophylline; hydrogen bond; myricetin; kaempferol.

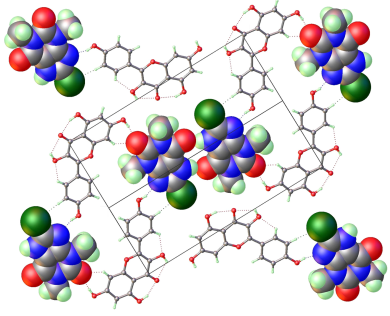
CCDC references: 2564329; 2564328; 2564327; 2564326; 2564325; 2564324; 2564323; 2564322; 2564321

Supporting information: this article has supporting information at journals.iucr.org/c

The tendency for cocrystal formation between alkaloids and flavonoids is found to be quite common (around 30%) and based on the optimization of hydrogen-bond formation between the acceptor-rich alkaloids and donor-rich flavonoids. Within each molecular family there is substantial variation in cocrystal tendency, leading to considerable scope for selective cocrystal precipitation to assist in the separation of complex mixtures of these natural product families. Theophylline ($C_7H_8N_4O_2$) follows caffeine and theacrine in forming a substantial number of flavonoid cocrystals – six in total, of which three were reported previously. By contrast, the 8-halo-substituted analogues 8-*X*-Tph [*X* = Cl ($C_7H_7ClN_4O_2$), Br ($C_7H_7BrN_4O_2$)] form cocrystals with just myricetin ($C_{15}H_{10}O_8$) and kaempferol ($C_{15}H_{10}O_6$). Weakening of the acceptor capability of the alkaloid N9 atom upon proximal halo substitution may play a role in the reduced tendency of 8-*X*-Tph towards cocrystal formation, since improved packing efficiency is not a key factor in the formation of these cocrystals. Most of the cocrystal phases may be conveniently prepared with good yield and purity through either liquid-assisted grinding (LAG) or microwave-assisted cocrystallization from 1-butanol.

1. Introduction

The formation of cocrystals of natural products has been of considerable recent interest. For those molecules with bioactivity that can be considered as pharmaceuticals, there is the associated interest of potential improvement in oral delivery, enhanced dissolution, bioavailability, solid-state stability, mechanical properties, tabletability, hygroscopic properties and avoidance of hydrate formation (Trask *et al.*, 2005; Good & Rodríguez-Hornedo, 2009; Chadha *et al.*, 2017; Zhu *et al.*, 2017). Cocrystals may also have important intellectual property implications (Sharma *et al.*, 2025). The report that cocrystal formation between caffeine and various flavonoids could lead to their enhanced separation *via* green technology based on differential solubility of the cocrystals was intriguing (Xia *et al.*, 2021). If such natural product mixtures could be separated based on differences in cocrystal solubility, how much more effective if the mixture was subject to a differential tendency to form such cocrystals in the first place? We thus investigated the general tendency of alkaloids to form crystals with flavonoids in a survey of co-former pairs. This was done relatively efficiently by screening for cocrystal formation *via* a liquid-assisted grinding (LAG) approach (Trask & Jones, 2005). This was helpful in screening for cocrystal formation between the highly potent antimalarial agent 11-azaartemisinin with a variety of coformers, including many carboxylic acids (Nisar *et al.*, 2018; Roy *et al.*, 2021; Li *et al.*, 2022). Successful cocrystal formation is quickly checked by running



powder X-ray diffractograms of the resulting powders after co-grinding for several hours. This practically ensures that for successful combinations, when a change in the powder X-ray diffraction (PXRD) pattern is observed, the resulting cocrystals represent thermodynamically preferred solid forms.

In order to find the structure and stoichiometry of such cocrystal products, a rational and consistent crystallization approach was also sought that could be rapidly tested and applied to successful coformer pair combinations. After various efforts, a standard methodology of heating/cooling aqueous methanolic solutions over a period of 6–12 h was adopted. The result for around 250 total combinations of common alkaloids and common flavonoids is that 75 cocrystal phases were readily isolable and have been characterized by single-crystal X-ray structure determination (Ye, 2024).

In this article, we report the comparative results of cocrystal formation with flavonoids for the alkaloid theophylline compared to its 8-halo analogues (Fig. 1). Recently, we reported the crystal structures of 8-Cl-Tph and 8-Br-Tph, and found four unreported forms that were structurally distinct from the polymorphs of the parent theophylline (Ye *et al.*, 2025). The marked switch of structure types appeared strongly linked to the electronic modification of the Tph molecule upon introduction of the 8-halo substituent. This renders the resulting protonated cations $[Tph\text{-}H]^+$ more acidic, but also lowers the basicity of the ring N9 atom that is adjacent to the halo substitution site. This apparently leads to a switch of polymorph hydrogen-bond preference from N—H···N found in most Tph polymorphs to N—H···O found in 8-*X*-Tph. We were curious to explore whether this electronic perturbation, which was supported by our DFT calculations, might also effect cocrystal formation for these related alkaloids.

The cocrystals reported here are: theophylline–kaempferol (1/1) (**1a**), theophylline–myricetin–water (2/2/1) (**1c**), theophylline–*rac*-hesperetin (2/1) (**1e**), 8-chlorotheophylline–kaempferol (1/1) (**2a**), 8-chlorotheophylline–myricetin (1/1) (**2c**), 8-bromotheophylline–kaempferol (**3a**), 8-bromotheophylline–myricetin (1/1) (**3c**), 8-bromotheophylline–kaempferol–methanol (1/1/1) (**3a′**). We also report the previously unpublished structure of kaempferol monohydrate (**a·H_2O**).

2. Experimental

2.1. Isolation and crystallization

2.1.1. General

The theophylline alkaloids (**1**–**3**), flavonoids (**a**–**f**) (Fig. 1) and solvents used were of reagent grade supplied by Meryer Chemicals or TCI Chemicals (Shanghai) with the following details: (**1**) theophylline, 99%, CAS 58-55-9, $C_7H_8N_4O_2$, M_r 180.17; (**2**) 8-chlorotheophylline, >98%, CAS 85-18-7, $C_7H_7ClN_4O_2$, M_r 214.61; (**3**) 8-bromotheophylline, 97%, CAS 10381-75-6, $C_7H_7BrN_4O_2$, M_r 259.06. Flavonoids resulting in the successful isolation of cocrystals are shown in Fig. 1 and are: (**a**) kaempferol hydrate, 98%, CAS 520-18-3, $C_{15}H_{10}O_6\cdot xH_2O$, M_r 286.24 (anhydrous); (**b**) quercetin dihydrate, 97%, CAS 6151-25-3, $C_{15}H_{10}O_7\cdot 2H_2O$, M_r 338.27; (**c**) myricetin, 97%, CAS 529-44-2, $C_{15}H_{10}O_8$, M_r 318.24; (**d**) baicalein, 98%, CAS 491-67-8, $C_{15}H_{10}O_5$, M_r 270.24; (**e**) *rac*-hesperetin, >97%, CAS 520-33-2, $C_{16}H_{14}O_6$, M_r 302.28; (**f**) *rac*-dihydromyricetin,

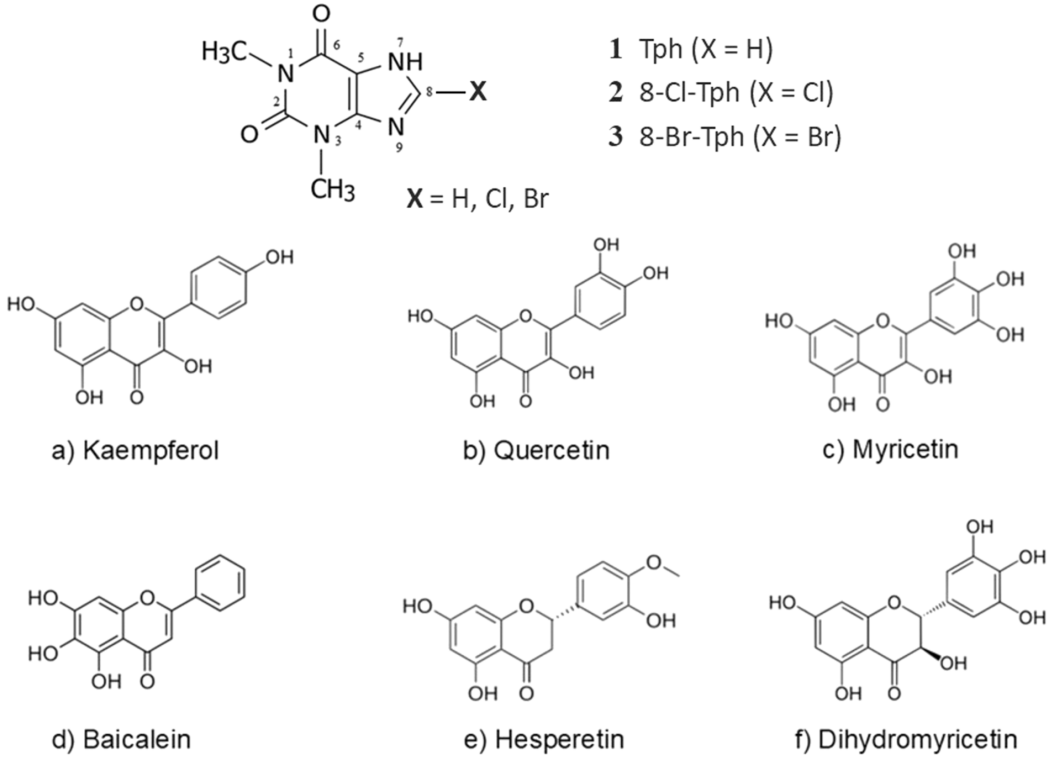


Figure 1
Structural scheme for theophyllines (**1**–**3**) and flavonoids (**a**–**f**) used in the cocrystal phases.

Table 1
Experimental details.

Experiments were carried out at 100 K.

	1a	**1c**	**1e**
Crystal data			
Chemical formula	$C_7H_8N_4O_2{\cdot}C_{15}H_{10}O_6$	$2C_7H_8N_4O_2{\cdot}2C_{15}H_{10}O_8{\cdot}H_2O$	$2C_7H_8N_4O_2{\cdot}C_{16}H_{14}O_6$
M_r	466.41	1014.82	662.62
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
a, b, c (Å)	6.51941 (9), 7.77386 (10), 39.0816 (5)	7.3270 (1), 32.4057 (4), 17.8062 (2)	6.8286 (2), 14.9790 (3), 16.0763 (4)
α, β, γ (°)	90, 92.4920 (12), 90	90, 97.827 (1), 90	88.624 (2), 83.176 (2), 87.020 (2)
V (Å^3)	1978.82 (5)	4188.46 (9)	1630.25 (7)
Z	4	4	2
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	1.03	1.12	0.88
Crystal size (mm)	0.2 × 0.2 × 0.2	0.1 × 0.02 × 0.01	0.05 × 0.03 × 0.01
Data collection			
Diffractometer	Agilent SuperNova Dual Source diffractometer with an Atlas detector	Agilent SuperNova Dual Source diffractometer with an Atlas detector	Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.807, 1.000	0.568, 1.000	0.935, 1.000
No. of measured, independent and observed reflections	13045, 4130, 3800 [$I \geq 2\sigma(I)$]	25287, 8359, 5887 [$I > 2\sigma(I)$]	26501, 6746, 6048 [$I > 2\sigma(I)$]
R_{int}	0.023	0.055	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.633	0.625	0.632
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.095, 1.06	0.049, 0.118, 0.99	0.048, 0.125, 1.05
No. of reflections	4130	8359	6746
No. of parameters	329	726	469
No. of restraints	0	0	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.27	0.58, −0.27	0.26, −0.19

	2a	**2c**	**3a**
Crystal data			
Chemical formula	$C_7H_7ClN_4O_2{\cdot}C_{15}H_{10}O_6$	$C_7H_7ClN_4O_2{\cdot}C_{15}H_{10}O_8$	$C_7H_7BrN_4O_2{\cdot}C_{15}H_{10}O_6$
M_r	500.85	532.84	545.30
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$
a, b, c (Å)	10.14353 (15), 19.7196 (3), 10.42202 (19)	7.7588 (5), 10.6363 (7), 13.4366 (7)	10.1578 (1), 19.8059 (3), 10.4534 (1)
α, β, γ (°)	90, 93.0362 (14), 90	78.246 (5), 81.294 (5), 81.946 (5)	90, 92.539 (1), 90
V (Å^3)	2081.75 (6)	1066.24 (12)	2101.00 (4)
Z	4	2	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	2.18	2.24	3.22
Crystal size (mm)	0.2 × 0.08 × 0.04	0.22 × 0.15 × 0.1	0.2 × 0.1 × 0.08
Data collection			
Diffractometer	Agilent SuperNova Dual Source diffractometer with an Atlas detector	Agilent SuperNova Dual Source diffractometer with an Atlas detector	Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.705, 1.000	0.481, 1.000	0.871, 1.000
No. of measured, independent and observed reflections	12088, 4177, 3533 [$I > 2\sigma(I)$]	6593, 4156, 3656 [$I > 2\sigma(I)$]	12439, 4220, 3606 [$I > 2\sigma(I)$]
R_{int}	0.030	0.029	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.626	0.626	0.625
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.100, 1.03	0.038, 0.105, 1.02	0.032, 0.086, 1.06
No. of reflections	4177	4156	4220
No. of parameters	338	364	334
No. of restraints	0	0	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.38	0.33, −0.38	0.42, −0.43

Table 1 (continued)

	3c	3a′	a·H_2O
Crystal data			
Chemical formula	$C_7H_7BrN_4O_2·C_{15}H_{10}O_8$	$C_7H_7BrN_4O_2·C_{15}H_{10}O_6·CH_4O$	$C_{15}H_{10}O_6·H_2O$
M_r	577.30	577.35	304.25
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Monoclinic, $C2/c$
a, b, c (Å)	7.7841 (3), 10.7050 (5), 13.4516 (6)	9.9847 (5), 10.6290 (4), 12.1537 (4)	27.7113 (11), 3.7151 (2), 24.7282 (11)
α, β, γ (°)	78.741 (4), 81.454 (4), 81.657 (4)	67.509 (4), 81.274 (3), 73.675 (4)	90, 98.208 (2), 90
V (Å^3)	1079.31 (8)	1142.27 (9)	2519.7 (2)
Z	2	2	8
Radiation type	Cu $K\alpha$	Mo $K\alpha$	Ga $K\alpha$, λ = 1.34139 Å
μ (mm^{-1})	3.25	1.86	0.70
Crystal size (mm)	0.09 × 0.06 × 0.05	0.3 × 0.2 × 0.1	0.03 × 0.03 × 0.03
Data collection			
Diffractometer	Agilent SuperNova Dual Source diffractometer with an Atlas detector	Agilent SuperNova Dual Source diffractometer with an Atlas detector	Bruker APEXII CCD
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.899, 1.000	0.615, 1.000	0.658, 0.751
No. of measured, independent and observed reflections	6335, 4197, 3614 [$I > 2\sigma(I)$]	7430, 4532, 4048 [$I > 2\sigma(I)$]	11000, 2557, 1890 [$I > 2\sigma(I)$]
R_{int}	0.026	0.035	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.626	0.622	0.626
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.097, 1.04	0.029, 0.066, 1.05	0.040, 0.110, 1.06
No. of reflections	4197	4532	2557
No. of parameters	342	358	224
No. of restraints	0	0	0
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.08, −0.58	0.49, −0.41	0.19, −0.17

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022, 2023), *APEX2* (Bruker, 2016), *SAINT* (Bruker, 2016), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXT2018/2019* (Sheldrick, 2015*a*), *olex2.refine* (Bourhis *et al.*, 2015), *SHELXL2019* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

97%, CAS 27200-12-0, $C_{15}H_{12}O_8$, M_r 320.25. The use of the flavonoids fisetin, luteolin, genistein, naringenin, biochanin a, chrysin or rutin was unsuccessful for cocrystal formation with these theophylline alkaloids.

2.1.2. Cocrystal screening and growth

Screening for cocrystallization between alkaloids 8-*X*-Tph (*X* = H, Br, Cl) and flavonoids was carried out by the LAG of 1:1 mixtures (Trask & Jones, 2005). Grinding used a Tencan XQM-0.4A mini-planetary ball mill with zirconia vessels and media (Nisar *et al.*, 2018). A minimal amount of methanol (η factor = 0.2 ml g^{-1}) was used to accelerate the solid-state transformations, which typically took between 30 min and 2 h.

Single crystals of alkaloid–flavonoid cocrystal phases were also grown by a standard approach of solvothermal cocrystallization (Karimi-Jafari *et al.*, 2018) using 0.25 mmol each of the alkaloid (45–65 mg) with the flavonoid (70–85 mg) in aqueous methanol comprising 2 ml MeOH and 0.1 ml H_2O. Solutions were heated in 23 ml Teflon-lined Parr-type pressure vessels at 120 °C and under autogenous pressure for 12–24 h, followed by slow cooling back to ambient conditions. Solids were filtered off, washed with cold water and ethanol, dried and inspected under an optical microscope. The cocrystal phases prepared in this manner had typical isolated yields of 40–60 mg (65–85% yield based on the limiting reagent).

All cocrystals displayed a 1:1 stoichiometry except for theophylline–baicalein (2:1) and theophylline–hesperetin (2:1). A previously reported theophylline–dihydromyricetin 1:1 ACN solvate phase from acetonitrile (Sun *et al.*, 2023) was prepared successfully from a modification of the cocrystallization solvent to 1:1 MeCN–H_2O. No change of PXRD pattern emerged in this system upon LAG or solvothermally using MeOH. Finally, cocrystal formation using microwave-assisted cocrystallization (Ahuja *et al.*, 2020) was adopted using a Biotage Initiator+ and a 2∼5 ml reaction vial. Most phases isolable by solvothermal or LAG methods could be prepared phase pure in a fast and convenient manner in high yield (80–90%) on a 1 mmol scale in 2 ml 1-butanol (20 min heating at 140 °C and 20 min cooling).

2.2. X-ray crystallography

Crystal data, data collection and structure refinement details are summarized in Table 1. Organic H atoms were placed geometrically and treated with riding constraints and displacement parameters derived from the C atoms to which they were attached. All CH and CH_2 groups had U_{iso}(H) values fixed at 1.2 times the U_{eq} of the parent C atom. Methyl groups were idealized as freely rotating CH_3 groups and U_{iso}(H) values were fixed at 1.5 times the U_{eq} of the parent C atom. H atoms on N atoms of the Tph alkaloids and H atoms

on O atoms of the flavonoids were located in difference maps and refined with individual isotropic displacement parameters. Disorder, for example in the case of racemic hesperetin and dihydromyricetin, was handled using *OLEX2* by defining separate parts, which assists maintaining separate geometry, appropriate bonding connectivity and riding H atoms (Nisar *et al.*, 2018).

3. Results and discussion

3.1. Background – alkaloid–flavonoid cocrystal formation

A search of the Cambridge Structural Database (CSD; Groom *et al.*, 2016) reveals that the structures of a considerable number of cocrystal phases between common alkaloid and flavonoid molecules have been reported. Most of the previous studies have concentrated on the xanthine alkaloids, such as caffeine (Trask *et al.*, 2005) and its demethylated analogues theobromine (Sanphui & Nangia, 2014) and theophylline (Trask *et al.*, 2006). A variety of crystallization methodologies were adopted in these various studies (Karimi-Jafari *et al.*, 2018; Sakhiya & Borkhataria, 2024). A report on the use of caffeine cocrystals to effect a 'green technology' separation of a mixture of common flavonoids, namely, myricetin, quercetin and baicalein [see Figs. 1(*b*)–(*d*)], based on differential solubility of the cocrystals formed, was quite noteworthy (Xia *et al.*, 2021).

We sought to investigate this issue further with a more general investigation of alkaloid–flavonoid cocrystal formation using two steps. The first is an LAG screening through co-grinding of starting materials, using a small amount of solvent to help the process. The polar protic solvent methanol is suitable as a common 'sparing' solvent for many compounds in both the alkaloid and the flavonoid families. Typically, grinding over a period of 3 h is then sufficient to determine whether any change will occur under these conditions and the resulting powders can be inspected by powder X-ray diffraction (PXRD). Examples of successful cocrystal formation are given in Fig. 2 for the molecular pairs theophylline–myricetin (**1c**, Tph–Myr) and 8-chlorotheophylline–kaempferol (**2a**, 8-Cl-Tph–Kmp).

In cases where a clear PXRD change is seen, follow-up crystallizations using a heating–cooling co-precipitation approach were undertaken. A mixed aqueous methanolic solution was heated solvothermally for several hours to effect full dissolution of the co-formers and then cooled slowly to afford cocrystal formation, typically in agreement with LAG-prepared phases with specimens of suitable size for crystal structure determination. We have carried out this process in around 250 alkaloid–flavonoid binary combinations, resulting in the isolation of 75 cocrystal phases (Ye, 2024). These results indicate a strong tendency for cocrystal formation between the two molecular families. This is readily ascribed to the propensity for more optimal hydrogen-bonding arrangements in the cocrystal phases, with alkaloids being rich in hydrogen-bond-acceptor moieties and flavonoids being rich in hydrogen-bond-donor groups. This was borne out by the variable tendency to cocrystal formation based on the degree to which these statements applied to individual molecules. Notwithstanding, all alkaloid and flavonoid molecules studied were found to form at least one cocrystal phase (Ye, 2024).

In order to check the packing efficiencies for cocrystals *versus* their co-formers, we undertook the structure determination of all co-formers studied at a common temperature of 100 K. In many cases, the structures for these common compounds are well established and, in some cases, several polymorphic forms have been reported. We were surprised, however, that 8-halotheophyllines (*X* = Br, Cl) were unreported and hence carried out new structure determinations for these (Ye *et al.*, 2025). Several new polymorphs were uncovered for these, which were all distinct from those for the parent molecule theophylline despite the perturbation of just one atom in the molecule (Fig. 1). Our density functional theory (DFT) calculations supported the notion that these structural changes came from lowering the basicity of the N9 atom. Electrostatic potential (ESP) calculations indicate a lowering of charge on N9 from −0.62 in Tph itself to −0.52 in 8-Cl-Tph and to −0.46 in 8-Br-Tph (Ye *et al.*, 2025). This supports the observed switch from N7—H···N9 hydrogen-bonded polymorphs for theophylline (Tph) to N7—H···O hydrogen-bonded polymorphs for 8-*X*-Tph. We speculated that this change might affect cocrystal formation of 8-*X*-Tph with flavonoids as well.

The powder X-ray diffractograms from LAG of stoichiometric ratios of the alkaloid and flavonoid were measured and

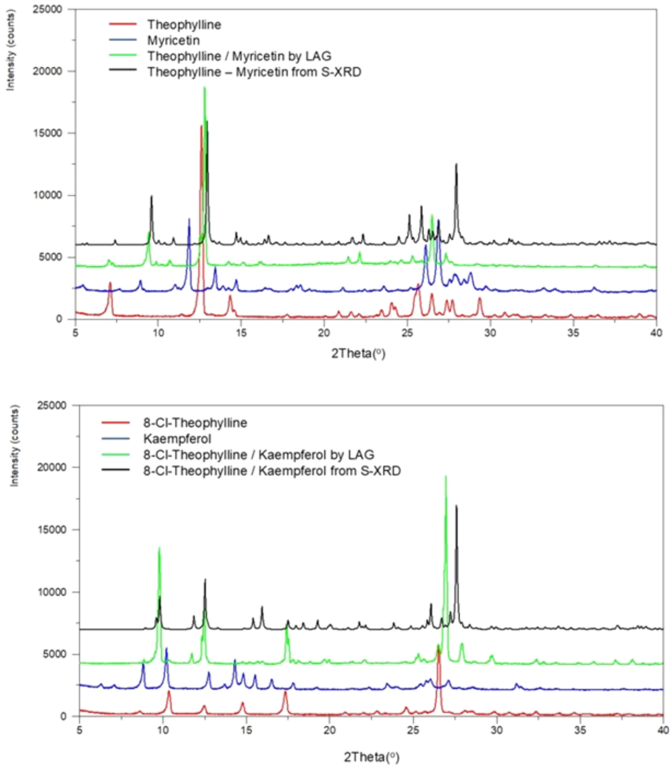


Figure 2
Powder X-ray diffractograms for alkaloid–flavonoid molecular pairs. Examples from the theophylline–myricetin (**1c**; upper) and 8-chlorotheophylline–kaempferol (**2a**, lower) systems.

Table 2
Theophylline (Tph)–flavonoid cocrystal phases.

Phase	Formula	Cocrystal System	Comments	Reference
1a	$C_{22}H_{18}N_4O_8$	Tph–kaempferol	R_1 = 3.78%	This work
1b	$C_{22}H_{18}N_4O_9{\cdot}0.5H_2O$	Tph–quercetin	$0.5H_2O$	*a*
1c	$C_{22}H_{18}N_4O_{10}{\cdot}0.5H_2O$	Tph–myricetin	$0.5H_2O$, R_1 = 4.87%	This work
1d	$C_{29}H_{26}N_8O_9{\cdot}3H_2O$	Tph–baicalein (2/1)	$3H_2O$	*b*
1e	$C_{30}H_{30}N_8O_{10}{\cdot}xH_2O$	Tph–*rac*-hesperetin (2/1)	Solvate, R_1 = 4.85	This work
1f	$C_{22}H_{20}N_4O_{10}{\cdot}CH_3CN$	Tph–dihydromyricetin	MeCN solvate	*c*

References: (*a*) Wang *et al.* (2022) (CSD refcode JATPIH); (*b*) Zhu *et al.* (2017) (CSD refcode KAMQIB); (*c*) Sun *et al.* (2023) (CSD refcode NEXSIW).

compared with the starting reagents and the simulated patterns of the isolated and structurally characterized cocrystals. Given sufficient grind time, product patterns matched well to those later simulated from single-crystal structure determinations, after taking into account the temperatures of measurement (Fig. 2).

3.2. Cocrystals of theophylline (Tph) and flavonoids

Three cocrystal phases of theophylline (Tph) have been reported previously (Wang *et al.*, 2022; Zhu *et al.*, 2017; Sun *et al.*, 2023). We were able to prepare all such phases in a reasonably phase-pure manner, by use of both LAG and our heating–cooling co-precipitation process. In all, around 15 common flavonoids were screened for cocrystal formation with Tph. A total of six cocrystal phases were isolated using the flavonoids depicted in Fig. 1, namely, kaempferol (**a**), quercetin (**b**), myricetin (**c**), baicalein (**d**), hesperetin (**e**) and dihydromyricetin (**f**), which are listed in Table 2. The previously reported phases (CSD refcodes JATPIH, KAMQIB and NEXSIW) will not be discussed in further detail here and references are given in Table 2. It may be noted that the phases may be of 1:1 or 2:1 stoichiometry, and often hydrated. NEXSIW is an acetonitrile solvate. Use of MeOH and our 'standard' method of cocrystallization yielded no other cocrystal phase in this system. Crystal data summaries at 100 K for the three new theophylline–flavonoid cocrystal phases **1a**, **1c** and **1e** are given in Table 1.

Our first unreported cocrystal phase from theophylline is produced with kaempferol and an unsolvated 1:1 phase is formed, so the asymmetric unit is a molecular pair, shown in Fig. 3. The phase Tph–Kmp (**1a**) forms a relatively complex packing, that results in the net hydrogen-bonding interactions to neighbours shown in Fig. 4. Each theophylline is hydrogen bonded to four kaempferol neighbours and *vice versa*. Three hydrogen bonds are donated by the flavonoid and one by the alkaloid, so there is *net hydrogen bonding* from one entity to the other, as postulated in our general concept of why these molecular classes should have a tendency to cocrystal formation with each other.

Of the other newly prepared cocrystal phases, Tph–myricetin is a hemihydrate that crystallizes in the space group $P2_1/c$. The asymmetric unit comprises two theophylline and two myricetin molecules and a water molecule (Fig. 5) The structure is notable in that the two independent Tph molecules form a hydrogen-bonded dimer *via* two N7—H···O6′ hydrogen bonds that create an $R_2^2(10)$ ring (for this topological notation of hydrogen bonds, see Etter *et al.*, 1990).

In the various polymorphs of theophylline itself, this is not thermodynamically favoured, though it is found in one metastable polymorph (Dyulgerov *et al.*, 2015). When viewed along the shorter 7.3 Å *a* axis, isolated molecular stacks of Tph dimers (space-filled) are seen surrounded by six myricetin stacks (Fig. 6). Overall each Tph dimer pair accepts four hydrogen bonds from phenolic groups on the two myricetins. The water acts as both a hydrogen-bond donor and acceptor to the

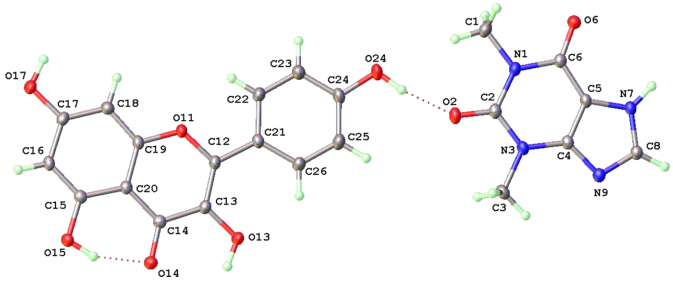


Figure 3
The molecular pair for theophylline–kaempferol (**1a**), showing the atom-labelling scheme (50% probability displacement ellipsoids).

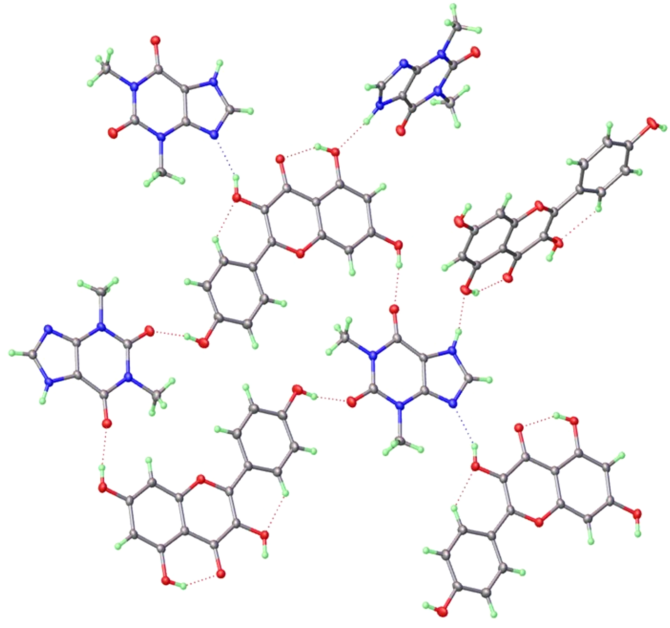


Figure 4
The packing environment for theophylline–kaempferol (**1a**), showing the alkaloid as net hydrogen-bond acceptor and the flavonoid as net hydrogen-bond donor.

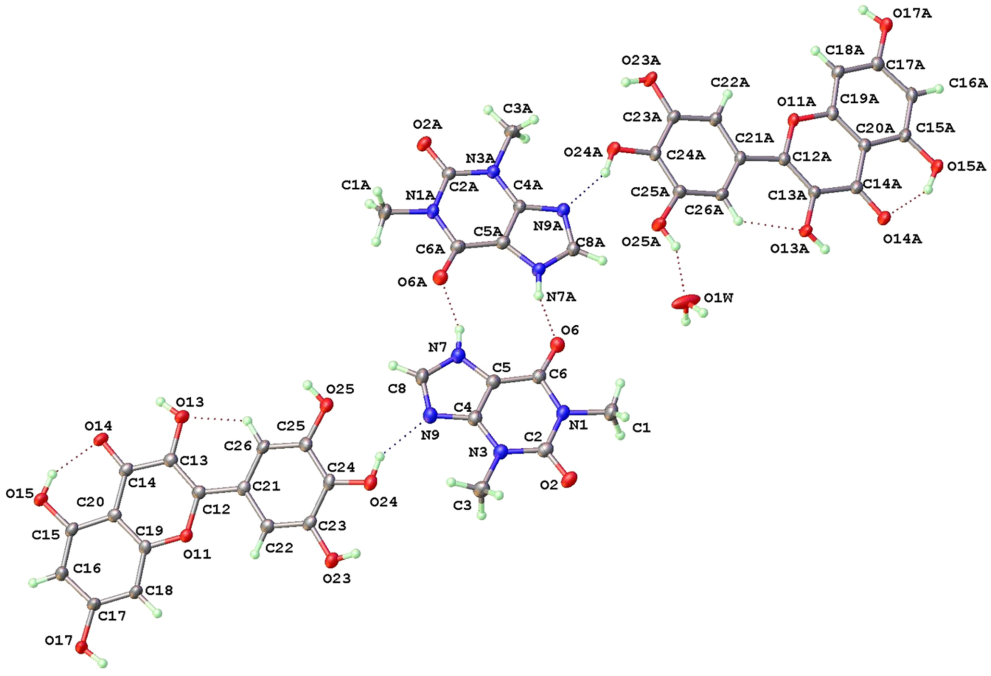


Figure 5
The asymmetric unit for theophylline–myricetin hemihydrate (**1c**), showing the atom-labelling scheme (50% probability displacement ellipsoids).

two myricetin molecules and breaks the pseudosymmetry that can be seen within the asymmetric unit (Fig. 5).

Finally, one further cocrystal phase that we have isolated from theophylline and flavonoids is the solvated 2:1 cocrystal theophylline–*rac*-hesperetin (**1e**). Hesperetin is a flavonoid with a single chiral centre and is found naturally as hesperidin, a rutinoside (6-*O*-α-L-rhamnosyl-D-glucoside), in which the chirality is 2*S* (Pryzynska, 2022). However, following isolation, it is usually offered commercially as a racemate. In this case, racemic hesperetin was cocrystallized with theophylline. The product phase crystallizes in the triclinic space group $P\overline{1}$ with a 2:1 ratio of alkaloid to flavonoid and a roughly 70:30 disorder of the racemic hesperetin at a single crystallographic site. In the asymmetric unit shown in Fig. 7, the chiral centre is 70%*R* C12 and 30%*S* C12*A*. The opposite enantiomeric ratio will be found at the inverted site of the unit cell. Attempted refinement in chiral *P*1 space group failed to improve the modelling indicating there is no spontaneous resolution of the *rac*-hesperetin in this cocrystal phase. The phase is highly solvated and, when viewed along the short 6.8 Å *a* axis, the structure reveals large channel voids of roughly 7 × 10 Å dimension (Fig. 8). The application of SQUEEZE (Spek, 2015) indicates around 37 e^- for a contiguous accessible void of around 112 Å

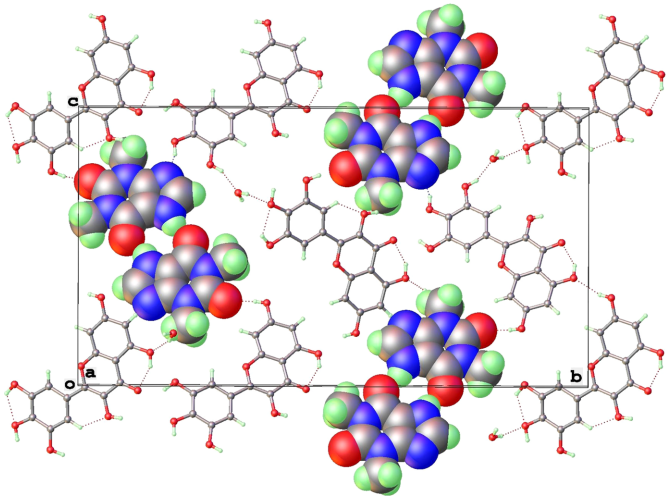


Figure 6
Packing diagram for theophylline–myricetin hemihydrate (**1c**), viewed along the *a* axis.

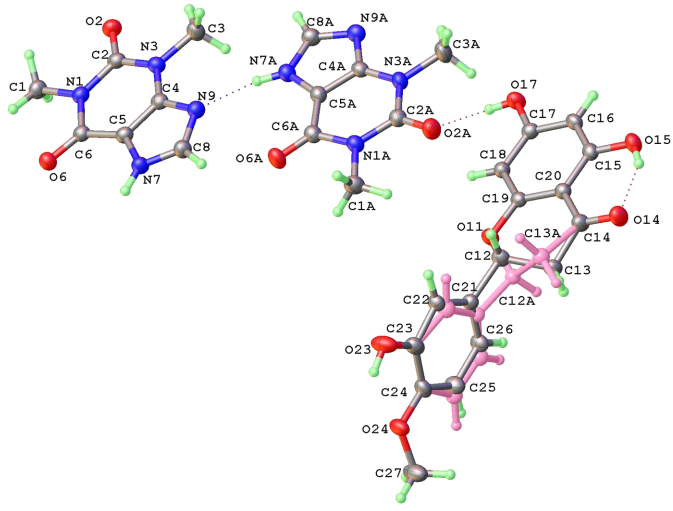


Figure 7
The asymmetric unit for theophylline–*rac*-hesperetin 2:1 cocrystal (**1e**), with the solvent excluded. The minor disordered component (30%) is shown in pink.

Table 3
8-Halotheophylline (8-*X*-Tph)–flavonoid cocrystal phases reported in this article.

Phase	Formula	Cocrystal system	Comments
2a	$C_{22}H_{17}ClN_4O_8$	8-Cl-Tph–kaempferol	R_1 3.61%
2c	$C_{22}H_{17}ClN_4O_{10}$	8-Cl-Tph–myricetin	R_1 3.82%
3a	$C_{22}H_{17}BrN_4O_8$	8-Br-Tph–kaempferol	R_1 3.18%
3a′	$C_{22}H_{17}BrN_4O_8{\cdot}CH_4O$	8-Br-Tph–kaempferol MeOH	R_1 2.86%
3c	$C_{22}H_{17}BrN_4O_{10}$	8-Br-Tph–myricetin	R_1 3.86%

per asymmetric unit. The disordered solvent is compatible with a combination of two waters and one methanol molecule.

3.3. Flavonoid cocrystal formation for 8-chloro and 8-bromotheophylline

No structural reports existed for 8-chloro- or 8-bromotheophylline polymorphs or cocrystals in the CSD. However, the well-known drug dimenhydrinate (Dramamine) is the diphenhydramine salt of the 8-Cl-Tph anion, which was structurally determined (Putra *et al.*, 2016). Recently, we have reported the polymorphic forms of 8-Cl-Tph and 8-Br-Tph (Ye *et al.*, 2025). Interestingly, these structures exclusively favoured N—H···O hydrogen bonding between the alkaloid molecules, that we believed was due to a reduction in N9 basicity. Since the introduction of the 8-halo substituent affected the structural polymorphism of these theophylline derivatives, we also wanted to see the potential effect on cocrystal formation. Accordingly, the same 15 flavonoids were screened for cocrystal formation with 8-*X*-Tph as for Tph. This time the only flavonoids affording cocrystals with 8-*X*-Tph were kaempferol (**a**) and myricetin (**c**). The phases produced were found by PXRD to be isostructural between the 8-Cl-

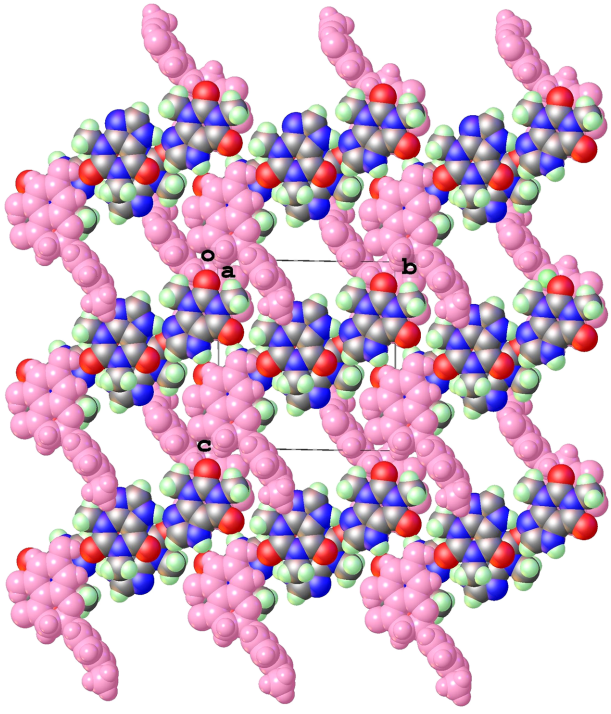


Figure 8
Space-filling diagram of the theophylline–*rac*-hesperetin 2:1 cocrystal (**1e**), viewed along the *a* axis, showing large open solvent channels. The hesperetin molecules are shaded in pink.

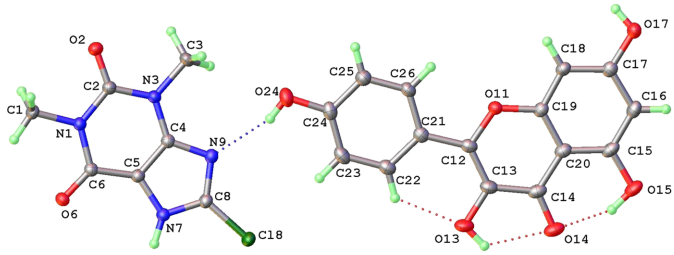


Figure 9
The labelling scheme for the 8-Cl-theophylline–kaempferol 1:1 cocrystal (**2a**), similarly adopted by the isostructural 8-Br analogue **3a**.

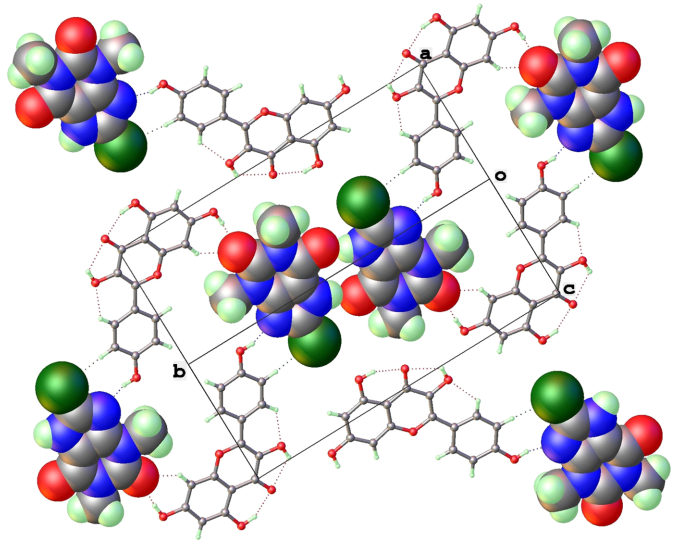


Figure 10
Packing diagram for the 8-Cl-theophylline–kaempferol 1:1 cocrystal (**2a**), viewed along [101] and showing 8-Cl-Tph dimers (space-filled molecules).

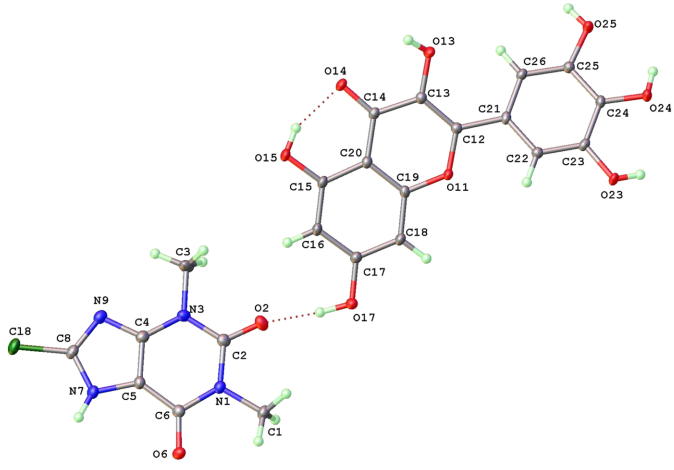


Figure 11
The labelling scheme for the 8-Cl-theophilline–myricetin 1:1 cocrystal (**2c**), similarly adopted by the isostructural 8-Br analogue **3c**.

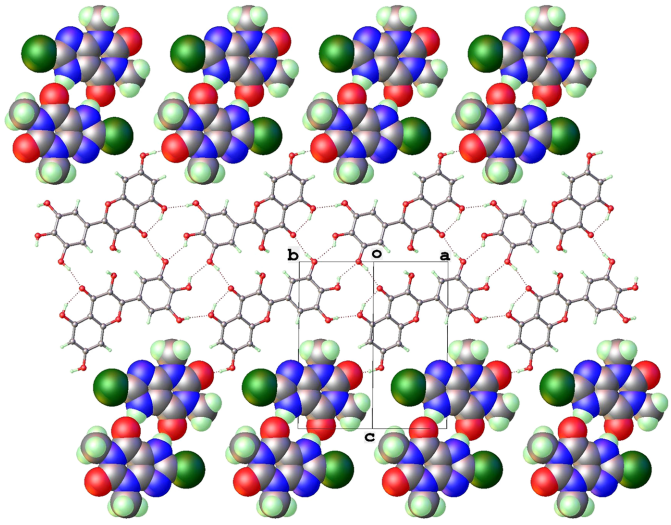


Figure 12
Packing diagram for **2c**, viewed along [110], showing the segregation of myricetin double-stranded chains and 8-Cl-Tph dimers (space-filled).

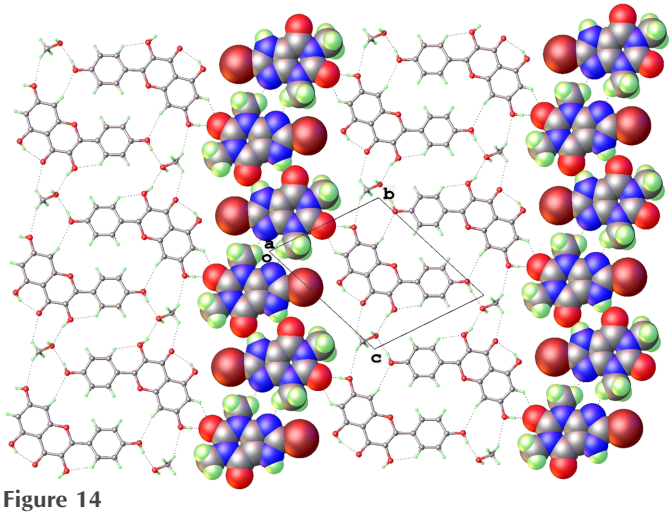


Figure 14
Packing diagram for the 8-Br-theophylline–kaempferol–MeOH 1:1:1 cocrystal (**3a**′), viewed along [100], showing alternating strands of 8-Br-Tph dimers and solvated kaempferol molecules.

Tph and 8-Br-Tph cases. The phase details are given in Table 3 and crystal data summaries in Table 1. Attempts to use microwave crystallization to afford these phases in a convenient manner from methanol also yielded a further solvated cocrystal phase for 8-Br-Tph and kaempferol (**3a**′). The solvated cocrystal formation can, however, be suppressed using 1-butanol (140 °C) in the microwave-assisted cocrystallization process from which pure **3a** can be isolated.

Phases **2a** and **3a** are isostructural, and the asymmetric unit is a simple molecular pair, as shown in Fig. 9. In all these cocrystals, the halotheophyllines form molecular dimers through N7—H···O6 hydrogen bonds, as described above for **1c**, and this can be seen in the packing diagram for **2a** shown in Fig. 10. The remaining keto O2 atom then typically serves as an acceptor of a strong hydrogen bond from a flavonoid OH group, whilst N9 is involved either in a weak hydrogen bond, as seen in Fig. 9 (N9—H···O24 = 2.87 Å), or not at all. In **2a** and **3a**, layer structures are found in which the 8-*X*-Tph dimers are surrounded by six neighbouring kaempferol molecules (see Fig. 10).

The cocrystals of 8-*X*-Tph with myricetin, **2c** (*X* = Cl) and **3c** (*X* = Br), are also an isostructural pair with simple halogen interchange. The asymmetric unit and atom-labelling scheme for **2c** are shown in Fig. 11. These phases also form layer structures, but in **2c** and **3c**, there is segregation of the 8-*X*-Tph dimers and myricetin molecules within the layers, as shown in the packing diagram in Fig. 12.

Finally, a further methanolated cocrystal phase **3a**′ was isolated for the 8-Br-Tph–Kmp system, upon microwave-assisted synthesis in MeOH, that was not found previously by LAG or solvothermal crystallization. The asymmetric unit and atom-labelling scheme is shown in Fig. 13. Unlike phase **3a**, the alkaloid dimers are not isolated but also segregate from solvated kaempferol in one-dimensional strands (Fig. 14). This phase type may be favoured through the formation of an attractive Br···O interaction of 3.024 (2) Å.

3.4. Structures of coformers: specific volume comparison for cocrystals

The crystal structures at 100 K of theophylline (Fucke *et al.*, 2012) and the 8-chloro and 8-bromo analogues 8-*X*-Tph were reported recently by us (Ye *et al.*, 2025). Several crystal structures of the various flavonoid coformers were reported previously by others, many of them as hydrated or solvated phases; where possible, we have redetermined these at 100 K. Interestingly, no structural entry existed for kaempferol. We

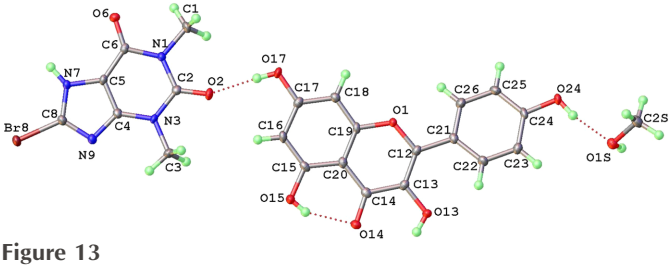


Figure 13
The asymmetric unit and labelling scheme for the 8-Br-theophylline–kaempferol–MeOH 1:1:1 cocrystal (**3a**′).

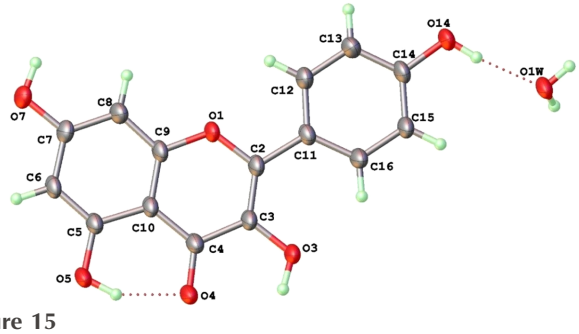


Figure 15
The asymmetric unit and atom-labelling scheme for kaempferol hydrate (**a**·H_2O) (50% probability displacement ellipsoids).

Table 4
Specific volumes for theophylline–flavonoid coformer/cocrystal phases.

(*a*) specific volumes for theophylline–flavonoid coformer phases

Phase	Formula	Unit cell volume (Å^3), *Z*	Molecular volume (anhydrous)[ii] (Å^3)	Reference/CSD refcode
1, theophylline (Tph)	$C_7H_8N_4O_2$	390.9, 2	195.5 (195.5)	Ye *et al.* (2025)
2, 8-Cl-theophylline (8-Cl-Tph)	$C_7H_7ClN_4O_2$	418.0, 2	209.0 (209.0)	Ye *et al.* (2025)
3, 8-Br-theophylline (8-Br-Tph)	$C_7H_7BrN_4O_2$	860.9, 4	215.2 (215.2)	Ye *et al.* (2025)
a, kaempferol, H_2O (Kmp)	$C_{15}H_{10}O_6.H_2O$	2519.7, 8	315.0 (295.0)	This work
b, quercetin, H_2O (Que)	$C_{15}H_{10}O_7.H_2O$	1273.8, 4	318.4 (298.4)	AKIJEK
c, myricetin, H_2O (Myr)[i]	$C_{15}H_{10}O_8.H_2O$	1305.5, 4	323.2 (303.2)	NIKLAX
d, baicalein (Bai)	$C_{15}H_{10}O_5$	1170.7, 4	292.7 (292.7)	RAMGOB01
e, hesperetin (Hes)	$C_{16}H_{14}O_6$	1359.5, 4	339.9 (339.9)	YEHROS
f, dihydromyricetin, $2H_2O$ (Dhm)	$C_{15}H_{10}O_8.2H_2O$	2928.6, 8	366.1 (326.1)	SIMVOA02

(*b*) specific volumes for theophylline–flavonoid cocrystal phases

Phase	Unit cell volume (Å^3), *Z*	Formula volume (anhydrous)[b] (Å^3)	Coformer volume (anhydrous) (ΔV Å^3)	Reference/CSD refcode
1a, Tph–Kmp (1/1)	1978.8, 4	494.7 (494.7)	490.5, −4.2	This work
1b, Tph–Que (1/1), $0.5H_2O$	4083.2, 8	510.4 (500.4)	493.9, −5.1	JATPIH
1c, Tph–Myr (1/1), $0.5H_2O$	4188.5, 8	523.6 (513.6)	498.7[a], −14.9	This work
1d, Tph–Bai (2/1), $3H_2O$	1496.2, 2	748.1 (688.1)	683.6, −4.7	KAMQIB
1e, Tph–Hes (2/1) MeOH, $2H_2O$	1630.2, 2	815.1 (735.1)	730.8, −4.3	This work
1f, Tph–Dhm (1/1), MeCN	2402.0, 4	600.5 (540.5)	521.6, −18.9	NEXSIW
2a, 8-Cl-Tph–Kmp	2081.8, 4	520.4 (520.4)	504.0, −16.4	This work
2c, 8-Cl-Tph–Myr	1066.2, 2	533.1 (533.1)	512.2, −20.9	This work
3a, 8-Br-Tph–Kmp	2101.0, 4	525.3 (525.3)	510.2, −15.1	This work
3a′, 8-Br-Tph–Kmp, MeOH	1142.3, 2	571.2 (531.2)	510.2, −21.2	This work
3c, 8-Br-Tph–Myr	1079.3, 2	539.7 (539.7)	518.4, −21.3	This work

Notes: (i) all unit-cell data at 100 K, except for Myr·H_2O (140 K), the coformer volume is reduced by 1% to approximate a 100 K structure; (ii) molecular volumes subtracted: H_2O = 20 Å^3, MeOH 40 Å^3 and MeCN 60 Å^3. References for earlier CSD structures: AKIJEK (Domagała *et al.*, 2011), NIKLAX (Ren *et al.*, 2019), RAMGOB01 (Rossi *et al.*, 2001), YEHROS (Maeda *et al.*, 1994), SIMVOA02 (Hu *et al.*, 2020), JATPIH (Wang *et al.*, 2022), KAMQIB (Zhu *et al.*, 2017) and NEXSIW (Sun *et al.*, 2023). Except for NIKLAX, these were all redetermined by us at 100 K.

have grown this as a monohydrate (**a·H_2O**) through the ambient evaporation of an acetonitrile solution and report its crystal structure here (Fig. 15). The structural details are given in Table 1.

Table 4(*a*) gives a summary of the alkaloid and flavonoid coformer structures (at or close to 100 K), along with the corresponding molecular volume (V_{mol}) for the alkaloid or flavonoid of interest. The variable volume of water or solvent molecules make comparison of specific molecular volumes for cocrystals and coformers slightly more involved. However, a useful comparison of packing efficiency may still be attempted and is provided for the cocrystals in Table 4(*b*). In general, it was anticipated that the specific volume for a cocrystal would be less than the sum of the individual coformers; however, surprisingly, this was not found to be the case for these cocrystals. If molecular volumes of 20, 40 and 60 Å^3 are assigned to water, methanol and acetonitrile, respectively, then the combined volumes of the alkaloid and flavonoid molecules in the cocrystals are actually *equal to or greater than* those from the parent coformer crystals themselves.

Table 5
Hydrogen bonds (HBs) for selected crystals and cocrystals.

Phase	No. of donors, HBs	Strongest HBs (Å)	Comment	Reference
1, Tph	1, 1	NH···N 2.820 (4)	chain	Ye *et al.* (2025)
2, 8-Cl-Tph	1, 1	NH···O 2.723 (2)	dimer	Ye *et al.* (2025)
3, 8-Br-Tph	1, 1	NH···O 2.760 (2)	dimer	Ye *et al.* (2025)
a·H_2O, Kmp H_2O	6, 6	OH···O 2.742 (2)	4 HBs with H_2O	This work
1a, Tph–Kmp	5, 5	OH···O 2.680 (2) OH···N9 2.794 (2)	3 HBs Kmp-to-Tph 1 HB Tph-to-Kmp	This work
2a. 8-Cl-Tph–Kmp	5, 5	NH···O6 2.667 (2) OH···O2 2.774 (2)	2 HBs Kmp-to-8-Cl-Tph OH···N9 2.866 Å (weak)	This work
3a, 8-Br-Tph–Kmp	5, 5	NH···O6 2.671 (2) OH···O2 2.769 (2)	2 HBs Kmp-to-8-Br-Tph OH···N9 2.881 Å (weak)	This work
3a′, 8-Br-Tph–Kmp MeOH	6, 6	NH···O6 2.698 (2) OH···O2 2.708 (2)	1 HB Kmp-to-8-Br-Tph No use of N9 acceptor	This work
1c, Tph–Myr $0.5H_2O$	8, 7	OH···N9 2.744 (3)	H_2O single donor	This work
2c, 8-Cl-Tph–Myr	7, 7	OH···O2 2.689 (2)	2 HBs Kmp-to-8X-Tph No use of N9 acceptor	This work
3c, 8-Br-Tph–Myr	7, 7	OH···O2 2.652 (2)	2 HBs Kmp-to-8X-Tph No use of N9 acceptor	This work

Table 6
Microwave-assisted formation for selected cocrystals*.

Phase	Alkaloid M_r (mg) Flavonoid M_r (mg)	Cocrystal M_r Yield (mg)	% yield
1a, Tph–Kmp (1/1)	180.2, 180 286.2, 300	466.4 413	88.6
1b, Tph–Que (1/1), 0.5H_2O	180.2, 180 302.2, 310	491.4 451	91.8
1c, Tph–Myr (1/1), 0.5H_2O	180.2, 180 318.2, 325	507.4 438	86.3
1d, Tph–Bai (2/1), 3H_2O	180.2, 270 (1.5 mmol) 270.2, 210 (0.75 mmol)	648.6 557	85.9
2a, 8-Cl-Tph–Kmp (1/1)	214.6, 215 286.2, 300	500.8 433	86.5
2c, 8-Cl-Tph–Myr (1/1)	214.6, 215 318.2, 325	532.8 490	92.0
3a, 8-Br-Tph–Kmp (1/1)	259.1, 130 286.2, 150	545.3 220	80.7
3c, 8-Br-Tph–Myr (1/1)	259.1, 130 318.2, 160	577.3 255.5	88.5

Note: (*) microwave-assisted cocrystal formation in Biotage Initiator+ using sealed 2–5 ml reaction vials; 1 mmol scale in 2 ml 1-butanol, except for **1d**, a 2:1 cocrystal on a 0.75 mmol scale in 2 ml 1-butanol, and **3a** and **3c** on a 0.5 mmol scale in 1 ml 1-butanol.

3.5. Rationalization of cocrystal formation

Historically, recrystallization of molecular mixtures was used as a tool for purification, relying on the lower solubility of one molecule than the others to initiate its precipitation. The advent of crystal engineering has developed ideas of molecular recognition in organic crystal structures that can also be adapted to assist the intentional cocrystallization of molecules, with driving forces for this based on intermolecular recognition, supramolecular synthon formation, electrostatic interactions or better hydrogen bonding or packing efficiency. Our idea in this project was to survey the tendency of cocrystal formation between two extensive and fundamental classes of natural product, *i.e.* alkaloids, which typically have a surplus of hydrogen-bond-acceptor functionalities (ring N or exocyclic keto O atoms), and flavonoids, which have a surplus of OH-donor functionalities to keto O-atom acceptors.

In examining the cocrystal formation for the antimalarial compound 11-azaartemisinin, we found that it could form cocrystals with around 50% of organic carboxylic acids studied (Nisar *et al.*, 2018). This appeared to be supported by a favourable supramolecular heterosynthon between the acid and the amide. In the cases that were successful, the specific volumes of the cocrystal phases were typically less than that for the component co-formers in their own crystal. However, when salicylic acids were studied exclusively, the cocrystal formation probability was over 95%, due to strengthening of this heterosynthon, and a number of these cocrystals were less efficiently packed than their parent phases (Li *et al.*, 2022; Li, 2024). Thus, a sufficient improvement in hydrogen bonding can overcome packing deficiency.

In a study of 350 organic cocrystals by DFT calculations, Taylor & Day (2018) found that the vast majority (>95%) were thermodynamically preferred and by an average of 8 kcal mol^{-1}. However, several factors appear to be operating in tandem to lead to this stabilization. By combining two key descriptors: (i) packing efficiency and (ii) hydrogen-bond strength; these gave good correlations with the stabilization of the cocrystal phases. In particular, those phases with lower packing efficiency typically had optimization of hydrogen bonds in the cocrystal phases to offset this.

Our surprising findings that the theophylline–flavonoid cocrystals in these studies typically have *less efficient* packing than the parent coformer phases led us to next examine hydrogen bonding and whether this was optimized in the cocrystal phases that we have isolated (Table 5). We present an analysis of Tph and 8-*X*-Tph with kaempferol and myricetin, since these phases are less solvated than some of the other systems and the structural effect of the 8-halo substituent can also be examined. In general, alkaloid–flavonoid cocrystallization does seem to be favoured due to net hydrogen-bond formation from the H-atom-donor-rich flavonoid to the H-atom-acceptor-rich alkaloid.

In principle, theophylline has three good acceptors, *i.e.* N9, O2 and O6. In cocrystal phase **1a** (Tph–Kmp), there are three hydrogen bonds from the flavonoid to these three acceptors, including a short O24—H···O2 interaction (2.68 Å), as well as a 'reverse' hydrogen bond from the theophylline N7—H group to the flavonoid. In the other Tph cocrystal phases **1c** (Tph–Myr·0.5H_2O) and **1e** (Tph–Hes 2:1 solvate), once more a surplus of two hydrogen bonds are donated from the flavonoid to the alkaloid and the N9 atom is involved as acceptor. The previously reported cocrystals of theophylline with flavonoids show generally similar findings (Wang *et al.*, 2022; Zhu *et al.*, 2017; Sun *et al.*, 2023).

In examining the structures of theophylline molecular crystals themselves, we found that the N9 position is deactivated as an acceptor upon substitution of the 8-*X* groups Cl and Br (Ye *et al.* 2025). This deactivation may lie at the heart of why fewer cocrystal phases are formed for the 8-halotheophyllines. This is supported by the observation that in those cocrystal types that are found for 8-*X*-Tph and flavonoids, the N9 group is either in just a weak hydrogen bond (OH···N9 = 2.87 Å for **2a** and OH···N9 = 2.88 Å for **3a**) or is not involved at all (phases **2c**, **3c** and **3a**′).

It may be noted that among the flavonoids screened for cocrystallization, kaempferol (Kmp) and myricetin (Myr) are the most active coformers, with 12 cocrystals each from 15 common alkaloids. This may be explained, in part, by the fact that the compounds themselves are difficult to crystallize in the anhydrous form, so that pure coformer crystals are uncompetitive solid phases. Both kaempferol (reported here) and myricetin (Ren *et al.*, 2019) form monohydrates, but so does quercetin (Domagała *et al.*, 2011). Analysis of the respective molecular volumes (Table 4) indicates that quercetin monohydrate is more efficiently packed. Hence, quercetin has a slightly less pronounced tendency to cocrystal formation, with eight cocrystals, a number of cocrystal phases that is similar to baicalein which has a phenyl group.

The 8-halo-Tph phases **2a**/**3a** and **2c**/**3c** are isostructural pairs; however, efforts to isolate the 8-Cl-Tph analogue of the methanol-solvated phase 8-Br-Tph–Kmp·MeOH (**3a**′) were unsuccessful. Examination of the **3a**′ structure shows that a favourable Br···O15 contact of 3.024 (2) Å for the halogen-bond interaction exists in this phase. This would be consider-

ably weaker if Cl is substituted for Br, offering an explanation of why the analogous isostructural methanol solvate **2a′** is not formed.

In the cases presented here, the thermodynamic stability of the majority of the cocrystal phases relative to their components was established through their formation *via* direct LAG of the coformers in the appropriate stoichiometric ratio (1:1 or 2:1). PXRD indicated that almost all cocrystal phases for theophylline could be formed, with the exception of dihydromyricetin, which is an acetonitrile solvate, and hesperetin, which forms a highly solvated cocrystal phase, which we believe is an unstable kinetic product.

Where LAG is successful, the use of microwave-assisted cocrystallization (Ahuja *et al.*, 2020) appears to be the most convenient approach to form stable phases in good yield and purity, as given for eight phases in Table 6. Yields of 80–90% were obtained for a typical 1 mmol scale in 2 ml 1-butanol, applying 20 min heating at 140 °C, followed by a 20 min cooling cycle, with the solids centrifuged, filtered and dried. When LAG is applied directly, some slight contamination of the cocrystals with starting phases is hard to eliminate entirely.

4. Conclusions

Screening for cocrystal formation of 13 common flavonoids with a family of alkaloids that are electronically perturbed, shows that theophylline (Tph) has a markedly stronger tendency for cocrystallization compared to its 8-halo analogues. A total of six flavonoid cocrystals were isolated for Tph, three of which were reported previously, whilst only two flavonoids work for 8-Cl-Tph or 8-Br-Tph. Principally, these are the unsolvated 1:1 phases with kaempferol (**2a**/**3a**) and myricetin (**2c**/**3c**), which are isostructural for the 8-Cl or 8-Br substituents. *None* of the successful cocrystallization outcomes of flavonoids with any of these theophylline alkaloids appear to be driven by improved packing efficiency. The marked difference in cocrystal formation tendency for Tph compared to its halo derivatives may be influenced by the deactivation of the alkaloid N9 atom as a hydrogen-bond acceptor in the halogenated molecules. In general, the cocrystal phases reported here are thermodynamically competitive and can be prepared by co-precipitation from cooling hot solutions (solvothermal), by LAG of co-formers or, most conveniently, through microwave-assisted cocrystallization from 1-butanol.

Funding information

Funding for this research was provided by: the Research Grants Council of Hong Kong through equipment grant C6022-20E. We also gratefully acknowledge the financial support of the Hong Kong Branch of the Southern Marine Science and Engineering Guangdong Laboratory (Guangzhou) (grant No. SMSEGL-20SC01-D) and the Hong Kong Branch of Chinese National Engineering Research Center for Tissue Restoration and Reconstruction, funded by the Innovation and Technology Commission (ITC-CNERC14SC01).

References

Ahuja, D., Ramisetty, K. A., Sumanth, P. K., Crowley, C. M., Lusi, M. & Rasmuson, C. (2020). *CrystEngComm* **22**, 1381–1394.
Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* A**71**, 59–75.
Bruker (2016). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chadha, K., Karan, M., Bhalla, Y., Chadha, R., Kullar, S., Mandal, S. & Vasisht, K. (2017). *Cryst. Growth Des.* **17**, 2386–2405.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Domagała, S., Munshi, P., Ahmed, M., Guillot, B. & Jelsch, C. (2011). *Acta Cryst.* B**67**, 63–78.
Dyulgerov, V. M., Dimowa, L. T., Kossev, K., Nikolova, R. P. & Shivachev, B. L. (2015). *Bulg. Chem. Commun.* **47**, 311–316.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Fucke, K., McIntyre, G. J., Wilkinson, C., Henry, M., Howard, J. A. K. & Steed, J. W. (2012). *Cryst. Growth Des.* **12**, 1395–1401.
Good, D. J. & Rodríguez-Hornedo, N. (2009). *Cryst. Growth Des.* **9**, 2252–2264.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Hu, H., Luo, F., Wang, M., Fu, Z. & Shu, X. (2020). *ACS Omega* **5**, 13955–13962.
Karimi-Jafari, M., Padrela, L., Walker, G. M. & Croker, D. M. (2018). *Cryst. Growth Des.* **18**, 6370–6387.
Li, K. (2024). PhD thesis, Hong Kong University of Science and Technology.
Li, K., Roy, M., Nisar, M., Wong, L. W.-Y., Sung, H. H.-Y., Haynes, R. K. & Williams, I. D. (2022). *Crystals* **12**, 1368.
Maeda, S., Masuda, H. & Tokoroyama, T. (1994). *Chem. Pharm. Bull.* **42**, 2500–2505.
Nisar, M., Sung, H. H.-Y., Puschmann, H., Lakerveld, R., Haynes, R. K. & Williams, I. D. (2018). *CrystEngComm* **20**, 1205–1219.
Pryzynska, K. (2022). *Nutrients* **14**, 2387.
Putra, O. D., Yoshida, T., Umeda, D., Higashi, K., Uekusa, H. & Yonemochi, E. (2016). *Cryst. Growth Des.* **16**, 5223–5229.
Ren, S., Liu, M., Hong, C., Li, G., Sun, J., Wang, J., Zhang, L. & Xie, Y. (2019). *Acta Pharm. Sin. B* **9**, 59–73.
Rigaku OD (2022). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Rigaku OD (2023). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Rossi, M., Meyer, R., Constantinou, P., Caruso, F., Castelbuono, D., O'Brien, M. & Narasimhan, V. (2001). *J. Nat. Prod.* **64**, 26–31.
Roy, M., Li, K., Nisar, M., Wong, L. W.-Y., Sung, H. H.-Y., Haynes, R. K. & Williams, I. D. (2021). *Acta Cryst.* C**77**, 262–270.
Sakhiya, D. C. & Borkhataria, C. H. (2024). *Heliyon* **10**, e29057.
Sanphui, A. & Nangia, A. (2014). *J. Chem. Sci.* **126**, 1249–1264.
Sharma, R. G., Vankar, S. D. & Sharma, M. G. (2025). *Mol. Divers.* **30**, 3343–3365.
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. (2015). *Acta Cryst.* C**71**, 9–18.
Sun, J., Wang, Y., Tang, W. & Gong, J. (2023). *IUCrJ* **10**, 164–176.
Taylor, C. R. & Day, G. M. (2018). *Cryst. Growth Des.* **18**, 892–904.
Trask, A. V. & Jones, W. (2005). *Top. Curr. Chem.*, **254**, 41-70.
Trask, A. V., Motherwell, W. D. S. & Jones, W. (2005). *Cryst. Growth Des.* **5**, 1013–1021.
Trask, A. V., Motherwell, W. D. S. & Jones, W. (2006). *Int. J. Pharm.* **320**, 114–123.
Wang, L., Li, S., Xu, X., Xu, X., Wang, Q., Li, D. & Zhang, H. (2022). *J. Drug. Deliv. Sci. Technol.* **70**, 103228.

Xia, Y., Wei, Y., Chen, H., Qian, S., Zhang, J. & Gao, Y. (2021). *IUCrJ* **8**, 195–207.
Ye, W. (2024). PhD thesis, Hong Kong University of Science and Technology.
Ye, W., Zhang, C., Sung, H. H.-Y., Wong, L. W.-Y., Sheong, F. E. K. & Williams, I. D. (2025). *Crystals* **15**, 340.
Zhu, B., Zhang, Q., Wang, J.-R. & Mei, X. (2017). *Cryst. Growth Des.* **17**, 1893–1901.

supporting information

Acta Cryst. (2026). C**82** [https://doi.org/10.1107/S2053229626006637]

Effect of molecular perturbation on cocrystal formation: theophylline and its 8-halo analogues with flavonoids

Weijian Ye, Herman H-Y. Sung and Ian D. Williams

Computing details

3,7-Dihydro-1,3-dimethyl-1*H*-purine-2,6-dione–3,5,7-trihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one (1a-ywj135Cu100k_auto)

Crystal data

$C_7H_8N_4O_2 \cdot C_{15}H_{10}O_6$
$M_r = 466.41$
Monoclinic, $P2_1/n$
$a = 6.51941\ (9)$ Å
$b = 7.77386\ (10)$ Å
$c = 39.0816\ (5)$ Å
$\beta = 92.4920\ (12)°$
$V = 1978.82\ (5)$ Å^3
$Z = 4$
$F(000) = 968$
$D_x = 1.566$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 7930 reflections
$\theta = 5.8–77.2°$
$\mu = 1.03$ mm^{-1}
$T = 100$ K
Block, yellow
0.2 × 0.2 × 0.2 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3577 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)
$T_{min} = 0.807$, $T_{max} = 1.000$
13045 measured reflections
4130 independent reflections
3800 reflections with $I \geq 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 77.2°$, $\theta_{min} = 5.8°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 8$
$l = -49 \rightarrow 49$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.095$
$S = 1.06$
4130 reflections
329 parameters
0 restraints
22 constraints
Primary atom site location: iterative
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.9648P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.44398 (17)	0.62449 (15)	0.38117 (3)	0.0185 (2)
C1	0.6394 (2)	0.54261 (19)	0.39194 (4)	0.0232 (3)
H1a	0.6380 (7)	0.4221 (4)	0.3846 (3)	0.0348 (4)*
H1b	0.7528 (3)	0.6030 (9)	0.3815 (2)	0.0348 (4)*
H1c	0.6577 (8)	0.5481 (13)	0.41694 (4)	0.0348 (4)*
O2	0.39234 (15)	0.70755 (14)	0.43662 (2)	0.0244 (2)
C2	0.3324 (2)	0.70499 (17)	0.40655 (3)	0.0186 (3)
N3	0.14915 (18)	0.78043 (15)	0.39649 (3)	0.0186 (2)
C3	0.0255 (2)	0.8574 (2)	0.42315 (3)	0.0236 (3)
H3a	−0.0926 (10)	0.9174 (13)	0.41236 (4)	0.0354 (4)*
H3b	−0.0230 (15)	0.7666 (3)	0.43821 (18)	0.0354 (4)*
H3c	0.1098 (6)	0.9394 (11)	0.43663 (19)	0.0354 (4)*
C4	0.0872 (2)	0.78206 (17)	0.36241 (3)	0.0165 (3)
C5	0.2016 (2)	0.70349 (17)	0.33814 (3)	0.0167 (3)
O6	0.49961 (15)	0.54368 (13)	0.32589 (2)	0.0216 (2)
C6	0.3896 (2)	0.61805 (17)	0.34601 (3)	0.0172 (3)
N7	0.09529 (17)	0.73265 (15)	0.30734 (3)	0.0169 (2)
C8	−0.0717 (2)	0.82358 (17)	0.31459 (3)	0.0177 (3)
H8	−0.1714 (2)	0.86028 (17)	0.29769 (3)	0.0212 (3)*
N9	−0.08420 (17)	0.85766 (15)	0.34800 (3)	0.0181 (2)
O11	0.87973 (15)	0.72884 (13)	0.63486 (2)	0.0201 (2)
C12	0.7062 (2)	0.79850 (17)	0.61910 (3)	0.0175 (3)
O13	0.39840 (15)	0.95705 (14)	0.62248 (2)	0.0230 (2)
C13	0.5682 (2)	0.88574 (17)	0.63790 (3)	0.0175 (3)
O14	0.47629 (14)	0.99131 (13)	0.69160 (2)	0.0203 (2)
C14	0.6018 (2)	0.90753 (17)	0.67453 (3)	0.0166 (3)
O15	0.71005 (15)	0.92751 (13)	0.74591 (2)	0.0201 (2)
C15	0.8398 (2)	0.84498 (16)	0.72499 (3)	0.0166 (3)
C16	1.0220 (2)	0.77873 (17)	0.73839 (3)	0.0176 (3)
H16	1.0569 (2)	0.78821 (17)	0.76217 (3)	0.0211 (3)*
O17	1.33559 (15)	0.63804 (13)	0.73051 (2)	0.0225 (2)
C17	1.1552 (2)	0.69688 (17)	0.71625 (3)	0.0175 (3)
C18	1.1055 (2)	0.67818 (17)	0.68159 (3)	0.0192 (3)
H18	1.1951 (2)	0.61998 (17)	0.66694 (3)	0.0231 (3)*
C19	0.9222 (2)	0.74654 (17)	0.66899 (3)	0.0172 (3)
C20	0.7851 (2)	0.83206 (16)	0.68964 (3)	0.0161 (3)
C21	0.7024 (2)	0.76391 (17)	0.58217 (3)	0.0184 (3)
C26	0.5346 (2)	0.80479 (19)	0.55985 (3)	0.0221 (3)
H26	0.4174 (2)	0.85906 (19)	0.56862 (3)	0.0265 (3)*
C25	0.5379 (2)	0.7670 (2)	0.52524 (4)	0.0242 (3)
H25	0.4227 (2)	0.7944 (2)	0.51051 (4)	0.0291 (4)*
O24	0.72138 (17)	0.64681 (16)	0.47835 (3)	0.0298 (3)
C24	0.7096 (2)	0.68900 (19)	0.51197 (3)	0.0225 (3)
C23	0.8796 (2)	0.6510 (2)	0.53356 (4)	0.0253 (3)
H23	0.9983 (2)	0.6004 (2)	0.52451 (4)	0.0304 (4)*

C22	0.8748 (2)	0.68710 (19)	0.56807 (4)	0.0227 (3)
H22	0.9906 (2)	0.65945 (19)	0.58264 (4)	0.0273 (4)*
H7	0.133 (3)	0.691 (3)	0.2865 (5)	0.039 (5)*
H17	1.406 (3)	0.582 (3)	0.7143 (6)	0.050 (6)*
H24	0.601 (4)	0.672 (3)	0.4662 (6)	0.054 (7)*
H15	0.602 (3)	0.962 (3)	0.7317 (6)	0.047 (6)*
H13	0.317 (4)	1.009 (3)	0.6372 (6)	0.065 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0175 (5)	0.0224 (6)	0.0154 (5)	0.0012 (4)	−0.0023 (4)	−0.0002 (4)
C1	0.0186 (6)	0.0296 (7)	0.0208 (6)	0.0026 (6)	−0.0040 (5)	0.0006 (5)
O2	0.0246 (5)	0.0339 (6)	0.0143 (5)	0.0013 (4)	−0.0030 (4)	−0.0009 (4)
C2	0.0198 (6)	0.0199 (6)	0.0159 (6)	−0.0023 (5)	−0.0006 (5)	0.0003 (5)
N3	0.0201 (6)	0.0234 (6)	0.0123 (5)	0.0010 (5)	0.0009 (4)	−0.0011 (4)
C3	0.0254 (7)	0.0309 (8)	0.0147 (6)	0.0030 (6)	0.0035 (5)	−0.0034 (5)
C4	0.0176 (6)	0.0176 (6)	0.0142 (6)	−0.0024 (5)	0.0006 (5)	0.0007 (5)
C5	0.0188 (6)	0.0188 (6)	0.0123 (6)	−0.0017 (5)	−0.0001 (5)	−0.0003 (5)
O6	0.0203 (5)	0.0269 (5)	0.0176 (5)	0.0043 (4)	0.0001 (4)	−0.0034 (4)
C6	0.0177 (6)	0.0178 (6)	0.0159 (6)	−0.0024 (5)	−0.0004 (5)	−0.0002 (5)
N7	0.0178 (5)	0.0201 (5)	0.0127 (5)	−0.0005 (4)	−0.0002 (4)	−0.0006 (4)
C8	0.0177 (6)	0.0202 (6)	0.0150 (6)	−0.0012 (5)	−0.0002 (5)	0.0006 (5)
N9	0.0175 (5)	0.0214 (6)	0.0152 (5)	−0.0003 (4)	0.0004 (4)	0.0007 (4)
O11	0.0198 (5)	0.0269 (5)	0.0135 (4)	0.0053 (4)	−0.0024 (3)	−0.0028 (4)
C12	0.0177 (6)	0.0197 (6)	0.0146 (6)	−0.0005 (5)	−0.0025 (5)	0.0007 (5)
O13	0.0211 (5)	0.0329 (6)	0.0148 (4)	0.0095 (4)	−0.0014 (4)	−0.0010 (4)
C13	0.0169 (6)	0.0188 (6)	0.0167 (6)	0.0005 (5)	−0.0014 (5)	0.0016 (5)
O14	0.0188 (5)	0.0254 (5)	0.0167 (4)	0.0043 (4)	0.0016 (3)	0.0003 (4)
C14	0.0171 (6)	0.0163 (6)	0.0165 (6)	−0.0020 (5)	0.0013 (5)	0.0011 (5)
O15	0.0210 (5)	0.0258 (5)	0.0136 (4)	0.0049 (4)	0.0017 (4)	−0.0002 (4)
C15	0.0186 (6)	0.0153 (6)	0.0160 (6)	−0.0012 (5)	0.0017 (5)	0.0003 (5)
C16	0.0213 (6)	0.0186 (6)	0.0126 (6)	−0.0008 (5)	−0.0011 (5)	0.0008 (5)
O17	0.0221 (5)	0.0281 (5)	0.0170 (5)	0.0078 (4)	−0.0036 (4)	−0.0009 (4)
C17	0.0173 (6)	0.0165 (6)	0.0185 (6)	0.0004 (5)	−0.0023 (5)	0.0019 (5)
C18	0.0204 (6)	0.0201 (6)	0.0173 (6)	0.0030 (5)	0.0008 (5)	−0.0017 (5)
C19	0.0198 (6)	0.0180 (6)	0.0136 (6)	−0.0007 (5)	−0.0012 (5)	−0.0007 (5)
C20	0.0171 (6)	0.0159 (6)	0.0153 (6)	−0.0015 (5)	0.0002 (5)	0.0002 (5)
C21	0.0207 (6)	0.0204 (6)	0.0140 (6)	0.0003 (5)	−0.0010 (5)	−0.0010 (5)
C26	0.0187 (6)	0.0301 (7)	0.0173 (6)	0.0030 (5)	−0.0003 (5)	−0.0001 (5)
C25	0.0199 (7)	0.0350 (8)	0.0174 (7)	0.0022 (6)	−0.0032 (5)	0.0009 (6)
O24	0.0252 (5)	0.0507 (7)	0.0133 (5)	0.0066 (5)	−0.0026 (4)	−0.0056 (4)
C24	0.0239 (7)	0.0303 (7)	0.0133 (6)	−0.0004 (6)	−0.0004 (5)	−0.0022 (5)
C23	0.0229 (7)	0.0348 (8)	0.0181 (7)	0.0057 (6)	−0.0005 (5)	−0.0044 (6)
C22	0.0212 (7)	0.0282 (7)	0.0184 (7)	0.0046 (6)	−0.0036 (5)	−0.0019 (5)

Geometric parameters (Å, º)

N1—C1	1.4691 (16)	C13—C14	1.4490 (17)
N1—C2	1.4025 (17)	O14—C14	1.2594 (16)
N1—C6	1.4048 (16)	C14—C20	1.4348 (18)
C1—H1a	0.9800	O15—C15	1.3624 (16)
C1—H1b	0.9800	O15—H15	0.92 (2)
C1—H1c	0.9800	C15—C16	1.3771 (18)
O2—C2	1.2223 (16)	C15—C20	1.4150 (17)
C2—N3	1.3731 (17)	C16—H16	0.9500
N3—C3	1.4719 (17)	C16—C17	1.4047 (18)
N3—C4	1.3750 (16)	O17—C17	1.3588 (16)
C3—H3a	0.9800	O17—H17	0.91 (2)
C3—H3b	0.9800	C17—C18	1.3867 (18)
C3—H3c	0.9800	C18—H18	0.9500
C4—C5	1.3748 (18)	C18—C19	1.3790 (19)
C4—N9	1.3627 (17)	C19—C20	1.3983 (18)
C5—C6	1.4160 (18)	C21—C26	1.4055 (18)
C5—N7	1.3812 (16)	C21—C22	1.4063 (19)
O6—C6	1.2314 (16)	C26—H26	0.9500
N7—C8	1.3385 (17)	C26—C25	1.3854 (19)
N7—H7	0.92 (2)	C25—H25	0.9500
C8—H8	0.9500	C25—C24	1.393 (2)
C8—N9	1.3383 (17)	O24—C24	1.3596 (16)
O11—C12	1.3757 (15)	O24—H24	0.92 (2)
O11—C19	1.3575 (15)	C24—C23	1.3953 (19)
C12—C13	1.3660 (19)	C23—H23	0.9500
C12—C21	1.4673 (17)	C23—C22	1.3794 (19)
O13—C13	1.3555 (15)	C22—H22	0.9500
O13—H13	0.89 (3)		
C2—N1—C1	117.41 (11)	C14—C13—O13	118.48 (11)
C6—N1—C1	116.34 (11)	O14—C14—C13	120.66 (12)
C6—N1—C2	126.22 (11)	C20—C14—C13	116.51 (11)
H1a—C1—N1	109.5	C20—C14—O14	122.82 (12)
H1b—C1—N1	109.5	H15—O15—C15	104.8 (14)
H1b—C1—H1a	109.5	C16—C15—O15	119.70 (11)
H1c—C1—N1	109.5	C20—C15—O15	118.86 (11)
H1c—C1—H1a	109.5	C20—C15—C16	121.43 (12)
H1c—C1—H1b	109.5	H16—C16—C15	120.62 (8)
O2—C2—N1	122.23 (12)	C17—C16—C15	118.76 (12)
N3—C2—N1	117.36 (11)	C17—C16—H16	120.62 (7)
N3—C2—O2	120.40 (12)	H17—O17—C17	109.2 (14)
C3—N3—C2	117.87 (11)	O17—C17—C16	116.55 (12)
C4—N3—C2	119.77 (11)	C18—C17—C16	121.65 (12)
C4—N3—C3	122.36 (11)	C18—C17—O17	121.79 (12)
H3a—C3—N3	109.5	H18—C18—C17	120.97 (8)
H3b—C3—N3	109.5	C19—C18—C17	118.05 (12)

H3b—C3—H3a	109.5	C19—C18—H18	120.97 (8)
H3c—C3—N3	109.5	C18—C19—O11	116.62 (12)
H3c—C3—H3a	109.5	C20—C19—O11	120.47 (12)
H3c—C3—H3b	109.5	C20—C19—C18	122.90 (12)
C5—C4—N3	121.27 (12)	C15—C20—C14	122.87 (12)
N9—C4—N3	127.25 (12)	C19—C20—C14	119.91 (12)
N9—C4—C5	111.48 (11)	C19—C20—C15	117.17 (12)
C6—C5—C4	123.27 (12)	C26—C21—C12	123.24 (12)
N7—C5—C4	105.17 (11)	C22—C21—C12	118.93 (12)
N7—C5—C6	131.54 (12)	C22—C21—C26	117.82 (12)
C5—C6—N1	112.04 (11)	H26—C26—C21	119.55 (8)
O6—C6—N1	120.80 (12)	C25—C26—C21	120.89 (13)
O6—C6—C5	127.16 (12)	C25—C26—H26	119.55 (8)
C8—N7—C5	106.50 (11)	H25—C25—C26	119.91 (8)
H7—N7—C5	124.7 (13)	C24—C25—C26	120.18 (13)
H7—N7—C8	128.7 (13)	C24—C25—H25	119.91 (8)
H8—C8—N7	123.34 (7)	H24—O24—C24	111.3 (14)
N9—C8—N7	113.31 (12)	O24—C24—C25	123.04 (13)
N9—C8—H8	123.34 (8)	C23—C24—C25	119.81 (13)
C8—N9—C4	103.53 (11)	C23—C24—O24	117.16 (13)
C19—O11—C12	121.92 (10)	H23—C23—C24	120.09 (8)
C13—C12—O11	120.18 (11)	C22—C23—C24	119.82 (13)
C21—C12—O11	110.27 (11)	C22—C23—H23	120.09 (8)
C21—C12—C13	129.55 (12)	C23—C22—C21	121.45 (13)
H13—O13—C13	113.2 (16)	H22—C22—C21	119.27 (8)
O13—C13—C12	120.54 (12)	H22—C22—C23	119.27 (8)
C14—C13—C12	120.97 (12)		

N1—C2—N3—C3	176.87 (12)	C12—C21—C22—C23	179.75 (13)
N1—C2—N3—C4	−3.15 (14)	O13—C13—C14—O14	−0.22 (14)
N1—C6—C5—C4	−0.32 (14)	O13—C13—C14—C20	−179.09 (12)
N1—C6—C5—N7	177.86 (10)	C13—C14—C20—C15	179.04 (11)
O2—C2—N3—C3	−2.35 (16)	C13—C14—C20—C19	1.43 (14)
O2—C2—N3—C4	177.63 (13)	O14—C14—C20—C15	0.20 (16)
C2—N3—C4—C5	2.57 (14)	O14—C14—C20—C19	−177.41 (13)
C2—N3—C4—N9	−176.80 (12)	C14—C20—C15—O15	2.29 (14)
N3—C4—C5—C6	−0.75 (15)	C14—C20—C15—C16	−176.60 (12)
N3—C4—C5—N7	−179.33 (13)	C14—C20—C19—C18	176.89 (12)
N3—C4—N9—C8	179.40 (15)	O15—C15—C16—C17	−178.86 (12)
C4—C5—C6—O6	179.34 (13)	O15—C15—C20—C19	179.97 (12)
C4—C5—N7—C8	−0.18 (12)	C15—C16—C17—O17	178.17 (12)
C4—N9—C8—N7	−0.10 (11)	C15—C16—C17—C18	−1.43 (15)
C5—N7—C8—N9	0.18 (11)	C15—C20—C19—C18	−0.85 (14)
O11—C12—C13—O13	178.97 (12)	C16—C17—C18—C19	1.64 (15)
O11—C12—C13—C14	0.33 (15)	O17—C17—C18—C19	−177.93 (13)
O11—C12—C21—C26	173.69 (11)	C17—C18—C19—C20	−0.47 (15)
O11—C12—C21—C22	−6.73 (13)	C21—C26—C25—C24	−0.63 (17)
O11—C19—C18—C17	178.84 (12)	C21—C22—C23—C24	−0.81 (17)

O11—C19—C20—C14	−2.39 (15)	C26—C25—C24—O24	179.19 (14)
O11—C19—C20—C15	179.86 (12)	C26—C25—C24—C23	−0.86 (18)
C12—C13—C14—O14	178.44 (13)	C25—C24—C23—C22	1.57 (18)
C12—C13—C14—C20	−0.43 (15)	O24—C24—C23—C22	−178.48 (14)
C12—C21—C26—C25	−179.05 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O15^{i}	0.92 (2)	1.92 (2)	2.8335 (15)	172.2 (18)
O17—H17···O6^{ii}	0.91 (2)	1.97 (2)	2.8663 (14)	168 (2)
O24—H24···O2	0.92 (2)	1.77 (2)	2.6796 (14)	170 (2)
O15—H15···O14	0.92 (2)	1.75 (2)	2.6064 (13)	154 (2)
O13—H13···N9^{iii}	0.89 (3)	1.95 (3)	2.7942 (15)	157 (2)

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*, −*y*+2, −*z*+1.

3,7-Dihydro-1,3-dimethyl-1*H*-purine-2,6-dione–\ 3,5,7-trihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one–\ water (2/2/1) (1c-ywj16bcult_auto)

Crystal data

$2C_7H_8N_4O_2·2C_{15}H_{10}O_8·H_2O$
M_r = 1014.82
Monoclinic, $P2_1/c$
a = 7.3270 (1) Å
b = 32.4057 (4) Å
c = 17.8062 (2) Å
β = 97.827 (1)°
V = 4188.46 (9) Å^3
Z = 4
$F(000)$ = 2104
D_x = 1.609 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 8351 reflections
θ = 2.7–74.7°
μ = 1.12 mm^{-1}
T = 100 K
Needle, yellow
0.1 × 0.02 × 0.01 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3577 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)
T_{min} = 0.568, T_{max} = 1.000
25287 measured reflections
8359 independent reflections
5887 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 74.6°, θ_{min} = 2.7°
h = −9→8
k = −39→39
l = −15→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.118
S = 0.99
8359 reflections
726 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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.

Refinement. X-ray diffraction data of **1** were collected at 100 K on Rigaku–Oxford Diffraction Supernova using a Cu *Kα* or Mo *Kα* source, or on a Bruker D8 Venture with Excillum Metal Jet with liquid gallium alloy Ga *Kα* source. *SADABS* (Bruker, 2008) was used for data reduction. All the structures were solved using the charge-flipping algorithm from an embedded *SHELXL* program (Sheldrick, 2015*b*) and refined from within the *OLEX2* suite (Dolomanov *et al.*, 2009; Bourhis *et al.* 2015).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2434 (3)	0.25340 (6)	0.40629 (11)	0.0239 (4)
C1	0.2511 (4)	0.29345 (7)	0.44569 (14)	0.0307 (5)
H1A	0.155020	0.311660	0.420423	0.046*
H1B	0.372169	0.306093	0.444374	0.046*
H1C	0.231396	0.289244	0.498486	0.046*
O2	0.3373 (2)	0.28594 (5)	0.30430 (9)	0.0283 (4)
C2	0.2921 (3)	0.25376 (7)	0.33314 (13)	0.0236 (5)
N3	0.2882 (3)	0.21711 (6)	0.29438 (11)	0.0241 (4)
C3	0.3488 (4)	0.21616 (8)	0.21892 (14)	0.0316 (6)
H3A	0.372411	0.187561	0.205175	0.047*
H3B	0.461937	0.232378	0.219837	0.047*
H3C	0.252283	0.227910	0.181499	0.047*
C4	0.2348 (3)	0.18171 (7)	0.32825 (13)	0.0234 (5)
C5	0.1902 (3)	0.18230 (7)	0.40065 (12)	0.0229 (5)
O6	0.1563 (2)	0.22088 (5)	0.51068 (9)	0.0272 (4)
C6	0.1927 (3)	0.21852 (7)	0.44546 (12)	0.0222 (5)
N7	0.1452 (3)	0.14215 (6)	0.41536 (11)	0.0261 (4)
C8	0.1637 (4)	0.12056 (8)	0.35277 (13)	0.0289 (5)
H8	0.140008	0.091788	0.348185	0.035*
N9	0.2182 (3)	0.14316 (6)	0.29758 (11)	0.0275 (4)
O11	−0.3211 (2)	−0.00655 (5)	−0.06654 (8)	0.0201 (3)
C12	−0.2750 (3)	−0.01447 (7)	0.00945 (12)	0.0202 (4)
O13	−0.2825 (2)	−0.06037 (5)	0.11379 (9)	0.0256 (3)
C13	−0.3197 (3)	−0.05149 (7)	0.03864 (12)	0.0205 (4)
O14	−0.4411 (2)	−0.11772 (5)	0.01930 (9)	0.0253 (3)
C14	−0.4065 (3)	−0.08374 (7)	−0.00914 (12)	0.0203 (4)
O15	−0.5684 (3)	−0.14184 (5)	−0.11795 (9)	0.0262 (4)
C15	−0.5335 (3)	−0.10338 (7)	−0.14156 (12)	0.0211 (4)
C16	−0.5775 (3)	−0.09292 (7)	−0.21645 (12)	0.0245 (5)
H16	−0.635347	−0.112388	−0.251851	0.029*
O17	−0.5840 (3)	−0.04534 (5)	−0.31522 (9)	0.0350 (5)
C17	−0.5360 (4)	−0.05323 (7)	−0.24013 (12)	0.0247 (5)
C18	−0.4493 (3)	−0.02400 (7)	−0.19030 (12)	0.0222 (5)
H18	−0.420086	0.002684	−0.207247	0.027*

C19	−0.4074 (3)	−0.03542 (7)	−0.11482 (12)	0.0190 (4)
C20	−0.4489 (3)	−0.07426 (7)	−0.08834 (11)	0.0193 (4)
C21	−0.1782 (3)	0.02056 (7)	0.04855 (12)	0.0190 (4)
C22	−0.1513 (3)	0.05638 (7)	0.00741 (12)	0.0212 (4)
H22	−0.198441	0.057943	−0.044920	0.025*
O23	−0.0259 (3)	0.12380 (5)	0.00140 (9)	0.0272 (4)
C23	−0.0564 (3)	0.08943 (7)	0.04278 (12)	0.0216 (5)
O24	0.1062 (2)	0.12221 (5)	0.14907 (9)	0.0254 (3)
C24	0.0128 (3)	0.08805 (7)	0.11971 (13)	0.0213 (5)
O25	0.0541 (3)	0.05305 (5)	0.23624 (9)	0.0280 (4)
C25	−0.0155 (3)	0.05276 (7)	0.16084 (12)	0.0226 (5)
C26	−0.1095 (3)	0.01904 (7)	0.12585 (12)	0.0216 (4)
H26	−0.127047	−0.005035	0.154460	0.026*
N1A	−0.0578 (3)	0.06189 (6)	0.62626 (10)	0.0236 (4)
C1A	−0.0530 (4)	0.02203 (7)	0.58675 (14)	0.0325 (6)
H1AA	0.054293	0.006135	0.609485	0.049*
H1AB	−0.044365	0.026911	0.533031	0.049*
H1AC	−0.165726	0.006528	0.591452	0.049*
O2A	−0.1410 (3)	0.02695 (5)	0.72736 (9)	0.0286 (4)
C2A	−0.1065 (3)	0.05993 (7)	0.69931 (12)	0.0218 (4)
N3A	−0.1144 (3)	0.09642 (6)	0.73775 (10)	0.0220 (4)
C3A	−0.1549 (4)	0.09551 (7)	0.81632 (12)	0.0269 (5)
H3AA	−0.052586	0.108057	0.849767	0.040*
H3AB	−0.170856	0.066852	0.831758	0.040*
H3AC	−0.268267	0.111012	0.819844	0.040*
C4A	−0.0766 (3)	0.13284 (7)	0.70346 (11)	0.0200 (4)
C5A	−0.0241 (3)	0.13367 (7)	0.63253 (12)	0.0205 (4)
O6A	0.0413 (3)	0.09507 (5)	0.52606 (9)	0.0286 (4)
C6A	−0.0085 (3)	0.09742 (7)	0.58942 (12)	0.0228 (5)
N7A	0.0020 (3)	0.17491 (6)	0.61789 (11)	0.0215 (4)
C8A	−0.0350 (3)	0.19561 (7)	0.67886 (12)	0.0222 (5)
H8A	−0.026818	0.224789	0.683290	0.027*
N9A	−0.0847 (3)	0.17121 (6)	0.73294 (10)	0.0220 (4)
O11A	−0.4403 (2)	0.32994 (5)	1.06778 (8)	0.0204 (3)
C12A	−0.3879 (3)	0.33807 (7)	0.99814 (12)	0.0193 (4)
O13A	−0.3451 (3)	0.38602 (5)	0.90218 (9)	0.0290 (4)
C13A	−0.3958 (3)	0.37751 (7)	0.97084 (12)	0.0216 (5)
O14A	−0.4637 (3)	0.44749 (5)	0.98821 (9)	0.0285 (4)
C14A	−0.4566 (3)	0.41140 (7)	1.01410 (12)	0.0227 (5)
O15A	−0.5714 (3)	0.47130 (5)	1.11445 (9)	0.0273 (4)
C15A	−0.5637 (3)	0.43117 (7)	1.13664 (12)	0.0227 (5)
C16A	−0.6119 (3)	0.42038 (7)	1.20625 (12)	0.0227 (5)
H16A	−0.650312	0.440783	1.239076	0.027*
O17A	−0.6539 (3)	0.36981 (5)	1.29622 (9)	0.0255 (4)
C17A	−0.6031 (3)	0.37859 (7)	1.22765 (12)	0.0216 (4)
C18A	−0.5460 (3)	0.34819 (7)	1.18129 (12)	0.0204 (4)
H18A	−0.540317	0.320071	1.196549	0.025*
C19A	−0.4974 (3)	0.36018 (7)	1.11179 (12)	0.0197 (4)

C20A	−0.5052 (3)	0.40110 (7)	1.08738 (12)	0.0210 (4)
C21A	−0.3303 (3)	0.30017 (7)	0.96276 (12)	0.0189 (4)
C22A	−0.3296 (3)	0.26307 (7)	1.00371 (12)	0.0205 (4)
H22A	−0.363515	0.263060	1.053334	0.025*
O23A	−0.2790 (3)	0.19101 (5)	1.01249 (9)	0.0285 (4)
C23A	−0.2795 (3)	0.22665 (7)	0.97184 (12)	0.0219 (5)
O24A	−0.1882 (3)	0.18869 (5)	0.87189 (9)	0.0266 (4)
C24A	−0.2315 (3)	0.22579 (7)	0.89908 (12)	0.0204 (4)
O25A	−0.1718 (3)	0.25842 (5)	0.78955 (9)	0.0320 (4)
C25A	−0.2280 (3)	0.26256 (7)	0.85876 (12)	0.0221 (5)
C26A	−0.2774 (3)	0.29965 (7)	0.88977 (12)	0.0222 (4)
H26A	−0.275455	0.324542	0.861721	0.027*
O1W	−0.1342 (4)	0.31328 (8)	0.68921 (14)	0.0574 (7)
H24A	−0.161 (5)	0.1892 (10)	0.827 (2)	0.043 (9)*
H7A	0.042 (4)	0.1860 (9)	0.5786 (16)	0.025 (7)*
H24	0.143 (5)	0.1177 (10)	0.201 (2)	0.043 (9)*
H7	0.110 (4)	0.1319 (10)	0.4618 (18)	0.040 (8)*
H23A	−0.242 (5)	0.1707 (11)	0.9869 (19)	0.044 (9)*
H23	0.049 (5)	0.1394 (10)	0.0342 (18)	0.038 (8)*
H25A	−0.171 (5)	0.2801 (12)	0.768 (2)	0.050 (10)*
H13A	−0.375 (5)	0.4076 (11)	0.894 (2)	0.045 (10)*
H13	−0.319 (4)	−0.0843 (10)	0.1198 (16)	0.026 (7)*
H15A	−0.543 (5)	0.4733 (10)	1.070 (2)	0.045 (9)*
H15	−0.541 (5)	−0.1424 (10)	−0.069 (2)	0.043 (9)*
H17	−0.558 (5)	−0.0231 (12)	−0.326 (2)	0.057 (11)*
H25	0.070 (6)	0.0288 (13)	0.254 (2)	0.064 (12)*
H17A	−0.654 (5)	0.3474 (11)	1.3011 (18)	0.035 (9)*
H1WA	−0.057 (7)	0.3303 (16)	0.685 (3)	0.094 (17)*
H1WB	−0.235 (7)	0.3205 (15)	0.663 (3)	0.085 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0289 (11)	0.0175 (9)	0.0257 (9)	−0.0008 (8)	0.0053 (8)	−0.0003 (7)
C1	0.0418 (15)	0.0192 (12)	0.0321 (12)	0.0004 (10)	0.0090 (11)	−0.0022 (9)
O2	0.0351 (10)	0.0193 (8)	0.0326 (8)	−0.0001 (7)	0.0117 (7)	0.0046 (7)
C2	0.0220 (11)	0.0232 (12)	0.0265 (11)	0.0011 (9)	0.0062 (9)	0.0037 (9)
N3	0.0290 (11)	0.0207 (10)	0.0242 (9)	0.0002 (8)	0.0090 (8)	0.0009 (7)
C3	0.0437 (15)	0.0260 (13)	0.0282 (12)	0.0012 (11)	0.0165 (11)	0.0012 (10)
C4	0.0253 (12)	0.0210 (11)	0.0246 (11)	0.0009 (9)	0.0059 (9)	−0.0004 (9)
C5	0.0262 (12)	0.0198 (11)	0.0235 (10)	−0.0007 (9)	0.0058 (9)	0.0006 (9)
O6	0.0380 (10)	0.0222 (8)	0.0226 (8)	−0.0015 (7)	0.0085 (7)	−0.0013 (6)
C6	0.0244 (12)	0.0196 (11)	0.0229 (10)	−0.0009 (9)	0.0042 (9)	0.0006 (8)
N7	0.0345 (12)	0.0206 (10)	0.0242 (9)	−0.0014 (8)	0.0078 (8)	−0.0014 (8)
C8	0.0408 (14)	0.0184 (11)	0.0288 (11)	−0.0004 (10)	0.0093 (10)	−0.0040 (9)
N9	0.0372 (12)	0.0216 (10)	0.0251 (9)	−0.0009 (8)	0.0093 (8)	−0.0024 (8)
O11	0.0272 (8)	0.0149 (7)	0.0195 (7)	−0.0014 (6)	0.0077 (6)	0.0006 (6)
C12	0.0223 (11)	0.0194 (11)	0.0202 (10)	0.0024 (9)	0.0080 (8)	0.0005 (8)

O13	0.0371 (10)	0.0183 (8)	0.0213 (7)	−0.0058 (7)	0.0041 (7)	0.0038 (6)
C13	0.0224 (11)	0.0196 (11)	0.0205 (10)	0.0023 (8)	0.0069 (8)	0.0012 (8)
O14	0.0358 (9)	0.0167 (8)	0.0235 (7)	−0.0036 (7)	0.0045 (7)	0.0062 (6)
C14	0.0216 (11)	0.0173 (10)	0.0236 (10)	0.0009 (8)	0.0089 (9)	0.0028 (8)
O15	0.0415 (10)	0.0165 (8)	0.0212 (8)	−0.0050 (7)	0.0069 (7)	0.0034 (6)
C15	0.0258 (11)	0.0142 (10)	0.0250 (10)	0.0009 (8)	0.0090 (9)	0.0020 (8)
C16	0.0363 (13)	0.0185 (11)	0.0204 (10)	−0.0026 (9)	0.0100 (9)	−0.0015 (8)
O17	0.0730 (14)	0.0155 (8)	0.0168 (8)	−0.0100 (9)	0.0073 (8)	0.0005 (6)
C17	0.0398 (14)	0.0179 (11)	0.0182 (10)	−0.0012 (9)	0.0106 (9)	0.0017 (8)
C18	0.0326 (13)	0.0147 (10)	0.0211 (10)	−0.0019 (9)	0.0105 (9)	0.0021 (8)
C19	0.0215 (11)	0.0168 (10)	0.0207 (10)	−0.0009 (8)	0.0095 (8)	−0.0024 (8)
C20	0.0221 (11)	0.0190 (11)	0.0189 (10)	0.0028 (8)	0.0096 (8)	0.0009 (8)
C21	0.0182 (11)	0.0164 (10)	0.0241 (10)	0.0007 (8)	0.0087 (8)	0.0001 (8)
C22	0.0241 (11)	0.0205 (11)	0.0204 (10)	0.0014 (9)	0.0085 (8)	0.0003 (8)
O23	0.0378 (10)	0.0178 (8)	0.0268 (8)	−0.0042 (7)	0.0078 (7)	0.0010 (6)
C23	0.0253 (11)	0.0158 (10)	0.0259 (11)	0.0033 (9)	0.0115 (9)	0.0019 (8)
O24	0.0324 (9)	0.0162 (8)	0.0280 (8)	−0.0004 (7)	0.0058 (7)	−0.0014 (6)
C24	0.0224 (11)	0.0154 (10)	0.0280 (11)	0.0006 (8)	0.0100 (9)	−0.0044 (8)
O25	0.0422 (11)	0.0176 (8)	0.0233 (8)	0.0012 (7)	0.0009 (7)	−0.0003 (6)
C25	0.0276 (12)	0.0184 (11)	0.0220 (10)	0.0047 (9)	0.0036 (9)	−0.0016 (8)
C26	0.0278 (12)	0.0138 (10)	0.0243 (10)	0.0027 (9)	0.0073 (9)	0.0021 (8)
N1A	0.0340 (11)	0.0159 (9)	0.0223 (9)	0.0009 (8)	0.0082 (8)	−0.0021 (7)
C1A	0.0556 (17)	0.0162 (11)	0.0282 (11)	0.0018 (11)	0.0148 (11)	−0.0028 (9)
O2A	0.0451 (11)	0.0158 (8)	0.0262 (8)	0.0003 (7)	0.0098 (7)	0.0030 (6)
C2A	0.0281 (12)	0.0184 (11)	0.0194 (10)	0.0028 (9)	0.0051 (9)	0.0020 (8)
N3A	0.0317 (11)	0.0175 (9)	0.0177 (8)	0.0014 (8)	0.0066 (7)	0.0003 (7)
C3A	0.0445 (15)	0.0197 (11)	0.0183 (10)	0.0011 (10)	0.0115 (10)	0.0026 (8)
C4A	0.0251 (11)	0.0166 (10)	0.0183 (9)	0.0018 (8)	0.0033 (8)	−0.0007 (8)
C5A	0.0233 (11)	0.0189 (11)	0.0201 (10)	0.0008 (9)	0.0057 (8)	0.0022 (8)
O6A	0.0438 (10)	0.0217 (8)	0.0231 (8)	0.0023 (7)	0.0149 (7)	−0.0005 (6)
C6A	0.0272 (12)	0.0209 (11)	0.0212 (10)	−0.0003 (9)	0.0058 (9)	0.0027 (9)
N7A	0.0283 (10)	0.0168 (9)	0.0209 (9)	−0.0001 (8)	0.0084 (8)	0.0013 (7)
C8A	0.0275 (12)	0.0174 (11)	0.0224 (10)	−0.0002 (9)	0.0059 (9)	−0.0008 (8)
N9A	0.0293 (10)	0.0172 (9)	0.0204 (8)	0.0003 (8)	0.0066 (8)	−0.0003 (7)
O11A	0.0307 (9)	0.0127 (7)	0.0192 (7)	0.0011 (6)	0.0081 (6)	−0.0018 (6)
C12A	0.0244 (11)	0.0163 (10)	0.0176 (9)	−0.0008 (8)	0.0043 (8)	−0.0011 (8)
O13A	0.0576 (12)	0.0134 (8)	0.0178 (7)	0.0063 (8)	0.0114 (7)	0.0022 (6)
C13A	0.0317 (12)	0.0172 (11)	0.0162 (9)	0.0016 (9)	0.0045 (9)	−0.0012 (8)
O14A	0.0498 (11)	0.0149 (8)	0.0218 (7)	0.0037 (7)	0.0085 (7)	0.0004 (6)
C14A	0.0306 (12)	0.0168 (11)	0.0198 (10)	0.0010 (9)	0.0008 (9)	0.0005 (8)
O15A	0.0492 (11)	0.0135 (8)	0.0207 (8)	0.0042 (7)	0.0100 (7)	−0.0011 (6)
C15A	0.0324 (12)	0.0143 (10)	0.0211 (10)	0.0023 (9)	0.0023 (9)	−0.0004 (8)
C16A	0.0276 (12)	0.0178 (11)	0.0231 (10)	0.0006 (9)	0.0052 (9)	−0.0064 (8)
O17A	0.0406 (10)	0.0158 (9)	0.0231 (8)	−0.0001 (7)	0.0148 (7)	−0.0015 (6)
C17A	0.0248 (11)	0.0208 (11)	0.0196 (10)	−0.0031 (9)	0.0046 (8)	−0.0026 (8)
C18A	0.0245 (11)	0.0153 (10)	0.0225 (10)	0.0009 (8)	0.0067 (8)	0.0002 (8)
C19A	0.0212 (11)	0.0166 (11)	0.0215 (10)	0.0014 (8)	0.0032 (8)	−0.0034 (8)
C20A	0.0257 (11)	0.0179 (11)	0.0194 (10)	0.0008 (9)	0.0031 (8)	−0.0019 (8)

C21A	0.0225 (11)	0.0144 (10)	0.0199 (10)	0.0006 (8)	0.0034 (8)	−0.0025 (8)
C22A	0.0266 (12)	0.0165 (10)	0.0198 (10)	−0.0009 (9)	0.0089 (8)	−0.0002 (8)
O23A	0.0516 (12)	0.0125 (8)	0.0256 (8)	0.0034 (7)	0.0197 (8)	0.0021 (6)
C23A	0.0285 (12)	0.0159 (11)	0.0224 (10)	0.0001 (9)	0.0076 (9)	0.0024 (8)
O24A	0.0472 (11)	0.0146 (8)	0.0209 (8)	0.0051 (7)	0.0151 (7)	0.0004 (6)
C24A	0.0256 (12)	0.0147 (10)	0.0216 (10)	0.0027 (8)	0.0056 (9)	−0.0029 (8)
O25A	0.0627 (13)	0.0168 (8)	0.0208 (8)	0.0050 (8)	0.0207 (8)	0.0028 (7)
C25A	0.0313 (13)	0.0178 (11)	0.0185 (10)	0.0008 (9)	0.0082 (9)	−0.0001 (8)
C26A	0.0323 (12)	0.0162 (10)	0.0188 (10)	−0.0005 (9)	0.0057 (9)	0.0024 (8)
O1W	0.0462 (14)	0.0630 (16)	0.0572 (13)	−0.0248 (12)	−0.0139 (11)	0.0407 (12)

Geometric parameters (Å, º)

N1—C1	1.473 (3)	N1A—C2A	1.396 (3)
N1—C2	1.397 (3)	N1A—C6A	1.397 (3)
N1—C6	1.405 (3)	C1A—H1AA	0.9800
C1—H1A	0.9800	C1A—H1AB	0.9800
C1—H1B	0.9800	C1A—H1AC	0.9800
C1—H1C	0.9800	O2A—C2A	1.221 (3)
O2—C2	1.228 (3)	C2A—N3A	1.371 (3)
C2—N3	1.372 (3)	N3A—C3A	1.469 (3)
N3—C3	1.472 (3)	N3A—C4A	1.374 (3)
N3—C4	1.377 (3)	C3A—H3AA	0.9800
C3—H3A	0.9800	C3A—H3AB	0.9800
C3—H3B	0.9800	C3A—H3AC	0.9800
C3—H3C	0.9800	C4A—C5A	1.369 (3)
C4—C5	1.373 (3)	C4A—N9A	1.354 (3)
C4—N9	1.362 (3)	C5A—C6A	1.416 (3)
C5—C6	1.418 (3)	C5A—N7A	1.380 (3)
C5—N7	1.376 (3)	O6A—C6A	1.234 (3)
O6—C6	1.229 (3)	N7A—C8A	1.335 (3)
N7—C8	1.338 (3)	N7A—H7A	0.87 (3)
N7—H7	0.96 (3)	C8A—H8A	0.9500
C8—H8	0.9500	C8A—N9A	1.334 (3)
C8—N9	1.330 (3)	O11A—C12A	1.373 (3)
O11—C12	1.374 (3)	O11A—C19A	1.356 (3)
O11—C19	1.366 (3)	C12A—C13A	1.366 (3)
C12—C13	1.365 (3)	C12A—C21A	1.468 (3)
C12—C21	1.463 (3)	O13A—C13A	1.353 (3)
O13—C13	1.360 (3)	O13A—H13A	0.74 (4)
O13—H13	0.83 (3)	C13A—C14A	1.446 (3)
C13—C14	1.440 (3)	O14A—C14A	1.256 (3)
O14—C14	1.252 (3)	C14A—C20A	1.438 (3)
C14—C20	1.435 (3)	O15A—C15A	1.358 (3)
O15—C15	1.351 (3)	O15A—H15A	0.84 (4)
O15—H15	0.87 (3)	C15A—C16A	1.380 (3)
C15—C16	1.371 (3)	C15A—C20A	1.416 (3)
C15—C20	1.419 (3)	C16A—H16A	0.9500

C16—H16	0.9500	C16A—C17A	1.406 (3)
C16—C17	1.400 (3)	O17A—C17A	1.354 (3)
O17—C17	1.360 (3)	O17A—H17A	0.73 (3)
O17—H17	0.77 (4)	C17A—C18A	1.386 (3)
C17—C18	1.391 (3)	C18A—H18A	0.9500
C18—H18	0.9500	C18A—C19A	1.389 (3)
C18—C19	1.388 (3)	C19A—C20A	1.394 (3)
C19—C20	1.392 (3)	C21A—C22A	1.406 (3)
C21—C22	1.401 (3)	C21A—C26A	1.406 (3)
C21—C26	1.401 (3)	C22A—H22A	0.9500
C22—H22	0.9500	C22A—C23A	1.381 (3)
C22—C23	1.381 (3)	O23A—C23A	1.363 (3)
O23—C23	1.370 (3)	O23A—H23A	0.86 (3)
O23—H23	0.90 (3)	C23A—C24A	1.388 (3)
C23—C24	1.395 (3)	O24A—C24A	1.350 (3)
O24—C24	1.367 (3)	O24A—H24A	0.85 (3)
O24—H24	0.93 (3)	C24A—C25A	1.393 (3)
C24—C25	1.389 (3)	O25A—C25A	1.358 (3)
O25—C25	1.370 (3)	O25A—H25A	0.81 (4)
O25—H25	0.85 (4)	C25A—C26A	1.391 (3)
C25—C26	1.393 (3)	C26A—H26A	0.9500
C26—H26	0.9500	O1W—H1WA	0.80 (5)
N1A—C1A	1.474 (3)	O1W—H1WB	0.85 (5)

C2—N1—C1	116.03 (19)	C2A—N1A—C6A	126.16 (19)
C2—N1—C6	126.03 (19)	C6A—N1A—C1A	118.29 (18)
C6—N1—C1	117.92 (19)	N1A—C1A—H1AA	109.5
N1—C1—H1A	109.5	N1A—C1A—H1AB	109.5
N1—C1—H1B	109.5	N1A—C1A—H1AC	109.5
N1—C1—H1C	109.5	H1AA—C1A—H1AB	109.5
H1A—C1—H1B	109.5	H1AA—C1A—H1AC	109.5
H1A—C1—H1C	109.5	H1AB—C1A—H1AC	109.5
H1B—C1—H1C	109.5	O2A—C2A—N1A	120.9 (2)
O2—C2—N1	121.0 (2)	O2A—C2A—N3A	121.70 (19)
O2—C2—N3	120.9 (2)	N3A—C2A—N1A	117.35 (19)
N3—C2—N1	118.13 (19)	C2A—N3A—C3A	119.06 (18)
C2—N3—C3	119.29 (19)	C2A—N3A—C4A	119.58 (17)
C2—N3—C4	119.25 (18)	C4A—N3A—C3A	121.30 (18)
C4—N3—C3	121.41 (19)	N3A—C3A—H3AA	109.5
N3—C3—H3A	109.5	N3A—C3A—H3AB	109.5
N3—C3—H3B	109.5	N3A—C3A—H3AC	109.5
N3—C3—H3C	109.5	H3AA—C3A—H3AB	109.5
H3A—C3—H3B	109.5	H3AA—C3A—H3AC	109.5
H3A—C3—H3C	109.5	H3AB—C3A—H3AC	109.5
H3B—C3—H3C	109.5	C5A—C4A—N3A	121.7 (2)
C5—C4—N3	121.2 (2)	N9A—C4A—N3A	126.44 (18)
N9—C4—N3	127.1 (2)	N9A—C4A—C5A	111.84 (19)
N9—C4—C5	111.7 (2)	C4A—C5A—C6A	122.5 (2)

C4—C5—C6	123.6 (2)	C4A—C5A—N7A	104.89 (19)
C4—C5—N7	105.0 (2)	N7A—C5A—C6A	132.63 (19)
N7—C5—C6	131.3 (2)	N1A—C6A—C5A	112.58 (18)
N1—C6—C5	111.75 (19)	O6A—C6A—N1A	120.4 (2)
O6—C6—N1	121.7 (2)	O6A—C6A—C5A	127.0 (2)
O6—C6—C5	126.6 (2)	C5A—N7A—H7A	128.5 (18)
C5—N7—H7	126.3 (19)	C8A—N7A—C5A	106.51 (18)
C8—N7—C5	106.35 (19)	C8A—N7A—H7A	124.9 (18)
C8—N7—H7	127.3 (19)	N7A—C8A—H8A	123.4
N7—C8—H8	123.1	N9A—C8A—N7A	113.2 (2)
N9—C8—N7	113.7 (2)	N9A—C8A—H8A	123.4
N9—C8—H8	123.1	C8A—N9A—C4A	103.52 (17)
C8—N9—C4	103.19 (19)	C19A—O11A—C12A	121.98 (17)
C19—O11—C12	121.52 (17)	O11A—C12A—C21A	111.06 (18)
O11—C12—C21	111.22 (18)	C13A—C12A—O11A	119.84 (19)
C13—C12—O11	120.0 (2)	C13A—C12A—C21A	129.10 (19)
C13—C12—C21	128.8 (2)	C13A—O13A—H13A	105 (3)
C13—O13—H13	107 (2)	C12A—C13A—C14A	121.47 (19)
C12—C13—C14	121.5 (2)	O13A—C13A—C12A	120.57 (19)
O13—C13—C12	122.3 (2)	O13A—C13A—C14A	117.96 (19)
O13—C13—C14	116.20 (19)	O14A—C14A—C13A	120.79 (19)
O14—C14—C13	119.8 (2)	O14A—C14A—C20A	123.1 (2)
O14—C14—C20	123.8 (2)	C20A—C14A—C13A	116.1 (2)
C20—C14—C13	116.39 (19)	C15A—O15A—H15A	110 (2)
C15—O15—H15	108 (2)	O15A—C15A—C16A	119.90 (19)
O15—C15—C16	120.1 (2)	O15A—C15A—C20A	118.87 (19)
O15—C15—C20	119.32 (19)	C16A—C15A—C20A	121.2 (2)
C16—C15—C20	120.6 (2)	C15A—C16A—H16A	120.7
C15—C16—H16	120.4	C15A—C16A—C17A	118.65 (19)
C15—C16—C17	119.2 (2)	C17A—C16A—H16A	120.7
C17—C16—H16	120.4	C17A—O17A—H17A	109 (2)
C17—O17—H17	112 (3)	O17A—C17A—C16A	116.01 (19)
O17—C17—C16	115.6 (2)	O17A—C17A—C18A	122.0 (2)
O17—C17—C18	122.3 (2)	C18A—C17A—C16A	122.00 (19)
C18—C17—C16	122.2 (2)	C17A—C18A—H18A	121.1
C17—C18—H18	121.4	C17A—C18A—C19A	117.8 (2)
C19—C18—C17	117.3 (2)	C19A—C18A—H18A	121.1
C19—C18—H18	121.4	O11A—C19A—C18A	116.70 (19)
O11—C19—C18	116.72 (19)	O11A—C19A—C20A	120.65 (19)
O11—C19—C20	120.65 (19)	C18A—C19A—C20A	122.6 (2)
C18—C19—C20	122.6 (2)	C15A—C20A—C14A	122.4 (2)
C15—C20—C14	122.0 (2)	C19A—C20A—C14A	119.94 (19)
C19—C20—C14	119.9 (2)	C19A—C20A—C15A	117.67 (19)
C19—C20—C15	118.09 (19)	C22A—C21A—C12A	118.27 (18)
C22—C21—C12	119.2 (2)	C22A—C21A—C26A	119.16 (19)
C26—C21—C12	121.75 (19)	C26A—C21A—C12A	122.56 (19)
C26—C21—C22	119.1 (2)	C21A—C22A—H22A	120.0
C21—C22—H22	119.9	C23A—C22A—C21A	120.02 (19)

C23—C22—C21	120.1 (2)	C23A—C22A—H22A	120.0
C23—C22—H22	119.9	C23A—O23A—H23A	110 (2)
C23—O23—H23	104 (2)	C22A—C23A—C24A	121.1 (2)
C22—C23—C24	121.1 (2)	O23A—C23A—C22A	119.21 (18)
O23—C23—C22	119.6 (2)	O23A—C23A—C24A	119.73 (19)
O23—C23—C24	119.3 (2)	C24A—O24A—H24A	115 (2)
C24—O24—H24	108 (2)	C23A—C24A—C25A	119.25 (19)
O24—C24—C23	116.5 (2)	O24A—C24A—C23A	117.03 (19)
O24—C24—C25	124.6 (2)	O24A—C24A—C25A	123.72 (19)
C25—C24—C23	118.9 (2)	C25A—O25A—H25A	112 (3)
C25—O25—H25	111 (3)	O25A—C25A—C24A	114.34 (19)
C24—C25—C26	120.7 (2)	O25A—C25A—C26A	125.0 (2)
O25—C25—C24	116.7 (2)	C26A—C25A—C24A	120.69 (19)
O25—C25—C26	122.6 (2)	C21A—C26A—H26A	120.1
C21—C26—H26	119.9	C25A—C26A—C21A	119.8 (2)
C25—C26—C21	120.1 (2)	C25A—C26A—H26A	120.1
C25—C26—H26	119.9	H1WA—O1W—H1WB	110 (5)
C2A—N1A—C1A	115.51 (18)		

N1—C2—N3—C3	−176.6 (2)	N1A—C2A—N3A—C3A	−176.6 (2)
N1—C2—N3—C4	0.8 (3)	N1A—C2A—N3A—C4A	0.6 (3)
C1—N1—C2—O2	−0.4 (3)	C1A—N1A—C2A—O2A	0.4 (3)
C1—N1—C2—N3	179.6 (2)	C1A—N1A—C2A—N3A	−179.3 (2)
C1—N1—C6—C5	179.7 (2)	C1A—N1A—C6A—C5A	178.3 (2)
C1—N1—C6—O6	−0.3 (4)	C1A—N1A—C6A—O6A	−1.5 (4)
O2—C2—N3—C3	3.3 (4)	O2A—C2A—N3A—C3A	3.6 (4)
O2—C2—N3—C4	−179.3 (2)	O2A—C2A—N3A—C4A	−179.2 (2)
C2—N1—C6—C5	−1.9 (3)	C2A—N1A—C6A—C5A	−4.0 (3)
C2—N1—C6—O6	178.1 (2)	C2A—N1A—C6A—O6A	176.2 (2)
C2—N3—C4—C5	−1.7 (4)	C2A—N3A—C4A—C5A	−2.4 (3)
C2—N3—C4—N9	178.1 (2)	C2A—N3A—C4A—N9A	177.6 (2)
N3—C4—C5—C6	0.8 (4)	N3A—C4A—C5A—C6A	1.1 (4)
N3—C4—C5—N7	−179.8 (2)	N3A—C4A—C5A—N7A	−179.7 (2)
N3—C4—N9—C8	180.0 (2)	N3A—C4A—N9A—C8A	179.6 (2)
C3—N3—C4—C5	175.6 (2)	C3A—N3A—C4A—C5A	174.6 (2)
C3—N3—C4—N9	−4.6 (4)	C3A—N3A—C4A—N9A	−5.3 (4)
C4—C5—C6—N1	0.9 (3)	C4A—C5A—C6A—N1A	1.9 (3)
C4—C5—C6—O6	−179.1 (2)	C4A—C5A—C6A—O6A	−178.3 (2)
C4—C5—N7—C8	−0.4 (3)	C4A—C5A—N7A—C8A	0.0 (3)
C5—C4—N9—C8	−0.2 (3)	C5A—C4A—N9A—C8A	−0.4 (3)
C5—N7—C8—N9	0.3 (3)	C5A—N7A—C8A—N9A	−0.3 (3)
C6—N1—C2—O2	−178.8 (2)	C6A—N1A—C2A—O2A	−177.3 (2)
C6—N1—C2—N3	1.1 (4)	C6A—N1A—C2A—N3A	2.9 (4)
C6—C5—N7—C8	178.9 (3)	C6A—C5A—N7A—C8A	179.1 (3)
N7—C5—C6—N1	−178.3 (2)	N7A—C5A—C6A—N1A	−177.0 (2)
N7—C5—C6—O6	1.7 (4)	N7A—C5A—C6A—O6A	2.8 (4)
N7—C8—N9—C4	0.0 (3)	N7A—C8A—N9A—C4A	0.4 (3)
N9—C4—C5—C6	−179.0 (2)	N9A—C4A—C5A—C6A	−178.9 (2)

N9—C4—C5—N7	0.4 (3)	N9A—C4A—C5A—N7A	0.2 (3)
O11—C12—C13—O13	−177.88 (19)	O11A—C12A—C13A—O13A	−179.8 (2)
O11—C12—C13—C14	3.3 (3)	O11A—C12A—C13A—C14A	0.6 (4)
O11—C12—C21—C22	2.2 (3)	O11A—C12A—C21A—C22A	−2.3 (3)
O11—C12—C21—C26	−176.95 (19)	O11A—C12A—C21A—C26A	177.3 (2)
O11—C19—C20—C14	2.1 (3)	O11A—C19A—C20A—C14A	1.5 (3)
O11—C19—C20—C15	−178.14 (19)	O11A—C19A—C20A—C15A	−179.1 (2)
C12—O11—C19—C18	179.3 (2)	C12A—O11A—C19A—C18A	−179.7 (2)
C12—O11—C19—C20	−1.0 (3)	C12A—O11A—C19A—C20A	0.1 (3)
C12—C13—C14—O14	178.1 (2)	C12A—C13A—C14A—O14A	−179.8 (2)
C12—C13—C14—C20	−2.1 (3)	C12A—C13A—C14A—C20A	0.9 (4)
C12—C21—C22—C23	−178.4 (2)	C12A—C21A—C22A—C23A	178.8 (2)
C12—C21—C26—C25	179.0 (2)	C12A—C21A—C26A—C25A	−178.6 (2)
O13—C13—C14—O14	−0.8 (3)	O13A—C13A—C14A—O14A	0.7 (4)
O13—C13—C14—C20	179.02 (19)	O13A—C13A—C14A—C20A	−178.7 (2)
C13—C12—C21—C22	−178.2 (2)	C13A—C12A—C21A—C22A	178.3 (2)
C13—C12—C21—C26	2.7 (4)	C13A—C12A—C21A—C26A	−2.2 (4)
C13—C14—C20—C15	179.66 (19)	C13A—C14A—C20A—C15A	178.7 (2)
C13—C14—C20—C19	−0.6 (3)	C13A—C14A—C20A—C19A	−1.9 (3)
O14—C14—C20—C15	−0.5 (3)	O14A—C14A—C20A—C15A	−0.6 (4)
O14—C14—C20—C19	179.2 (2)	O14A—C14A—C20A—C19A	178.7 (2)
O15—C15—C16—C17	−178.4 (2)	O15A—C15A—C16A—C17A	179.8 (2)
O15—C15—C20—C14	−2.9 (3)	O15A—C15A—C20A—C14A	−1.1 (4)
O15—C15—C20—C19	177.3 (2)	O15A—C15A—C20A—C19A	179.6 (2)
C15—C16—C17—O17	180.0 (2)	C15A—C16A—C17A—O17A	−179.3 (2)
C15—C16—C17—C18	0.8 (4)	C15A—C16A—C17A—C18A	0.6 (4)
C16—C15—C20—C14	177.9 (2)	C16A—C15A—C20A—C14A	179.1 (2)
C16—C15—C20—C19	−1.8 (3)	C16A—C15A—C20A—C19A	−0.2 (4)
C16—C17—C18—C19	−1.1 (4)	C16A—C17A—C18A—C19A	−0.2 (4)
O17—C17—C18—C19	179.8 (2)	O17A—C17A—C18A—C19A	179.7 (2)
C17—C18—C19—O11	179.56 (19)	C17A—C18A—C19A—O11A	179.4 (2)
C17—C18—C19—C20	−0.1 (3)	C17A—C18A—C19A—C20A	−0.4 (4)
C18—C19—C20—C14	−178.2 (2)	C18A—C19A—C20A—C14A	−178.7 (2)
C18—C19—C20—C15	1.5 (3)	C18A—C19A—C20A—C15A	0.6 (4)
C19—O11—C12—C13	−1.8 (3)	C19A—O11A—C12A—C13A	−1.2 (3)
C19—O11—C12—C21	177.89 (17)	C19A—O11A—C12A—C21A	179.3 (2)
C20—C15—C16—C17	0.7 (4)	C20A—C15A—C16A—C17A	−0.4 (4)
C21—C12—C13—O13	2.5 (4)	C21A—C12A—C13A—O13A	−0.4 (4)
C21—C12—C13—C14	−176.3 (2)	C21A—C12A—C13A—C14A	−180.0 (2)
C21—C22—C23—O23	177.94 (19)	C21A—C22A—C23A—O23A	179.8 (2)
C21—C22—C23—C24	−0.6 (3)	C21A—C22A—C23A—C24A	−0.8 (4)
C22—C21—C26—C25	−0.2 (3)	C22A—C21A—C26A—C25A	0.9 (4)
C22—C23—C24—O24	179.38 (19)	C22A—C23A—C24A—O24A	−178.7 (2)
C22—C23—C24—C25	−0.2 (3)	C22A—C23A—C24A—C25A	2.2 (4)
O23—C23—C24—O24	0.9 (3)	O23A—C23A—C24A—O24A	0.8 (3)
O23—C23—C24—C25	−178.7 (2)	O23A—C23A—C24A—C25A	−178.4 (2)
C23—C24—C25—O25	−179.6 (2)	C23A—C24A—C25A—O25A	177.4 (2)
C23—C24—C25—C26	0.7 (3)	C23A—C24A—C25A—C26A	−2.0 (4)

O24—C24—C25—O25	0.9 (3)	O24A—C24A—C25A—O25A	−1.7 (4)
O24—C24—C25—C26	−178.8 (2)	O24A—C24A—C25A—C26A	178.9 (2)
C24—C25—C26—C21	−0.6 (3)	C24A—C25A—C26A—C21A	0.5 (4)
O25—C25—C26—C21	179.8 (2)	O25A—C25A—C26A—C21A	−178.8 (2)
C26—C21—C22—C23	0.7 (3)	C26A—C21A—C22A—C23A	−0.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O24*A*—H24*A*···N9*A*	0.85 (3)	1.92 (3)	2.744 (2)	161 (3)
N7*A*—H7*A*···O6	0.87 (3)	1.93 (3)	2.780 (2)	165 (3)
O24—H24···N9	0.93 (3)	1.93 (3)	2.745 (3)	146 (3)
N7—H7···O6*A*	0.96 (3)	1.77 (3)	2.684 (2)	158 (3)
O23—H23···O24	0.90 (3)	2.10 (3)	2.676 (2)	120 (3)
O25*A*—H25*A*···O1*W*	0.81 (4)	1.81 (4)	2.562 (3)	155 (4)
O13*A*—H13*A*···O17^{i}	0.74 (4)	2.06 (4)	2.720 (2)	148 (4)
O13*A*—H13*A*···O14*A*	0.74 (4)	2.29 (3)	2.726 (2)	119 (3)
O13—H13···O14	0.83 (3)	2.18 (3)	2.665 (2)	117 (2)
O15*A*—H15*A*···O14*A*	0.84 (4)	1.85 (3)	2.599 (2)	148 (3)
O15—H15···O14	0.87 (3)	1.82 (3)	2.614 (2)	150 (3)
O17—H17···O15*A*^{ii}	0.77 (4)	1.98 (4)	2.714 (2)	157 (4)
O25—H25···O2*A*^{iii}	0.85 (4)	1.90 (4)	2.727 (2)	166 (4)
O17*A*—H17*A*···O2^{iv}	0.73 (3)	1.99 (3)	2.723 (2)	175 (3)
O1*W*—H1*WB*···O15^{i}	0.85 (5)	1.97 (5)	2.778 (3)	157 (5)

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

3,7-Dihydro-1,3-dimethyl-1*H*-purine-2,6-dione–*rac*–5,7-dihydroxy-2-(3-hydroxy-4-methoxyphenyl)-2,3-dihydro-4*H*-1-benzopyran-4-one (1f-ywj137acu100k_auto)

Crystal data

$2C_7H_8N_4O_2·C_{16}H_{14}O_6$	Z = 2
M_r = 662.62	$F(000)$ = 692
Triclinic, $P\overline{1}$	D_x = 1.350 Mg m^{-3}
a = 6.8286 (2) Å	Cu $K\alpha$ radiation, λ = 1.54184 Å
b = 14.9790 (3) Å	Cell parameters from 12687 reflections
c = 16.0763 (4) Å	θ = 2.7–76.8°
α = 88.624 (2)°	μ = 0.88 mm^{-1}
β = 83.176 (2)°	T = 100 K
γ = 87.020 (2)°	Prism, colourless
V = 1630.25 (7) Å^3	0.05 × 0.03 × 0.01 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector	T_{min} = 0.935, T_{max} = 1.000
	26501 measured reflections
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source	6746 independent reflections
	6048 reflections with $I > 2\sigma(I)$
Mirror monochromator	R_{int} = 0.029
Detector resolution: 10.3577 pixels mm^{-1}	θ_{max} = 77.1°, θ_{min} = 3.0°
ω scans	h = −8→8
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)	k = −18→18
	l = −19→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.125$
$S = 1.05$
6746 reflections
469 parameters
3 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.9542P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O11	0.75613 (18)	0.16694 (8)	0.84601 (7)	0.0269 (3)	
O6	0.54233 (18)	1.06700 (8)	0.38537 (7)	0.0294 (3)	
O17	0.88283 (19)	0.11735 (8)	0.55313 (7)	0.0303 (3)	
O15	0.94956 (19)	−0.12590 (8)	0.73807 (8)	0.0307 (3)	
O24	0.50476 (18)	0.35328 (9)	1.20176 (8)	0.0333 (3)	
O2	0.70088 (19)	0.93804 (8)	0.12937 (7)	0.0316 (3)	
O6A	0.7177 (2)	0.59707 (8)	0.54129 (8)	0.0345 (3)	
O14	0.8863 (2)	−0.09825 (8)	0.89644 (8)	0.0358 (3)	
O2A	0.7817 (2)	0.29229 (9)	0.55070 (8)	0.0389 (3)	
O23	0.8907 (2)	0.33772 (11)	1.14515 (9)	0.0429 (4)	
N3	0.7030 (2)	0.84628 (9)	0.24398 (9)	0.0266 (3)	
N1	0.6211 (2)	1.00110 (9)	0.25813 (9)	0.0256 (3)	
N1A	0.7425 (2)	0.44369 (9)	0.54403 (9)	0.0278 (3)	
N7A	0.7829 (2)	0.58019 (10)	0.34929 (9)	0.0282 (3)	
N7	0.6080 (2)	0.87763 (9)	0.46015 (9)	0.0268 (3)	
N3A	0.8023 (2)	0.35747 (9)	0.42125 (9)	0.0284 (3)	
N9	0.7020 (2)	0.76324 (10)	0.37586 (9)	0.0288 (3)	
N9A	0.8267 (2)	0.44517 (10)	0.29076 (9)	0.0291 (3)	
C5	0.6194 (2)	0.91087 (11)	0.37915 (10)	0.0251 (4)	
C17	0.8717 (2)	0.08734 (11)	0.63331 (10)	0.0251 (4)	
C19	0.8101 (2)	0.10919 (11)	0.78164 (10)	0.0233 (3)	
C15	0.9085 (2)	−0.03750 (11)	0.72635 (10)	0.0253 (4)	
C18	0.8176 (2)	0.14448 (11)	0.70089 (10)	0.0245 (3)	
H18	0.786777	0.206206	0.691511	0.029*	
C20	0.8562 (2)	0.01776 (11)	0.79653 (10)	0.0236 (3)	
C16	0.9165 (2)	−0.00340 (11)	0.64546 (10)	0.0256 (4)	
H16	0.951874	−0.041242	0.598929	0.031*	
C5A	0.7733 (2)	0.51758 (11)	0.41383 (11)	0.0260 (4)	
C6	0.5894 (2)	0.99854 (11)	0.34590 (10)	0.0245 (4)	
C4	0.6768 (2)	0.83906 (11)	0.32953 (10)	0.0251 (4)	

C6A	0.7426 (2)	0.52726 (11)	0.50203 (11)	0.0275 (4)	
C2	0.6765 (2)	0.92830 (11)	0.20570 (10)	0.0264 (4)	
C4A	0.8016 (2)	0.43633 (11)	0.37568 (10)	0.0256 (4)	
C23	0.7573 (3)	0.29778 (12)	1.10345 (11)	0.0310 (4)	
C24	0.5550 (3)	0.30540 (11)	1.13014 (11)	0.0290 (4)	
C8A	0.8143 (3)	0.53374 (12)	0.27812 (11)	0.0293 (4)	
H8A	0.826537	0.561181	0.223988	0.035*	
C1	0.5956 (2)	1.08871 (10)	0.21617 (9)	0.0302 (4)	
H1AA	0.473023	1.091056	0.189923	0.045*	0.7
H1AB	0.707706	1.097257	0.173164	0.045*	0.7
H1AC	0.589165	1.136118	0.257515	0.045*	0.7
C14	0.8436 (3)	−0.01882 (11)	0.88098 (11)	0.0305 (4)	
C8	0.6576 (3)	0.79034 (11)	0.45496 (11)	0.0293 (4)	
H8	0.661120	0.751519	0.502424	0.035*	
C2A	0.7766 (3)	0.36003 (11)	0.50686 (11)	0.0287 (4)	
C1A	0.7085 (3)	0.44503 (13)	0.63626 (11)	0.0336 (4)	
H1AD	0.829512	0.461036	0.658313	0.050*	
H1AE	0.671777	0.385729	0.657892	0.050*	
H1AF	0.601631	0.489199	0.653892	0.050*	
C3	0.7564 (3)	0.76666 (12)	0.19374 (11)	0.0339 (4)	
H3A	0.887963	0.742963	0.204027	0.051*	
H3B	0.757052	0.782462	0.134196	0.051*	
H3C	0.659795	0.721201	0.209315	0.051*	
C25	0.4390 (7)	0.2618 (2)	1.08403 (19)	0.0323 (8)	0.7
H25	0.299887	0.267533	1.098180	0.039*	0.7
C22	0.8412 (6)	0.2493 (2)	1.0354 (2)	0.0253 (7)	0.7
H22	0.979429	0.248382	1.018574	0.030*	0.7
C21	0.7176 (6)	0.20199 (17)	0.99207 (15)	0.0232 (6)	0.7
C26	0.5150 (6)	0.2083 (2)	1.01605 (18)	0.0274 (7)	0.7
H26	0.428680	0.176692	0.986621	0.033*	0.7
C3A	0.8426 (3)	0.27088 (12)	0.38059 (13)	0.0415 (5)	
H3AA	0.837253	0.278577	0.320186	0.062*	
H3AB	0.743444	0.229209	0.403802	0.062*	
H3AC	0.974264	0.246923	0.390593	0.062*	
C27	0.3095 (3)	0.34459 (17)	1.24263 (15)	0.0493 (6)	
H27A	0.214797	0.375793	1.209424	0.074*	
H27B	0.298574	0.370846	1.298343	0.074*	
H27C	0.280904	0.281141	1.248231	0.074*	
C13A	0.7478 (5)	0.04197 (17)	0.94699 (15)	0.0275 (5)	0.7
H13A	0.788339	0.022750	1.002036	0.033*	0.7
H13B	0.602505	0.039487	0.950282	0.033*	0.7
C12A	0.8076 (4)	0.13636 (16)	0.92663 (14)	0.0253 (5)	0.7
H12A	0.954409	0.136849	0.924782	0.030*	0.7
C12	0.6896 (10)	0.1278 (4)	0.9286 (3)	0.0245 (10)*	0.3
H12	0.563661	0.098580	0.922896	0.029*	0.3
C13	0.8348 (11)	0.0545 (4)	0.9526 (4)	0.0268 (14)*	0.3
H13C	0.788907	0.028161	1.008158	0.032*	0.3
H13D	0.966668	0.078302	0.954747	0.032*	0.3

C22A	0.7886 (13)	0.2367 (6)	1.0325 (7)	0.027 (2)*	0.3
H22A	0.921345	0.222330	1.010238	0.032*	0.3
C21A	0.6411 (15)	0.1981 (4)	0.9950 (5)	0.0189 (17)*	0.3
C26A	0.4459 (13)	0.2165 (5)	1.0227 (5)	0.027 (2)*	0.3
H26A	0.346921	0.193501	0.993523	0.033*	0.3
C25A	0.3923 (2)	0.26714 (10)	1.09113 (9)	0.023 (2)*	0.3
H25A	0.257821	0.277615	1.113001	0.028*	0.3
H1BD	0.463355	1.109293	0.240891	0.045*	0.3
H1BE	0.589025	1.079383	0.156461	0.045*	0.3
H1BF	0.669775	1.131223	0.243841	0.045*	0.3
H23	0.830785	0.364614	1.190901	0.048 (7)*	
H7A	0.763785	0.646454	0.356071	0.059 (8)*	
H15	0.935805	−0.134196	0.791331	0.049 (7)*	
H7	0.566795	0.905904	0.507971	0.037 (6)*	
H17	0.853365	0.177414	0.551611	0.059 (8)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0378 (6)	0.0242 (6)	0.0178 (6)	0.0011 (5)	−0.0006 (5)	−0.0024 (4)
O6	0.0377 (7)	0.0260 (6)	0.0242 (6)	−0.0009 (5)	−0.0019 (5)	−0.0021 (5)
O17	0.0417 (7)	0.0295 (6)	0.0189 (6)	0.0013 (5)	−0.0012 (5)	−0.0015 (4)
O15	0.0417 (7)	0.0220 (6)	0.0286 (7)	0.0010 (5)	−0.0054 (5)	−0.0039 (5)
O24	0.0312 (6)	0.0368 (7)	0.0316 (7)	−0.0039 (5)	0.0016 (5)	−0.0162 (5)
O2	0.0396 (7)	0.0330 (6)	0.0216 (6)	−0.0048 (5)	0.0007 (5)	0.0000 (5)
O6A	0.0516 (8)	0.0260 (6)	0.0260 (6)	0.0003 (5)	−0.0044 (5)	−0.0061 (5)
O14	0.0513 (8)	0.0260 (6)	0.0286 (7)	0.0029 (5)	−0.0007 (6)	0.0033 (5)
O2A	0.0551 (8)	0.0271 (7)	0.0330 (7)	0.0002 (6)	0.0001 (6)	0.0027 (5)
O23	0.0331 (7)	0.0639 (10)	0.0313 (7)	−0.0027 (6)	0.0018 (5)	−0.0179 (6)
N3	0.0305 (7)	0.0275 (7)	0.0215 (7)	−0.0017 (5)	−0.0007 (5)	−0.0021 (5)
N1	0.0279 (7)	0.0263 (7)	0.0225 (7)	−0.0036 (5)	−0.0022 (5)	0.0009 (5)
N1A	0.0328 (7)	0.0266 (7)	0.0236 (7)	−0.0002 (5)	−0.0023 (6)	−0.0024 (5)
N7A	0.0330 (7)	0.0266 (7)	0.0247 (7)	−0.0007 (5)	−0.0024 (6)	−0.0018 (5)
N7	0.0335 (7)	0.0264 (7)	0.0199 (7)	−0.0020 (5)	−0.0011 (5)	0.0000 (5)
N3A	0.0346 (7)	0.0221 (7)	0.0278 (7)	−0.0012 (5)	−0.0007 (6)	−0.0035 (5)
N9	0.0335 (7)	0.0267 (7)	0.0257 (7)	−0.0024 (6)	−0.0010 (6)	−0.0001 (5)
N9A	0.0329 (7)	0.0306 (7)	0.0239 (7)	−0.0034 (6)	−0.0028 (6)	−0.0038 (5)
C5	0.0269 (8)	0.0282 (8)	0.0202 (8)	−0.0038 (6)	−0.0021 (6)	−0.0001 (6)
C17	0.0258 (7)	0.0284 (8)	0.0214 (8)	−0.0040 (6)	−0.0021 (6)	−0.0013 (6)
C19	0.0235 (7)	0.0245 (8)	0.0219 (8)	−0.0035 (6)	−0.0012 (6)	−0.0039 (6)
C15	0.0254 (7)	0.0239 (8)	0.0269 (8)	−0.0022 (6)	−0.0040 (6)	−0.0033 (6)
C18	0.0273 (8)	0.0234 (7)	0.0225 (8)	−0.0025 (6)	−0.0008 (6)	−0.0018 (6)
C20	0.0255 (7)	0.0244 (8)	0.0211 (8)	−0.0028 (6)	−0.0023 (6)	−0.0020 (6)
C16	0.0281 (8)	0.0261 (8)	0.0226 (8)	−0.0018 (6)	−0.0020 (6)	−0.0072 (6)
C5A	0.0273 (8)	0.0245 (8)	0.0262 (8)	−0.0023 (6)	−0.0026 (6)	−0.0024 (6)
C6	0.0243 (7)	0.0266 (8)	0.0226 (8)	−0.0036 (6)	−0.0018 (6)	−0.0005 (6)
C4	0.0242 (7)	0.0283 (8)	0.0228 (8)	−0.0036 (6)	−0.0019 (6)	−0.0015 (6)
C6A	0.0296 (8)	0.0276 (8)	0.0255 (8)	−0.0009 (6)	−0.0043 (6)	−0.0029 (6)

C2	0.0256 (8)	0.0308 (8)	0.0226 (8)	−0.0049 (6)	0.0000 (6)	−0.0015 (6)
C4A	0.0254 (8)	0.0269 (8)	0.0243 (8)	−0.0008 (6)	−0.0012 (6)	−0.0046 (6)
C23	0.0403 (9)	0.0304 (9)	0.0214 (8)	0.0017 (7)	−0.0011 (7)	−0.0029 (6)
C24	0.0393 (9)	0.0250 (8)	0.0227 (8)	−0.0002 (7)	−0.0037 (7)	−0.0061 (6)
C8A	0.0325 (8)	0.0322 (9)	0.0232 (8)	−0.0012 (7)	−0.0031 (6)	−0.0015 (6)
C1	0.0368 (9)	0.0285 (8)	0.0251 (8)	−0.0030 (7)	−0.0031 (7)	0.0034 (6)
C14	0.0379 (9)	0.0257 (8)	0.0265 (9)	0.0006 (7)	0.0011 (7)	0.0011 (6)
C8	0.0366 (9)	0.0271 (8)	0.0238 (8)	−0.0018 (7)	−0.0023 (7)	0.0003 (6)
C2A	0.0300 (8)	0.0272 (8)	0.0287 (9)	−0.0017 (6)	−0.0024 (6)	−0.0009 (6)
C1A	0.0397 (10)	0.0360 (9)	0.0245 (9)	0.0019 (7)	−0.0031 (7)	−0.0009 (7)
C3	0.0476 (10)	0.0288 (9)	0.0244 (8)	−0.0024 (7)	0.0001 (7)	−0.0044 (7)
C25	0.0261 (14)	0.0400 (18)	0.0316 (16)	−0.0036 (11)	−0.0039 (11)	−0.0068 (11)
C22	0.0296 (16)	0.0272 (15)	0.0194 (14)	−0.0016 (13)	−0.0036 (13)	−0.0051 (10)
C21	0.0263 (15)	0.0247 (13)	0.0187 (13)	−0.0030 (11)	−0.0021 (11)	−0.0023 (9)
C26	0.030 (2)	0.0301 (15)	0.0235 (14)	−0.0025 (13)	−0.0078 (14)	−0.0072 (10)
C3A	0.0623 (13)	0.0251 (9)	0.0353 (10)	−0.0018 (8)	0.0026 (9)	−0.0065 (7)
C27	0.0363 (10)	0.0595 (14)	0.0506 (13)	−0.0097 (9)	0.0094 (9)	−0.0263 (10)
C13A	0.0340 (14)	0.0288 (13)	0.0189 (11)	−0.0021 (11)	−0.0001 (10)	0.0001 (9)
C12A	0.0306 (13)	0.0281 (12)	0.0174 (11)	−0.0009 (9)	−0.0040 (9)	−0.0020 (8)

Geometric parameters (Å, º)

O11—C19	1.3691 (19)	C5A—C4A	1.370 (2)
O11—C12A	1.441 (2)	C23—C24	1.397 (3)
O11—C12	1.469 (6)	C23—C22	1.378 (4)
O6—C6	1.227 (2)	C23—C22A	1.468 (11)
O17—C17	1.3498 (19)	C24—C25	1.349 (3)
O17—H17	0.9121	C24—C25A	1.485 (2)
O15—C15	1.353 (2)	C8A—H8A	0.9500
O15—H15	0.8570	C1—H1AA	0.9800
O24—C24	1.370 (2)	C1—H1AB	0.9800
O24—C27	1.426 (2)	C1—H1AC	0.9800
O2—C2	1.225 (2)	C1—H1BD	0.9800
O6A—C6A	1.228 (2)	C1—H1BE	0.9800
O14—C14	1.237 (2)	C1—H1BF	0.9800
O2A—C2A	1.223 (2)	C14—C13A	1.484 (3)
O23—C23	1.363 (2)	C14—C13	1.604 (7)
O23—H23	0.8923	C8—H8	0.9500
N3—C4	1.368 (2)	C1A—H1AD	0.9800
N3—C2	1.374 (2)	C1A—H1AE	0.9800
N3—C3	1.462 (2)	C1A—H1AF	0.9800
N1—C6	1.402 (2)	C3—H3A	0.9800
N1—C2	1.403 (2)	C3—H3B	0.9800
N1—C1	1.4728 (19)	C3—H3C	0.9800
N1A—C6A	1.408 (2)	C25—H25	0.9500
N1A—C2A	1.398 (2)	C25—C26	1.403 (5)
N1A—C1A	1.474 (2)	C22—H22	0.9500
N7A—C5A	1.380 (2)	C22—C21	1.387 (5)

N7A—C8A	1.343 (2)	C21—C26	1.390 (5)
N7A—H7A	1.0013	C21—C12A	1.511 (3)
N7—C5	1.378 (2)	C26—H26	0.9500
N7—C8	1.336 (2)	C3A—H3AA	0.9800
N7—H7	0.8952	C3A—H3AB	0.9800
N3A—C4A	1.375 (2)	C3A—H3AC	0.9800
N3A—C2A	1.368 (2)	C27—H27A	0.9800
N3A—C3A	1.465 (2)	C27—H27B	0.9800
N9—C4	1.358 (2)	C27—H27C	0.9800
N9—C8	1.339 (2)	C13A—H13A	0.9900
N9A—C4A	1.360 (2)	C13A—H13B	0.9900
N9A—C8A	1.337 (2)	C13A—C12A	1.510 (3)
C5—C6	1.420 (2)	C12A—H12A	1.0000
C5—C4	1.368 (2)	C12—H12	1.0000
C17—C18	1.402 (2)	C12—C13	1.515 (8)
C17—C16	1.392 (2)	C12—C21A	1.514 (9)
C19—C18	1.386 (2)	C13—H13C	0.9900
C19—C20	1.410 (2)	C13—H13D	0.9900
C15—C20	1.416 (2)	C22A—H22A	0.9500
C15—C16	1.382 (2)	C22A—C21A	1.391 (11)
C18—H18	0.9500	C21A—C26A	1.370 (10)
C20—C14	1.447 (2)	C26A—H26A	0.9500
C16—H16	0.9500	C26A—C25A	1.355 (9)
C5A—C6A	1.418 (2)	C25A—H25A	0.9500

C19—O11—C12A	115.28 (14)	H1BD—C1—H1BE	107.3
C19—O11—C12	117.3 (2)	H1BD—C1—H1BF	97.2
C17—O17—H17	110.0	H1BE—C1—H1BF	130.2
C15—O15—H15	105.1	O14—C14—C20	122.60 (16)
C24—O24—C27	116.49 (15)	O14—C14—C13A	121.67 (17)
C23—O23—H23	110.8	O14—C14—C13	120.5 (3)
C4—N3—C2	119.71 (14)	C20—C14—C13A	115.14 (16)
C4—N3—C3	119.94 (14)	C20—C14—C13	114.5 (3)
C2—N3—C3	120.35 (14)	N7—C8—N9	112.98 (15)
C6—N1—C2	126.48 (14)	N7—C8—H8	123.5
C6—N1—C1	117.25 (13)	N9—C8—H8	123.5
C2—N1—C1	116.27 (13)	O2A—C2A—N1A	119.99 (16)
C6A—N1A—C1A	116.58 (14)	O2A—C2A—N3A	122.24 (16)
C2A—N1A—C6A	126.34 (14)	N3A—C2A—N1A	117.77 (15)
C2A—N1A—C1A	117.05 (14)	N1A—C1A—H1AD	109.5
C5A—N7A—H7A	125.4	N1A—C1A—H1AE	109.5
C8A—N7A—C5A	106.08 (14)	N1A—C1A—H1AF	109.5
C8A—N7A—H7A	128.4	H1AD—C1A—H1AE	109.5
C5—N7—H7	128.7	H1AD—C1A—H1AF	109.5
C8—N7—C5	106.60 (14)	H1AE—C1A—H1AF	109.5
C8—N7—H7	124.5	N3—C3—H3A	109.5
C4A—N3A—C3A	121.64 (15)	N3—C3—H3B	109.5
C2A—N3A—C4A	119.28 (14)	N3—C3—H3C	109.5

C2A—N3A—C3A	118.95 (15)	H3A—C3—H3B	109.5
C8—N9—C4	103.59 (14)	H3A—C3—H3C	109.5
C8A—N9A—C4A	103.20 (14)	H3B—C3—H3C	109.5
N7—C5—C6	132.16 (15)	C24—C25—H25	118.6
C4—C5—N7	105.25 (14)	C24—C25—C26	122.8 (4)
C4—C5—C6	122.56 (15)	C26—C25—H25	118.6
O17—C17—C18	121.80 (15)	C23—C22—H22	121.0
O17—C17—C16	116.51 (15)	C23—C22—C21	118.0 (3)
C16—C17—C18	121.69 (15)	C21—C22—H22	121.0
O11—C19—C18	117.07 (14)	C22—C21—C26	119.4 (3)
O11—C19—C20	121.65 (14)	C22—C21—C12A	119.1 (3)
C18—C19—C20	121.28 (15)	C26—C21—C12A	121.1 (3)
O15—C15—C20	119.74 (15)	C25—C26—H26	120.2
O15—C15—C16	118.82 (15)	C21—C26—C25	119.6 (3)
C16—C15—C20	121.45 (15)	C21—C26—H26	120.2
C17—C18—H18	120.6	N3A—C3A—H3AA	109.5
C19—C18—C17	118.72 (15)	N3A—C3A—H3AB	109.5
C19—C18—H18	120.6	N3A—C3A—H3AC	109.5
C19—C20—C15	118.00 (14)	H3AA—C3A—H3AB	109.5
C19—C20—C14	120.84 (15)	H3AA—C3A—H3AC	109.5
C15—C20—C14	121.12 (15)	H3AB—C3A—H3AC	109.5
C17—C16—H16	120.6	O24—C27—H27A	109.5
C15—C16—C17	118.87 (15)	O24—C27—H27B	109.5
C15—C16—H16	120.6	O24—C27—H27C	109.5
N7A—C5A—C6A	131.38 (15)	H27A—C27—H27B	109.5
C4A—C5A—N7A	105.30 (15)	H27A—C27—H27C	109.5
C4A—C5A—C6A	123.32 (16)	H27B—C27—H27C	109.5
O6—C6—N1	120.72 (15)	C14—C13A—H13A	109.8
O6—C6—C5	127.15 (15)	C14—C13A—H13B	109.8
N1—C6—C5	112.13 (14)	C14—C13A—C12A	109.56 (19)
N9—C4—N3	126.27 (15)	H13A—C13A—H13B	108.2
N9—C4—C5	111.58 (14)	C12A—C13A—H13A	109.8
C5—C4—N3	122.15 (15)	C12A—C13A—H13B	109.8
O6A—C6A—N1A	120.88 (15)	O11—C12A—C21	108.71 (19)
O6A—C6A—C5A	127.60 (16)	O11—C12A—C13A	112.03 (19)
N1A—C6A—C5A	111.52 (14)	O11—C12A—H12A	107.8
O2—C2—N3	121.90 (15)	C21—C12A—H12A	107.8
O2—C2—N1	121.16 (15)	C13A—C12A—C21	112.5 (2)
N3—C2—N1	116.94 (14)	C13A—C12A—H12A	107.8
N9A—C4A—N3A	126.45 (15)	O11—C12—H12	106.6
N9A—C4A—C5A	111.87 (15)	O11—C12—C13	111.0 (5)
C5A—C4A—N3A	121.68 (15)	O11—C12—C21A	112.3 (4)
O23—C23—C24	121.53 (15)	C13—C12—H12	106.6
O23—C23—C22	114.0 (2)	C21A—C12—H12	106.6
O23—C23—C22A	130.2 (4)	C21A—C12—C13	113.3 (5)
C24—C23—C22A	108.1 (4)	C14—C13—H13C	110.7
C22—C23—C24	124.5 (2)	C14—C13—H13D	110.7
O24—C24—C23	114.70 (15)	C12—C13—C14	105.4 (5)

O24—C24—C25A	117.41 (15)	C12—C13—H13C	110.7
C23—C24—C25A	127.88 (15)	C12—C13—H13D	110.7
C25—C24—O24	129.6 (3)	H13C—C13—H13D	108.8
C25—C24—C23	115.7 (3)	C23—C22A—H22A	117.1
N7A—C8A—H8A	123.2	C21A—C22A—C23	125.8 (7)
N9A—C8A—N7A	113.55 (15)	C21A—C22A—H22A	117.1
N9A—C8A—H8A	123.2	C22A—C21A—C12	121.4 (9)
N1—C1—H1AA	109.5	C26A—C21A—C12	117.6 (7)
N1—C1—H1AB	109.5	C26A—C21A—C22A	120.7 (8)
N1—C1—H1AC	109.5	C21A—C26A—H26A	119.6
N1—C1—H1BD	102.0	C25A—C26A—C21A	120.8 (7)
N1—C1—H1BE	108.8	C25A—C26A—H26A	119.6
N1—C1—H1BF	107.3	C24—C25A—H25A	121.8
H1AA—C1—H1AB	109.5	C26A—C25A—C24	116.4 (4)
H1AA—C1—H1AC	109.5	C26A—C25A—H25A	121.8
H1AB—C1—H1AC	109.5		

O11—C19—C18—C17	−179.82 (14)	C4A—N3A—C2A—O2A	−177.77 (16)
O11—C19—C20—C15	179.32 (14)	C4A—N3A—C2A—N1A	2.6 (2)
O11—C19—C20—C14	1.5 (2)	C4A—N9A—C8A—N7A	−0.1 (2)
O11—C12—C13—C14	−60.0 (6)	C4A—C5A—C6A—O6A	179.37 (17)
O11—C12—C21A—C22A	−78.9 (8)	C4A—C5A—C6A—N1A	−0.8 (2)
O11—C12—C21A—C26A	107.0 (7)	C23—C24—C25—C26	3.1 (4)
O17—C17—C18—C19	−179.73 (15)	C23—C24—C25A—C26A	2.0 (5)
O17—C17—C16—C15	179.63 (14)	C23—C22—C21—C26	3.0 (4)
O15—C15—C20—C19	−178.57 (14)	C23—C22—C21—C12A	−169.9 (2)
O15—C15—C20—C14	−0.7 (2)	C23—C22A—C21A—C12	−173.0 (6)
O15—C15—C16—C17	179.17 (15)	C23—C22A—C21A—C26A	1.0 (13)
O24—C24—C25—C26	−173.8 (2)	C24—C23—C22—C21	−2.6 (4)
O24—C24—C25A—C26A	−177.0 (4)	C24—C23—C22A—C21A	3.9 (9)
O14—C14—C13A—C12A	150.4 (2)	C24—C25—C26—C21	−2.7 (4)
O14—C14—C13—C12	−152.9 (4)	C8A—N7A—C5A—C6A	−179.85 (18)
O23—C23—C24—O24	−1.8 (3)	C8A—N7A—C5A—C4A	0.37 (18)
O23—C23—C24—C25	−179.2 (2)	C8A—N9A—C4A—N3A	−179.87 (16)
O23—C23—C24—C25A	179.13 (16)	C8A—N9A—C4A—C5A	0.37 (19)
O23—C23—C22—C21	176.3 (2)	C1—N1—C6—O6	0.0 (2)
O23—C23—C22A—C21A	178.8 (6)	C1—N1—C6—C5	−179.65 (13)
N7A—C5A—C6A—O6A	−0.4 (3)	C1—N1—C2—O2	−0.3 (2)
N7A—C5A—C6A—N1A	179.44 (16)	C1—N1—C2—N3	179.29 (13)
N7A—C5A—C4A—N3A	179.75 (15)	C14—C13A—C12A—O11	58.0 (3)
N7A—C5A—C4A—N9A	−0.47 (19)	C14—C13A—C12A—C21	−179.1 (2)
N7—C5—C6—O6	−0.7 (3)	C8—N7—C5—C6	−177.87 (18)
N7—C5—C6—N1	178.88 (16)	C8—N7—C5—C4	−0.01 (18)
N7—C5—C4—N3	179.65 (15)	C8—N9—C4—N3	−179.52 (16)
N7—C5—C4—N9	−0.19 (19)	C8—N9—C4—C5	0.31 (19)
C5—N7—C8—N9	0.2 (2)	C2A—N1A—C6A—O6A	−177.34 (16)
C19—O11—C12A—C21	−173.4 (2)	C2A—N1A—C6A—C5A	2.8 (2)
C19—O11—C12A—C13A	−48.4 (2)	C2A—N3A—C4A—N9A	179.39 (16)

C19—O11—C12—C13	50.0 (6)	C2A—N3A—C4A—C5A	−0.9 (2)
C19—O11—C12—C21A	178.0 (5)	C1A—N1A—C6A—O6A	0.8 (2)
C19—C20—C14—O14	−178.71 (17)	C1A—N1A—C6A—C5A	−179.02 (15)
C19—C20—C14—C13A	10.0 (3)	C1A—N1A—C2A—O2A	−1.6 (2)
C19—C20—C14—C13	−16.3 (4)	C1A—N1A—C2A—N3A	178.01 (15)
C15—C20—C14—O14	3.5 (3)	C3—N3—C4—N9	2.4 (3)
C15—C20—C14—C13A	−167.75 (19)	C3—N3—C4—C5	−177.39 (16)
C15—C20—C14—C13	165.9 (3)	C3—N3—C2—O2	−1.8 (2)
C18—C17—C16—C15	−0.5 (2)	C3—N3—C2—N1	178.61 (15)
C18—C19—C20—C15	−0.8 (2)	C22—C23—C24—O24	176.9 (2)
C18—C19—C20—C14	−178.62 (15)	C22—C23—C24—C25	−0.4 (3)
C20—C19—C18—C17	0.3 (2)	C22—C21—C26—C25	−0.6 (4)
C20—C15—C16—C17	−0.1 (2)	C22—C21—C12A—O11	−116.1 (3)
C20—C14—C13A—C12A	−38.2 (3)	C22—C21—C12A—C13A	119.2 (3)
C20—C14—C13—C12	44.3 (5)	C26—C21—C12A—O11	71.1 (3)
C16—C17—C18—C19	0.4 (2)	C26—C21—C12A—C13A	−53.5 (3)
C16—C15—C20—C19	0.7 (2)	C3A—N3A—C4A—N9A	3.5 (3)
C16—C15—C20—C14	178.52 (16)	C3A—N3A—C4A—C5A	−176.79 (17)
C5A—N7A—C8A—N9A	−0.2 (2)	C3A—N3A—C2A—O2A	−1.7 (3)
C6—N1—C2—O2	−179.83 (15)	C3A—N3A—C2A—N1A	178.65 (16)
C6—N1—C2—N3	−0.3 (2)	C27—O24—C24—C23	−165.02 (18)
C6—C5—C4—N3	−2.2 (3)	C27—O24—C24—C25	11.9 (3)
C6—C5—C4—N9	177.92 (15)	C27—O24—C24—C25A	14.1 (2)
C4—N3—C2—O2	179.02 (15)	C12A—O11—C19—C18	−161.55 (16)
C4—N3—C2—N1	−0.5 (2)	C12A—O11—C19—C20	18.4 (2)
C4—N9—C8—N7	−0.3 (2)	C12A—C21—C26—C25	172.2 (2)
C4—C5—C6—O6	−178.29 (16)	C12—O11—C19—C18	161.9 (3)
C4—C5—C6—N1	1.3 (2)	C12—O11—C19—C20	−18.1 (3)
C6A—N1A—C2A—O2A	176.53 (17)	C12—C21A—C26A—C25A	169.0 (5)
C6A—N1A—C2A—N3A	−3.8 (3)	C13—C12—C21A—C22A	47.8 (9)
C6A—C5A—C4A—N3A	0.0 (3)	C13—C12—C21A—C26A	−126.3 (7)
C6A—C5A—C4A—N9A	179.72 (15)	C22A—C23—C24—O24	173.6 (4)
C2—N3—C4—N9	−178.41 (15)	C22A—C23—C24—C25A	−5.4 (5)
C2—N3—C4—C5	1.8 (2)	C22A—C21A—C26A—C25A	−5.1 (12)
C2—N1—C6—O6	179.53 (15)	C21A—C12—C13—C14	172.6 (5)
C2—N1—C6—C5	−0.1 (2)	C21A—C26A—C25A—C24	3.7 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O23—H23···O24	0.89	2.23	2.6841 (19)	111
N7*A*—H7*A*···N9	1.00	1.80	2.801 (2)	173
O15—H15···O14	0.86	1.77	2.5712 (18)	154
N7—H7···O6^{i}	0.90	1.83	2.7045 (18)	164
O17—H17···O2*A*	0.91	1.76	2.6756 (18)	177

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.

8-Chloro-1,3-dimethyl-7*H*-purine-2,6-dione–3,5,7-trihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one (2a-ywj65cult100k_auto)

Crystal data

$C_7H_7ClN_4O_2 \cdot C_{15}H_{10}O_6$
$M_r = 500.85$
Monoclinic, $P2_1/n$
$a = 10.14353\ (15)$ Å
$b = 19.7196\ (3)$ Å
$c = 10.42202\ (19)$ Å
$\beta = 93.0362\ (14)°$
$V = 2081.75\ (6)$ Å^3
$Z = 4$
$F(000) = 1032$
$D_x = 1.598$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 5518 reflections
$\theta = 4.2–74.7°$
$\mu = 2.18$ mm^{-1}
$T = 100$ K
Needle, colourless
0.2 × 0.08 × 0.04 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3577 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)
$T_{min} = 0.705$, $T_{max} = 1.000$
12088 measured reflections
4177 independent reflections
3533 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 74.9°$, $\theta_{min} = 4.5°$
$h = -12\rightarrow8$
$k = -24\rightarrow23$
$l = -12\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.100$
$S = 1.03$
4177 reflections
338 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.5825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl8	0.77141 (4)	0.33918 (2)	0.42698 (4)	0.02520 (12)
O11	1.00921 (10)	−0.05831 (6)	0.31784 (10)	0.0205 (2)
O13	1.17840 (13)	0.10283 (7)	0.29520 (13)	0.0316 (3)
O6	0.31031 (10)	0.48646 (6)	0.53932 (11)	0.0217 (2)
O24	0.56534 (13)	0.14433 (7)	0.44625 (13)	0.0314 (3)
O17	1.14151 (12)	−0.28596 (6)	0.27053 (12)	0.0264 (3)
O14	1.37244 (12)	0.01475 (7)	0.24898 (12)	0.0311 (3)
N7	0.55291 (12)	0.39977 (7)	0.49178 (12)	0.0185 (3)

O15	1.45391 (11)	−0.11163 (7)	0.21924 (12)	0.0297 (3)
C8	0.61478 (15)	0.34083 (8)	0.47805 (15)	0.0196 (3)
O2	0.10117 (11)	0.28677 (6)	0.61856 (12)	0.0262 (3)
N9	0.54381 (12)	0.28594 (7)	0.50348 (12)	0.0192 (3)
N3	0.31595 (12)	0.27929 (7)	0.56928 (13)	0.0196 (3)
N1	0.20696 (12)	0.38574 (7)	0.57840 (12)	0.0187 (3)
C25	0.68197 (16)	0.04410 (9)	0.40628 (16)	0.0243 (3)
H25	0.604243	0.018858	0.420177	0.029*
C6	0.31701 (14)	0.42419 (8)	0.54912 (14)	0.0179 (3)
C12	1.03122 (16)	0.01037 (8)	0.32052 (15)	0.0212 (3)
C26	0.79599 (16)	0.01107 (8)	0.37480 (15)	0.0224 (3)
H26	0.795723	−0.036928	0.366966	0.027*
C22	0.90991 (17)	0.11794 (8)	0.36761 (16)	0.0242 (3)
H22	0.987671	0.143470	0.355380	0.029*
C20	1.23312 (15)	−0.08105 (8)	0.26736 (15)	0.0214 (3)
C1	0.08172 (15)	0.42128 (8)	0.59713 (16)	0.0219 (3)
H1A	0.016094	0.407782	0.529552	0.033*
H1B	0.096038	0.470367	0.592991	0.033*
H1C	0.049715	0.409354	0.681292	0.033*
C17	1.16810 (15)	−0.21858 (9)	0.26986 (15)	0.0217 (3)
C13	1.15267 (16)	0.03498 (8)	0.29476 (16)	0.0238 (3)
C3	0.31327 (17)	0.20483 (8)	0.57409 (17)	0.0259 (4)
H3A	0.301202	0.186758	0.486670	0.039*
H3B	0.240057	0.189964	0.624975	0.039*
H3C	0.396766	0.188121	0.613847	0.039*
C14	1.26073 (16)	−0.00991 (9)	0.26826 (15)	0.0239 (3)
C18	1.07171 (15)	−0.17089 (8)	0.29452 (15)	0.0203 (3)
H18	0.984610	−0.184464	0.312005	0.024*
C2	0.20300 (15)	0.31553 (8)	0.59072 (15)	0.0197 (3)
C15	1.32883 (15)	−0.13117 (9)	0.24321 (15)	0.0235 (3)
C5	0.42901 (15)	0.38299 (8)	0.52990 (14)	0.0181 (3)
C21	0.91253 (15)	0.04733 (8)	0.35414 (14)	0.0212 (3)
C23	0.79546 (17)	0.15107 (9)	0.39853 (16)	0.0259 (4)
H23	0.795261	0.199051	0.406697	0.031*
C24	0.68125 (17)	0.11472 (9)	0.41764 (15)	0.0244 (3)
C4	0.42668 (15)	0.31383 (8)	0.53601 (14)	0.0182 (3)
C16	1.29724 (16)	−0.19896 (9)	0.24328 (15)	0.0242 (3)
H16	1.361730	−0.232129	0.225609	0.029*
C19	1.10638 (15)	−0.10333 (8)	0.29281 (14)	0.0198 (3)
H7	0.590 (2)	0.4409 (11)	0.4832 (19)	0.030 (5)*
H24	0.578 (2)	0.1845 (13)	0.457 (2)	0.041 (7)*
H17	1.065 (3)	−0.2906 (13)	0.297 (2)	0.041 (6)*
H15	1.450 (3)	−0.0663 (15)	0.221 (3)	0.057 (8)*
H13	1.260 (3)	0.1042 (15)	0.278 (3)	0.064 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl8	0.01687 (18)	0.0218 (2)	0.0373 (2)	0.00008 (13)	0.00453 (15)	−0.00172 (15)
O11	0.0187 (5)	0.0176 (5)	0.0252 (6)	−0.0011 (4)	0.0014 (4)	0.0008 (4)
O13	0.0300 (7)	0.0246 (7)	0.0405 (7)	−0.0112 (5)	0.0054 (5)	−0.0007 (5)
O6	0.0200 (5)	0.0159 (5)	0.0293 (6)	−0.0001 (4)	0.0023 (4)	0.0004 (4)
O24	0.0309 (6)	0.0231 (7)	0.0403 (7)	0.0062 (5)	0.0022 (5)	−0.0063 (6)
O17	0.0225 (6)	0.0216 (6)	0.0355 (7)	0.0026 (5)	0.0047 (5)	0.0009 (5)
O14	0.0229 (6)	0.0375 (7)	0.0332 (7)	−0.0111 (5)	0.0035 (5)	0.0012 (5)
N7	0.0164 (6)	0.0172 (6)	0.0218 (6)	−0.0010 (5)	0.0005 (5)	−0.0009 (5)
O15	0.0156 (5)	0.0399 (8)	0.0337 (7)	−0.0028 (5)	0.0032 (5)	0.0034 (6)
C8	0.0165 (7)	0.0208 (8)	0.0213 (7)	0.0003 (6)	−0.0002 (6)	−0.0014 (6)
O2	0.0212 (5)	0.0207 (6)	0.0374 (7)	−0.0021 (4)	0.0083 (5)	−0.0004 (5)
N9	0.0176 (6)	0.0194 (7)	0.0207 (6)	0.0011 (5)	0.0004 (5)	−0.0002 (5)
N3	0.0185 (6)	0.0156 (7)	0.0249 (7)	−0.0010 (5)	0.0031 (5)	0.0005 (5)
N1	0.0176 (6)	0.0167 (7)	0.0218 (6)	−0.0001 (5)	0.0018 (5)	−0.0014 (5)
C25	0.0242 (8)	0.0241 (9)	0.0245 (8)	−0.0001 (6)	0.0000 (6)	−0.0001 (6)
C6	0.0182 (7)	0.0185 (8)	0.0168 (7)	−0.0004 (6)	−0.0004 (5)	−0.0015 (5)
C12	0.0242 (7)	0.0199 (8)	0.0191 (7)	−0.0038 (6)	−0.0017 (6)	0.0015 (6)
C26	0.0260 (8)	0.0170 (8)	0.0240 (8)	−0.0008 (6)	−0.0002 (6)	−0.0010 (6)
C22	0.0289 (8)	0.0206 (8)	0.0226 (8)	−0.0031 (6)	−0.0023 (6)	0.0007 (6)
C20	0.0177 (7)	0.0283 (9)	0.0179 (7)	−0.0034 (6)	−0.0020 (6)	0.0023 (6)
C1	0.0160 (7)	0.0213 (8)	0.0284 (8)	0.0015 (6)	0.0020 (6)	−0.0006 (6)
C17	0.0206 (7)	0.0258 (8)	0.0186 (7)	0.0010 (6)	−0.0009 (6)	0.0016 (6)
C13	0.0259 (8)	0.0215 (8)	0.0235 (8)	−0.0063 (6)	−0.0015 (6)	0.0004 (6)
C3	0.0270 (8)	0.0147 (8)	0.0368 (9)	0.0001 (6)	0.0073 (7)	0.0014 (6)
C14	0.0208 (7)	0.0313 (9)	0.0194 (7)	−0.0075 (6)	−0.0015 (6)	0.0031 (6)
C18	0.0171 (7)	0.0241 (8)	0.0197 (7)	−0.0013 (6)	0.0005 (6)	0.0020 (6)
C2	0.0196 (7)	0.0186 (8)	0.0212 (7)	−0.0011 (6)	0.0024 (6)	−0.0003 (6)
C15	0.0155 (7)	0.0357 (9)	0.0192 (7)	−0.0028 (6)	−0.0006 (6)	0.0032 (7)
C5	0.0173 (7)	0.0184 (8)	0.0184 (7)	−0.0010 (6)	0.0003 (6)	−0.0007 (5)
C21	0.0245 (8)	0.0219 (8)	0.0167 (7)	0.0000 (6)	−0.0018 (6)	0.0004 (6)
C23	0.0352 (9)	0.0188 (8)	0.0232 (8)	0.0019 (7)	−0.0028 (7)	−0.0018 (6)
C24	0.0289 (8)	0.0239 (9)	0.0199 (8)	0.0063 (6)	−0.0025 (6)	−0.0023 (6)
C4	0.0182 (7)	0.0191 (7)	0.0171 (7)	−0.0002 (6)	−0.0007 (5)	−0.0010 (6)
C16	0.0183 (7)	0.0317 (9)	0.0225 (8)	0.0046 (6)	−0.0001 (6)	0.0018 (6)
C19	0.0174 (7)	0.0243 (8)	0.0174 (7)	0.0003 (6)	−0.0016 (5)	0.0014 (6)

Geometric parameters (Å, º)

Cl8—C8	1.7024 (16)	C6—C5	1.420 (2)
O11—C12	1.373 (2)	C12—C13	1.364 (2)
O11—C19	1.3620 (19)	C12—C21	1.466 (2)
O13—C13	1.363 (2)	C26—H26	0.9500
O13—H13	0.85 (3)	C26—C21	1.408 (2)
O6—C6	1.2337 (19)	C22—H22	0.9500
O24—C24	1.360 (2)	C22—C21	1.400 (2)

O24—H24	0.81 (3)	C22—C23	1.385 (2)
O17—C17	1.356 (2)	C20—C14	1.431 (2)
O17—H17	0.84 (3)	C20—C15	1.417 (2)
O14—C14	1.259 (2)	C20—C19	1.397 (2)
N7—C8	1.332 (2)	C1—H1A	0.9800
N7—C5	1.3784 (19)	C1—H1B	0.9800
N7—H7	0.90 (2)	C1—H1C	0.9800
O15—C15	1.3617 (19)	C17—C18	1.390 (2)
O15—H15	0.89 (3)	C17—C16	1.407 (2)
C8—N9	1.334 (2)	C13—C14	1.447 (2)
O2—C2	1.2266 (19)	C3—H3A	0.9800
N9—C4	1.368 (2)	C3—H3B	0.9800
N3—C3	1.469 (2)	C3—H3C	0.9800
N3—C2	1.379 (2)	C18—H18	0.9500
N3—C4	1.374 (2)	C18—C19	1.378 (2)
N1—C6	1.3965 (19)	C15—C16	1.375 (3)
N1—C1	1.4729 (19)	C5—C4	1.366 (2)
N1—C2	1.391 (2)	C23—H23	0.9500
C25—H25	0.9500	C23—C24	1.386 (3)
C25—C26	1.382 (2)	C16—H16	0.9500
C25—C24	1.398 (2)		

C19—O11—C12	121.90 (12)	C18—C17—C16	121.43 (15)
C13—O13—H13	103 (2)	O13—C13—C12	121.55 (16)
C24—O24—H24	108.7 (18)	O13—C13—C14	117.02 (14)
C17—O17—H17	107.1 (17)	C12—C13—C14	121.42 (15)
C8—N7—C5	105.27 (13)	N3—C3—H3A	109.5
C8—N7—H7	124.9 (14)	N3—C3—H3B	109.5
C5—N7—H7	129.7 (14)	N3—C3—H3C	109.5
C15—O15—H15	103.4 (18)	H3A—C3—H3B	109.5
N7—C8—Cl8	120.21 (12)	H3A—C3—H3C	109.5
N7—C8—N9	115.10 (14)	H3B—C3—H3C	109.5
N9—C8—Cl8	124.67 (12)	O14—C14—C20	123.73 (16)
C8—N9—C4	101.99 (13)	O14—C14—C13	119.45 (16)
C2—N3—C3	119.70 (13)	C20—C14—C13	116.82 (14)
C4—N3—C3	121.39 (13)	C17—C18—H18	121.0
C4—N3—C2	118.79 (13)	C19—C18—C17	117.97 (14)
C6—N1—C1	118.40 (13)	C19—C18—H18	121.0
C2—N1—C6	126.00 (13)	O2—C2—N3	121.07 (15)
C2—N1—C1	115.60 (12)	O2—C2—N1	120.72 (14)
C26—C25—H25	120.1	N3—C2—N1	118.20 (13)
C26—C25—C24	119.86 (16)	O15—C15—C20	119.27 (16)
C24—C25—H25	120.1	O15—C15—C16	119.66 (16)
O6—C6—N1	121.15 (13)	C16—C15—C20	121.08 (14)
O6—C6—C5	126.80 (14)	N7—C5—C6	130.69 (14)
N1—C6—C5	112.02 (13)	C4—C5—N7	105.76 (13)
O11—C12—C21	111.14 (13)	C4—C5—C6	123.31 (14)
C13—C12—O11	119.65 (15)	C26—C21—C12	119.41 (15)

C13—C12—C21	129.21 (16)	C22—C21—C12	122.60 (15)
C25—C26—H26	119.4	C22—C21—C26	117.99 (15)
C25—C26—C21	121.13 (15)	C22—C23—H23	119.8
C21—C26—H26	119.4	C22—C23—C24	120.46 (16)
C21—C22—H22	119.6	C24—C23—H23	119.8
C23—C22—H22	119.6	O24—C24—C25	117.05 (16)
C23—C22—C21	120.87 (16)	O24—C24—C23	123.27 (16)
C15—C20—C14	123.32 (15)	C23—C24—C25	119.67 (16)
C19—C20—C14	119.26 (15)	N9—C4—N3	126.56 (14)
C19—C20—C15	117.41 (15)	C5—C4—N9	111.88 (14)
N1—C1—H1A	109.5	C5—C4—N3	121.55 (14)
N1—C1—H1B	109.5	C17—C16—H16	120.4
N1—C1—H1C	109.5	C15—C16—C17	119.13 (15)
H1A—C1—H1B	109.5	C15—C16—H16	120.4
H1A—C1—H1C	109.5	O11—C19—C20	120.93 (15)
H1B—C1—H1C	109.5	O11—C19—C18	116.10 (14)
O17—C17—C18	121.32 (14)	C18—C19—C20	122.98 (15)
O17—C17—C16	117.25 (15)		

Cl8—C8—N9—C4	−178.10 (11)	C1—N1—C2—N3	177.52 (13)
O11—C12—C13—O13	179.37 (14)	C17—C18—C19—O11	−179.80 (13)
O11—C12—C13—C14	−1.9 (2)	C17—C18—C19—C20	0.1 (2)
O11—C12—C21—C26	−1.8 (2)	C13—C12—C21—C26	178.93 (16)
O11—C12—C21—C22	177.83 (14)	C13—C12—C21—C22	−1.5 (3)
O13—C13—C14—O14	1.1 (2)	C3—N3—C2—O2	2.8 (2)
O13—C13—C14—C20	−179.74 (14)	C3—N3—C2—N1	−176.48 (14)
O6—C6—C5—N7	−3.0 (3)	C3—N3—C4—N9	0.4 (2)
O6—C6—C5—C4	−176.45 (15)	C3—N3—C4—C5	179.18 (14)
O17—C17—C18—C19	179.66 (14)	C14—C20—C15—O15	−0.1 (2)
O17—C17—C16—C15	−179.20 (14)	C14—C20—C15—C16	−179.92 (15)
N7—C8—N9—C4	0.04 (17)	C14—C20—C19—O11	0.3 (2)
N7—C5—C4—N9	0.04 (17)	C14—C20—C19—C18	−179.64 (15)
N7—C5—C4—N3	−178.89 (13)	C18—C17—C16—C15	0.8 (2)
O15—C15—C16—C17	179.16 (14)	C2—N3—C4—N9	−175.54 (14)
C8—N7—C5—C6	−174.36 (15)	C2—N3—C4—C5	3.2 (2)
C8—N7—C5—C4	−0.01 (16)	C2—N1—C6—O6	179.45 (15)
C8—N9—C4—N3	178.81 (14)	C2—N1—C6—C5	1.1 (2)
C8—N9—C4—C5	−0.04 (17)	C15—C20—C14—O14	−0.7 (3)
N1—C6—C5—N7	175.25 (14)	C15—C20—C14—C13	−179.86 (14)
N1—C6—C5—C4	1.8 (2)	C15—C20—C19—O11	179.54 (13)
C25—C26—C21—C12	−179.81 (14)	C15—C20—C19—C18	−0.4 (2)
C25—C26—C21—C22	0.6 (2)	C5—N7—C8—Cl8	178.21 (11)
C6—N1—C2—O2	178.98 (14)	C5—N7—C8—N9	−0.02 (18)
C6—N1—C2—N3	−1.8 (2)	C21—C12—C13—O13	−1.4 (3)
C6—C5—C4—N9	174.91 (13)	C21—C12—C13—C14	177.38 (15)
C6—C5—C4—N3	−4.0 (2)	C21—C22—C23—C24	0.4 (2)
C12—O11—C19—C20	−0.7 (2)	C23—C22—C21—C12	179.50 (14)
C12—O11—C19—C18	179.24 (14)	C23—C22—C21—C26	−0.9 (2)

C12—C13—C14—O14	−177.75 (15)	C24—C25—C26—C21	0.2 (2)
C12—C13—C14—C20	1.5 (2)	C4—N3—C2—O2	178.79 (14)
C26—C25—C24—O24	178.69 (14)	C4—N3—C2—N1	−0.4 (2)
C26—C25—C24—C23	−0.7 (2)	C16—C17—C18—C19	−0.3 (2)
C22—C23—C24—O24	−178.97 (15)	C19—O11—C12—C13	1.5 (2)
C22—C23—C24—C25	0.4 (2)	C19—O11—C12—C21	−177.91 (12)
C20—C15—C16—C17	−1.0 (2)	C19—C20—C14—O14	178.54 (15)
C1—N1—C6—O6	0.2 (2)	C19—C20—C14—C13	−0.6 (2)
C1—N1—C6—C5	−178.16 (13)	C19—C20—C15—O15	−179.35 (14)
C1—N1—C2—O2	−1.7 (2)	C19—C20—C15—C16	0.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22···O13	0.95	2.22	2.880 (2)	126
N7—H7···O6^{i}	0.90 (2)	1.77 (2)	2.6666 (17)	170 (2)
O24—H24···N9	0.81 (3)	2.09 (3)	2.8661 (19)	160 (2)
O17—H17···O2^{ii}	0.84 (3)	1.94 (3)	2.7745 (17)	169 (2)
O15—H15···O14	0.89 (3)	1.81 (3)	2.649 (2)	155 (3)
O13—H13···O14	0.85 (3)	2.13 (3)	2.6876 (19)	122 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1.

8-Chloro-1,3-dimethyl-7*H*-purine-2,6-dione–3,5,7-trihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one (2c-ywj68cult100k_auto)

Crystal data

$C_7H_7ClN_4O_2·C_{15}H_{10}O_8$
M_r = 532.84
Triclinic, $P\overline{1}$
a = 7.7588 (5) Å
b = 10.6363 (7) Å
c = 13.4366 (7) Å
α = 78.246 (5)°
β = 81.294 (5)°
γ = 81.946 (5)°
V = 1066.24 (12) Å^3
Z = 2
F(000) = 548
D_x = 1.660 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 3860 reflections
θ = 3.4–74.6°
μ = 2.24 mm^{-1}
T = 100 K
Block, colourless
0.22 × 0.15 × 0.1 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.1788 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)
T_{min} = 0.481, T_{max} = 1.000
6593 measured reflections
4156 independent reflections
3656 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 75.0°, θ_{min} = 3.4°
h = −8→9
k = −10→13
l = −13→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.105$
$S = 1.02$
4156 reflections
364 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.1084P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.25019 (18)	0.76093 (13)	0.83107 (11)	0.0151 (3)
C1	0.3481 (2)	0.64524 (15)	0.88525 (13)	0.0180 (3)
H1A	0.357372	0.656994	0.954639	0.027*
H1B	0.465961	0.631872	0.848021	0.027*
H1C	0.286319	0.569749	0.889188	0.027*
O2	0.36089 (16)	0.69364 (12)	0.68134 (10)	0.0207 (3)
C2	0.2687 (2)	0.77462 (16)	0.72439 (13)	0.0162 (3)
N3	0.17811 (19)	0.88064 (14)	0.67097 (11)	0.0180 (3)
C3	0.1977 (3)	0.9006 (2)	0.55891 (14)	0.0309 (4)
H3A	0.160800	0.826581	0.537842	0.046*
H3B	0.320908	0.908949	0.531574	0.046*
H3C	0.124638	0.979559	0.532342	0.046*
C4	0.0752 (2)	0.96730 (16)	0.72275 (13)	0.0163 (3)
C5	0.0612 (2)	0.95288 (15)	0.82731 (12)	0.0144 (3)
O6	0.13096 (17)	0.82032 (11)	0.98382 (9)	0.0189 (3)
C6	0.1454 (2)	0.84519 (15)	0.88890 (13)	0.0147 (3)
N7	−0.05829 (19)	1.05458 (13)	0.85188 (11)	0.0161 (3)
H7	−0.085 (4)	1.082 (3)	0.912 (2)	0.043 (8)*
Cl8	−0.24487 (6)	1.25846 (4)	0.75369 (3)	0.02111 (12)
C8	−0.1020 (2)	1.12134 (15)	0.76166 (13)	0.0164 (3)
N9	−0.02718 (19)	1.07314 (14)	0.68015 (11)	0.0182 (3)
O11	0.97342 (15)	0.39769 (10)	0.34534 (8)	0.0137 (2)
C12	1.0428 (2)	0.42084 (15)	0.24452 (12)	0.0124 (3)
O13	1.06961 (16)	0.56163 (11)	0.08335 (9)	0.0173 (3)
C13	0.9959 (2)	0.53509 (15)	0.18225 (12)	0.0129 (3)
O14	0.81959 (16)	0.73495 (11)	0.16023 (9)	0.0164 (2)
C14	0.8677 (2)	0.63139 (15)	0.22022 (12)	0.0126 (3)
O15	0.63394 (17)	0.81282 (11)	0.32009 (9)	0.0187 (3)
C15	0.6872 (2)	0.69633 (15)	0.37549 (12)	0.0137 (3)
C16	0.6290 (2)	0.66810 (15)	0.47814 (13)	0.0152 (3)

H16	0.552154	0.729798	0.510419	0.018*
O17	0.62691 (17)	0.51509 (11)	0.63595 (9)	0.0189 (3)
C17	0.6844 (2)	0.54652 (15)	0.53534 (12)	0.0149 (3)
C18	0.8014 (2)	0.45605 (15)	0.49093 (12)	0.0150 (3)
H18	0.840134	0.375084	0.530384	0.018*
C19	0.8597 (2)	0.48765 (15)	0.38762 (12)	0.0129 (3)
C20	0.8045 (2)	0.60609 (15)	0.32661 (12)	0.0135 (3)
C21	1.1649 (2)	0.31125 (14)	0.21722 (12)	0.0125 (3)
C22	1.2417 (2)	0.22428 (15)	0.29541 (12)	0.0133 (3)
H22	1.214975	0.237649	0.364109	0.016*
O23	1.43286 (16)	0.03918 (12)	0.35102 (9)	0.0177 (3)
C23	1.3565 (2)	0.11864 (14)	0.27326 (12)	0.0133 (3)
O24	1.51057 (16)	−0.00680 (11)	0.15546 (9)	0.0169 (2)
C24	1.3939 (2)	0.09712 (14)	0.17314 (12)	0.0134 (3)
O25	1.35292 (17)	0.14967 (12)	0.00043 (9)	0.0197 (3)
C25	1.3129 (2)	0.18198 (15)	0.09547 (12)	0.0136 (3)
C26	1.2010 (2)	0.28978 (14)	0.11602 (12)	0.0132 (3)
H26	1.149435	0.348359	0.062360	0.016*
H24	1.515 (3)	−0.016 (2)	0.099 (2)	0.020 (6)*
H15	0.678 (4)	0.808 (3)	0.259 (2)	0.035 (7)*
H13	1.065 (4)	0.638 (3)	0.063 (2)	0.039 (7)*
H25	1.293 (4)	0.197 (3)	−0.043 (3)	0.051 (9)*
H23	1.481 (4)	−0.028 (3)	0.337 (2)	0.037 (7)*
H17	0.535 (4)	0.578 (3)	0.657 (3)	0.055 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0154 (7)	0.0135 (6)	0.0154 (7)	0.0000 (5)	−0.0023 (5)	−0.0013 (5)
C1	0.0181 (8)	0.0148 (7)	0.0195 (8)	0.0020 (6)	−0.0046 (6)	−0.0005 (6)
O2	0.0176 (6)	0.0251 (6)	0.0197 (6)	0.0036 (5)	−0.0020 (5)	−0.0090 (5)
C2	0.0135 (8)	0.0192 (8)	0.0162 (8)	−0.0018 (6)	−0.0021 (6)	−0.0036 (6)
N3	0.0177 (7)	0.0217 (7)	0.0128 (7)	0.0018 (6)	−0.0019 (5)	−0.0022 (5)
C3	0.0354 (11)	0.0393 (11)	0.0119 (8)	0.0162 (8)	−0.0033 (7)	−0.0043 (7)
C4	0.0155 (8)	0.0167 (7)	0.0158 (8)	−0.0018 (6)	−0.0024 (6)	−0.0008 (6)
C5	0.0142 (8)	0.0127 (7)	0.0158 (8)	−0.0002 (6)	−0.0016 (6)	−0.0026 (6)
O6	0.0277 (7)	0.0146 (5)	0.0131 (5)	0.0019 (5)	−0.0031 (5)	−0.0020 (4)
C6	0.0154 (8)	0.0133 (7)	0.0158 (7)	−0.0028 (6)	−0.0016 (6)	−0.0032 (6)
N7	0.0190 (7)	0.0122 (6)	0.0165 (7)	0.0008 (5)	−0.0034 (5)	−0.0023 (5)
Cl8	0.0233 (2)	0.01376 (19)	0.0251 (2)	0.00315 (14)	−0.00780 (16)	−0.00098 (15)
C8	0.0175 (8)	0.0127 (7)	0.0184 (8)	−0.0008 (6)	−0.0050 (6)	0.0000 (6)
N9	0.0173 (7)	0.0177 (7)	0.0179 (7)	−0.0002 (5)	−0.0039 (5)	0.0001 (5)
O11	0.0157 (6)	0.0110 (5)	0.0122 (5)	0.0036 (4)	−0.0013 (4)	−0.0007 (4)
C12	0.0123 (7)	0.0130 (7)	0.0120 (7)	−0.0006 (6)	−0.0022 (6)	−0.0026 (6)
O13	0.0244 (6)	0.0115 (5)	0.0126 (5)	0.0029 (4)	0.0009 (5)	0.0002 (4)
C13	0.0133 (8)	0.0126 (7)	0.0130 (7)	−0.0001 (6)	−0.0030 (6)	−0.0028 (6)
O14	0.0215 (6)	0.0114 (5)	0.0139 (5)	0.0036 (4)	−0.0036 (5)	0.0007 (4)
C14	0.0132 (8)	0.0111 (7)	0.0138 (7)	−0.0002 (5)	−0.0045 (6)	−0.0015 (5)

O15	0.0247 (7)	0.0130 (5)	0.0147 (6)	0.0080 (5)	−0.0026 (5)	−0.0005 (4)
C15	0.0140 (8)	0.0116 (7)	0.0148 (7)	0.0024 (6)	−0.0054 (6)	−0.0012 (6)
C16	0.0164 (8)	0.0127 (7)	0.0154 (7)	0.0043 (6)	−0.0038 (6)	−0.0028 (6)
O17	0.0237 (6)	0.0166 (5)	0.0126 (5)	0.0046 (5)	0.0008 (5)	−0.0008 (4)
C17	0.0166 (8)	0.0141 (7)	0.0130 (7)	0.0012 (6)	−0.0029 (6)	−0.0013 (6)
C18	0.0175 (8)	0.0117 (7)	0.0142 (7)	0.0025 (6)	−0.0030 (6)	−0.0006 (6)
C19	0.0122 (7)	0.0122 (7)	0.0146 (7)	0.0017 (5)	−0.0030 (6)	−0.0041 (6)
C20	0.0131 (8)	0.0118 (7)	0.0150 (7)	0.0010 (6)	−0.0028 (6)	−0.0019 (6)
C21	0.0113 (7)	0.0095 (6)	0.0163 (7)	−0.0002 (5)	−0.0020 (6)	−0.0018 (5)
C22	0.0142 (8)	0.0131 (7)	0.0121 (7)	−0.0002 (6)	−0.0020 (6)	−0.0019 (6)
O23	0.0235 (6)	0.0146 (6)	0.0128 (5)	0.0086 (5)	−0.0060 (5)	−0.0017 (4)
C23	0.0133 (8)	0.0113 (7)	0.0143 (7)	0.0001 (6)	−0.0044 (6)	0.0007 (6)
O24	0.0216 (6)	0.0146 (5)	0.0128 (6)	0.0081 (4)	−0.0043 (5)	−0.0039 (4)
C24	0.0137 (8)	0.0102 (7)	0.0155 (7)	0.0017 (6)	−0.0019 (6)	−0.0024 (6)
O25	0.0270 (7)	0.0178 (6)	0.0120 (5)	0.0103 (5)	−0.0054 (5)	−0.0038 (4)
C25	0.0154 (8)	0.0131 (7)	0.0120 (7)	0.0001 (6)	−0.0024 (6)	−0.0026 (6)
C26	0.0140 (8)	0.0115 (7)	0.0131 (7)	0.0006 (6)	−0.0037 (6)	−0.0001 (5)

Geometric parameters (Å, º)

N1—C1	1.470 (2)	O14—C14	1.2715 (19)
N1—C2	1.398 (2)	C14—C20	1.423 (2)
N1—C6	1.399 (2)	O15—C15	1.3542 (19)
C1—H1A	0.9800	O15—H15	0.84 (3)
C1—H1B	0.9800	C15—C16	1.370 (2)
C1—H1C	0.9800	C15—C20	1.418 (2)
O2—C2	1.222 (2)	C16—H16	0.9500
C2—N3	1.372 (2)	C16—C17	1.410 (2)
N3—C3	1.465 (2)	O17—C17	1.347 (2)
N3—C4	1.363 (2)	O17—H17	0.96 (3)
C3—H3A	0.9800	C17—C18	1.390 (2)
C3—H3B	0.9800	C18—H18	0.9500
C3—H3C	0.9800	C18—C19	1.381 (2)
C4—C5	1.371 (2)	C19—C20	1.406 (2)
C4—N9	1.360 (2)	C21—C22	1.398 (2)
C5—C6	1.411 (2)	C21—C26	1.405 (2)
C5—N7	1.382 (2)	C22—H22	0.9500
O6—C6	1.240 (2)	C22—C23	1.386 (2)
N7—H7	0.90 (3)	O23—C23	1.3580 (19)
N7—C8	1.340 (2)	O23—H23	0.80 (3)
Cl8—C8	1.6991 (17)	C23—C24	1.391 (2)
C8—N9	1.324 (2)	O24—C24	1.3635 (19)
O11—C12	1.3659 (19)	O24—H24	0.78 (3)
O11—C19	1.3602 (19)	C24—C25	1.398 (2)
C12—C13	1.365 (2)	O25—C25	1.369 (2)
C12—C21	1.467 (2)	O25—H25	0.84 (3)
O13—C13	1.355 (2)	C25—C26	1.386 (2)
O13—H13	0.80 (3)	C26—H26	0.9500

C13—C14	1.440 (2)		
C2—N1—C1	115.68 (14)	O14—C14—C13	120.17 (15)
C2—N1—C6	125.80 (14)	O14—C14—C20	122.92 (15)
C6—N1—C1	118.51 (14)	C20—C14—C13	116.88 (14)
N1—C1—H1A	109.5	C15—O15—H15	104.2 (19)
N1—C1—H1B	109.5	O15—C15—C16	119.54 (15)
N1—C1—H1C	109.5	O15—C15—C20	119.46 (14)
H1A—C1—H1B	109.5	C16—C15—C20	120.99 (14)
H1A—C1—H1C	109.5	C15—C16—H16	120.4
H1B—C1—H1C	109.5	C15—C16—C17	119.29 (15)
O2—C2—N1	120.63 (15)	C17—C16—H16	120.4
O2—C2—N3	121.84 (16)	C17—O17—H17	111 (2)
N3—C2—N1	117.53 (15)	O17—C17—C16	120.66 (14)
C2—N3—C3	119.29 (15)	O17—C17—C18	117.73 (14)
C4—N3—C2	119.55 (14)	C18—C17—C16	121.61 (15)
C4—N3—C3	121.14 (15)	C17—C18—H18	121.1
N3—C3—H3A	109.5	C19—C18—C17	117.86 (14)
N3—C3—H3B	109.5	C19—C18—H18	121.1
N3—C3—H3C	109.5	O11—C19—C18	117.05 (14)
H3A—C3—H3B	109.5	O11—C19—C20	120.24 (14)
H3A—C3—H3C	109.5	C18—C19—C20	122.70 (15)
H3B—C3—H3C	109.5	C15—C20—C14	122.80 (14)
N3—C4—C5	121.88 (15)	C19—C20—C14	119.70 (15)
N9—C4—N3	125.73 (15)	C19—C20—C15	117.50 (15)
N9—C4—C5	112.36 (15)	C22—C21—C12	118.38 (14)
C4—C5—C6	122.59 (15)	C22—C21—C26	119.84 (14)
C4—C5—N7	105.28 (14)	C26—C21—C12	121.74 (14)
N7—C5—C6	131.86 (15)	C21—C22—H22	119.8
N1—C6—C5	112.56 (14)	C23—C22—C21	120.35 (15)
O6—C6—N1	120.57 (15)	C23—C22—H22	119.8
O6—C6—C5	126.85 (16)	C23—O23—H23	114 (2)
C5—N7—H7	129 (2)	C22—C23—C24	120.18 (14)
C8—N7—C5	104.78 (14)	O23—C23—C22	118.36 (14)
C8—N7—H7	125.2 (19)	O23—C23—C24	121.44 (14)
N7—C8—Cl8	121.67 (14)	C24—O24—H24	110.4 (18)
N9—C8—N7	115.62 (15)	C23—C24—C25	119.42 (15)
N9—C8—Cl8	122.71 (13)	O24—C24—C23	117.66 (14)
C8—N9—C4	101.95 (14)	O24—C24—C25	122.92 (15)
C19—O11—C12	121.66 (12)	C25—O25—H25	112 (2)
O11—C12—C21	111.79 (13)	O25—C25—C24	114.95 (14)
C13—C12—O11	120.48 (14)	O25—C25—C26	124.01 (14)
C13—C12—C21	127.73 (15)	C26—C25—C24	121.04 (15)
C13—O13—H13	111 (2)	C21—C26—H26	120.4
C12—C13—C14	120.83 (15)	C25—C26—C21	119.12 (14)
O13—C13—C12	121.06 (14)	C25—C26—H26	120.4
O13—C13—C14	118.10 (14)		

N1—C2—N3—C3	178.28 (16)	C12—C21—C22—C23	179.36 (14)
N1—C2—N3—C4	0.0 (2)	C12—C21—C26—C25	−177.73 (14)
C1—N1—C2—O2	0.2 (2)	O13—C13—C14—O14	3.6 (2)
C1—N1—C2—N3	179.38 (14)	O13—C13—C14—C20	−174.38 (13)
C1—N1—C6—C5	179.09 (13)	C13—C12—C21—C22	158.11 (15)
C1—N1—C6—O6	−2.4 (2)	C13—C12—C21—C26	−24.1 (2)
O2—C2—N3—C3	−2.6 (3)	C13—C14—C20—C15	175.60 (14)
O2—C2—N3—C4	179.15 (15)	C13—C14—C20—C19	−3.9 (2)
C2—N1—C6—C5	−2.4 (2)	O14—C14—C20—C15	−2.4 (2)
C2—N1—C6—O6	176.10 (15)	O14—C14—C20—C19	178.11 (14)
C2—N3—C4—C5	1.0 (2)	O15—C15—C16—C17	179.77 (14)
C2—N3—C4—N9	−176.99 (15)	O15—C15—C20—C14	−1.1 (2)
N3—C4—C5—C6	−2.8 (2)	O15—C15—C20—C19	178.47 (13)
N3—C4—C5—N7	−177.53 (14)	C15—C16—C17—O17	−178.78 (14)
N3—C4—N9—C8	178.27 (16)	C15—C16—C17—C18	2.2 (2)
C3—N3—C4—C5	−177.25 (17)	C16—C15—C20—C14	179.84 (15)
C3—N3—C4—N9	4.8 (3)	C16—C15—C20—C19	−0.6 (2)
C4—C5—C6—N1	3.3 (2)	C16—C17—C18—C19	−1.4 (2)
C4—C5—C6—O6	−175.06 (16)	O17—C17—C18—C19	179.55 (14)
C4—C5—N7—C8	−1.19 (17)	C17—C18—C19—O11	−179.66 (14)
C5—C4—N9—C8	0.14 (18)	C17—C18—C19—C20	−0.4 (2)
C5—N7—C8—Cl8	−178.79 (12)	C18—C19—C20—C14	−179.00 (14)
C5—N7—C8—N9	1.43 (19)	C18—C19—C20—C15	1.5 (2)
C6—N1—C2—O2	−178.30 (15)	C19—O11—C12—C13	−1.6 (2)
C6—N1—C2—N3	0.8 (2)	C19—O11—C12—C21	178.59 (13)
C6—C5—N7—C8	−175.19 (17)	C20—C15—C16—C17	−1.1 (2)
N7—C5—C6—N1	176.45 (15)	C21—C12—C13—O13	−3.2 (2)
N7—C5—C6—O6	−1.9 (3)	C21—C12—C13—C14	177.36 (14)
N7—C8—N9—C4	−0.99 (19)	C21—C22—C23—O23	177.69 (14)
Cl8—C8—N9—C4	179.23 (12)	C21—C22—C23—C24	−1.1 (2)
N9—C4—C5—C6	175.37 (14)	C22—C21—C26—C25	0.0 (2)
N9—C4—C5—N7	0.68 (18)	C22—C23—C24—O24	178.68 (14)
O11—C12—C13—O13	177.00 (13)	C22—C23—C24—C25	−0.8 (2)
O11—C12—C13—C14	−2.5 (2)	O23—C23—C24—O24	−0.1 (2)
O11—C12—C21—C22	−22.06 (19)	O23—C23—C24—C25	−179.62 (14)
O11—C12—C21—C26	155.70 (14)	C23—C24—C25—O25	−177.55 (14)
O11—C19—C20—C14	0.2 (2)	C23—C24—C25—C26	2.4 (2)
O11—C19—C20—C15	−179.36 (13)	O24—C24—C25—O25	3.0 (2)
C12—O11—C19—C18	−178.06 (13)	O24—C24—C25—C26	−177.07 (14)
C12—O11—C19—C20	2.7 (2)	C24—C25—C26—C21	−2.0 (2)
C12—C13—C14—O14	−176.87 (14)	O25—C25—C26—C21	177.98 (14)
C12—C13—C14—C20	5.1 (2)	C26—C21—C22—C23	1.6 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O6^{i}	0.90 (3)	1.88 (3)	2.7518 (19)	165 (3)
O24—H24···O25^{ii}	0.78 (3)	2.20 (3)	2.8272 (17)	139 (2)

O24—H24···O25	0.78 (3)	2.32 (2)	2.7191 (17)	113 (2)
O15—H15···O14	0.84 (3)	1.82 (3)	2.6131 (17)	155 (3)
O25—H25···O14^{iii}	0.84 (3)	1.89 (3)	2.7004 (17)	164 (3)
O23—H23···O15^{iv}	0.80 (3)	1.96 (3)	2.7500 (17)	168 (3)
O17—H17···O2	0.96 (3)	1.74 (3)	2.6891 (18)	174 (3)

Symmetry codes: (i) −*x*, −*y*+2, −*z*+2; (ii) −*x*+3, −*y*, −*z*; (iii) −*x*+2, −*y*+1, −*z*; (iv) *x*+1, *y*−1, *z*.

8-Bromo-1,3-dimethyl-7*H*-purine-2,6-dione–3,5,7-trihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one (3a_ywj75cult100k_auto)

Crystal data

$C_7H_7BrN_4O_2 \cdot C_{15}H_{10}O_6$
M_r = 545.30
Monoclinic, $P2_1/n$
a = 10.1578 (1) Å
b = 19.8059 (3) Å
c = 10.4534 (1) Å
β = 92.539 (1)°
V = 2101.00 (4) Å^3
Z = 4
$F(000)$ = 1104
D_x = 1.724 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 5868 reflections
θ = 4.5–74.9°
μ = 3.22 mm^{-1}
T = 100 K
Block, colourless
0.2 × 0.1 × 0.08 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3577 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)
T_{min} = 0.871, T_{max} = 1.000
12439 measured reflections
4220 independent reflections
3606 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 74.5°, θ_{min} = 4.5°
h = −12→11
k = −24→24
l = −7→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.06
4220 reflections
334 parameters
4 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 1.328P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br8	−0.28312 (2)	0.84226 (2)	0.59619 (2)	0.02291 (9)

O11	0.51187 (15)	0.55919 (8)	0.32110 (15)	0.0184 (3)
O6	0.18889 (15)	0.98561 (8)	0.46024 (15)	0.0203 (3)
O2	0.39863 (16)	0.78601 (8)	0.39101 (17)	0.0239 (4)
O17	0.64299 (17)	0.78660 (8)	0.28399 (17)	0.0234 (4)
O13	0.68049 (19)	0.39920 (9)	0.28718 (18)	0.0297 (4)
O15	0.95566 (16)	0.61447 (10)	0.22180 (17)	0.0276 (4)
O14	0.87450 (17)	0.48809 (9)	0.24441 (17)	0.0296 (4)
O24	0.06844 (19)	0.35537 (10)	0.44004 (19)	0.0297 (4)
N7	−0.05272 (17)	0.90020 (9)	0.51581 (17)	0.0163 (4)
H7	−0.084029	0.941118	0.526827	0.020*
N1	0.29208 (17)	0.88506 (9)	0.42487 (17)	0.0165 (4)
N3	0.18375 (18)	0.77908 (9)	0.44156 (18)	0.0176 (4)
N9	−0.04319 (18)	0.78687 (9)	0.51047 (18)	0.0173 (4)
C8	−0.1144 (2)	0.84183 (11)	0.5338 (2)	0.0169 (4)
C21	0.4156 (2)	0.45348 (12)	0.3518 (2)	0.0196 (4)
C12	0.5340 (2)	0.49060 (11)	0.3195 (2)	0.0193 (4)
C5	0.0711 (2)	0.88287 (11)	0.4761 (2)	0.0169 (4)
C22	0.2991 (2)	0.48896 (12)	0.3749 (2)	0.0215 (5)
H22	0.298828	0.536846	0.369724	0.026*
C2	0.2965 (2)	0.81502 (11)	0.4177 (2)	0.0183 (4)
C17	0.6700 (2)	0.71970 (12)	0.2802 (2)	0.0194 (4)
C18	0.5737 (2)	0.67150 (11)	0.3029 (2)	0.0181 (4)
H18	0.486619	0.684540	0.321514	0.022*
C19	0.6087 (2)	0.60457 (11)	0.2976 (2)	0.0176 (4)
C1	0.4166 (2)	0.92066 (11)	0.4025 (2)	0.0202 (5)
H1A	0.481830	0.909577	0.471135	0.030*
H1B	0.400922	0.969482	0.401426	0.030*
H1C	0.449691	0.906552	0.319981	0.030*
C3	0.1874 (2)	0.70499 (11)	0.4455 (2)	0.0236 (5)
H3A	0.268926	0.688990	0.408817	0.035*
H3B	0.111414	0.686839	0.395731	0.035*
H3C	0.184451	0.689706	0.534450	0.035*
C4	0.0726 (2)	0.81405 (11)	0.4742 (2)	0.0170 (4)
C23	0.1852 (2)	0.45568 (12)	0.4049 (2)	0.0230 (5)
H23	0.107517	0.480634	0.420039	0.028*
C6	0.1824 (2)	0.92365 (11)	0.4534 (2)	0.0161 (4)
C20	0.7350 (2)	0.58267 (12)	0.2696 (2)	0.0197 (4)
C13	0.6551 (2)	0.46702 (12)	0.2911 (2)	0.0223 (5)
C26	0.4129 (2)	0.38311 (12)	0.3621 (2)	0.0231 (5)
H26	0.490814	0.358004	0.348718	0.028*
C14	0.7633 (2)	0.51180 (12)	0.2664 (2)	0.0224 (5)
C24	0.1840 (2)	0.38491 (12)	0.4131 (2)	0.0229 (5)
C25	0.2987 (3)	0.34937 (12)	0.3914 (2)	0.0242 (5)
H25	0.298828	0.301487	0.396710	0.029*
C15	0.8309 (2)	0.63320 (13)	0.2479 (2)	0.0222 (5)
C16	0.7988 (2)	0.70103 (13)	0.2518 (2)	0.0224 (5)
H16	0.862903	0.734488	0.235508	0.027*
H13	0.753 (3)	0.3987 (16)	0.270 (3)	0.035 (9)*

H17	0.578 (3)	0.7922 (16)	0.306 (3)	0.030 (9)*
H24	0.079 (4)	0.3196 (14)	0.444 (3)	0.044 (11)*
H15	0.950 (3)	0.5736 (14)	0.222 (3)	0.039 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br8	0.01464 (13)	0.02006 (14)	0.03439 (15)	−0.00009 (9)	0.00507 (9)	0.00118 (10)
O11	0.0142 (7)	0.0154 (7)	0.0256 (8)	0.0015 (6)	0.0024 (6)	−0.0008 (6)
O6	0.0169 (8)	0.0132 (7)	0.0309 (9)	0.0005 (6)	0.0031 (6)	0.0010 (6)
O2	0.0168 (8)	0.0174 (8)	0.0383 (9)	0.0035 (6)	0.0096 (7)	−0.0005 (7)
O17	0.0166 (9)	0.0199 (8)	0.0342 (9)	−0.0019 (7)	0.0060 (7)	−0.0030 (7)
O13	0.0241 (10)	0.0235 (9)	0.0421 (11)	0.0122 (7)	0.0078 (8)	0.0019 (8)
O15	0.0126 (8)	0.0366 (11)	0.0339 (10)	0.0041 (7)	0.0047 (7)	−0.0015 (8)
O14	0.0208 (9)	0.0325 (10)	0.0360 (10)	0.0135 (7)	0.0057 (7)	0.0009 (8)
O24	0.0280 (10)	0.0204 (9)	0.0409 (11)	−0.0065 (8)	0.0031 (8)	0.0057 (8)
N7	0.0114 (8)	0.0145 (9)	0.0233 (9)	0.0004 (7)	0.0025 (7)	0.0015 (7)
N1	0.0130 (9)	0.0146 (9)	0.0222 (9)	0.0008 (7)	0.0030 (7)	0.0017 (7)
N3	0.0144 (9)	0.0138 (9)	0.0248 (9)	0.0014 (7)	0.0051 (7)	−0.0007 (7)
N9	0.0131 (9)	0.0166 (9)	0.0221 (9)	0.0000 (7)	0.0011 (7)	−0.0008 (7)
C8	0.0111 (10)	0.0183 (11)	0.0212 (11)	−0.0003 (8)	0.0012 (8)	0.0012 (8)
C21	0.0211 (11)	0.0201 (11)	0.0174 (10)	0.0012 (9)	−0.0005 (8)	0.0001 (8)
C12	0.0224 (11)	0.0168 (10)	0.0186 (11)	0.0034 (9)	−0.0006 (8)	0.0001 (8)
C5	0.0137 (10)	0.0175 (10)	0.0196 (10)	0.0035 (8)	0.0006 (8)	−0.0001 (8)
C22	0.0233 (12)	0.0166 (11)	0.0245 (11)	−0.0002 (9)	0.0010 (9)	0.0012 (9)
C2	0.0182 (11)	0.0159 (10)	0.0208 (11)	0.0000 (8)	0.0031 (9)	−0.0006 (8)
C17	0.0174 (11)	0.0223 (11)	0.0186 (10)	−0.0007 (9)	0.0001 (8)	−0.0021 (9)
C18	0.0107 (10)	0.0234 (11)	0.0201 (10)	0.0017 (8)	0.0014 (8)	−0.0023 (9)
C19	0.0146 (10)	0.0216 (11)	0.0166 (10)	−0.0014 (8)	0.0001 (8)	−0.0018 (8)
C1	0.0144 (10)	0.0183 (11)	0.0281 (12)	−0.0018 (8)	0.0044 (9)	0.0010 (9)
C3	0.0224 (12)	0.0133 (11)	0.0357 (13)	−0.0005 (9)	0.0077 (10)	−0.0020 (9)
C4	0.0149 (10)	0.0173 (10)	0.0185 (10)	0.0009 (8)	−0.0002 (8)	−0.0005 (8)
C23	0.0210 (12)	0.0209 (11)	0.0272 (12)	−0.0006 (9)	0.0029 (9)	0.0005 (9)
C6	0.0140 (10)	0.0160 (10)	0.0184 (10)	0.0018 (8)	0.0007 (8)	0.0011 (8)
C20	0.0141 (10)	0.0253 (12)	0.0196 (11)	0.0041 (9)	0.0003 (8)	−0.0008 (9)
C13	0.0235 (12)	0.0215 (11)	0.0217 (11)	0.0074 (9)	0.0001 (9)	0.0009 (9)
C26	0.0256 (12)	0.0184 (11)	0.0252 (11)	0.0033 (9)	0.0006 (9)	0.0003 (9)
C14	0.0191 (11)	0.0281 (12)	0.0200 (11)	0.0077 (9)	0.0009 (9)	−0.0014 (9)
C24	0.0280 (13)	0.0205 (11)	0.0200 (11)	−0.0055 (9)	−0.0008 (9)	0.0038 (9)
C25	0.0330 (14)	0.0165 (11)	0.0228 (12)	−0.0014 (9)	−0.0006 (10)	0.0022 (9)
C15	0.0127 (10)	0.0347 (13)	0.0193 (11)	0.0022 (9)	0.0009 (8)	−0.0022 (9)
C16	0.0150 (11)	0.0294 (12)	0.0227 (11)	−0.0041 (9)	0.0012 (8)	−0.0015 (9)

Geometric parameters (Å, º)

Br8—C8	1.860 (2)	C12—C13	1.360 (3)
O11—C12	1.377 (3)	C5—C4	1.363 (3)
O11—C19	1.363 (3)	C5—C6	1.418 (3)

O6—C6	1.231 (3)	C22—H22	0.9500
O2—C2	1.229 (3)	C22—C23	1.380 (3)
O17—C17	1.354 (3)	C17—C18	1.395 (3)
O17—H17	0.72 (3)	C17—C16	1.403 (3)
O13—C13	1.369 (3)	C18—H18	0.9500
O13—H13	0.76 (3)	C18—C19	1.374 (3)
O15—C15	1.359 (3)	C19—C20	1.398 (3)
O15—H15	0.81 (3)	C1—H1A	0.9800
O14—C14	1.254 (3)	C1—H1B	0.9800
O24—C24	1.353 (3)	C1—H1C	0.9800
O24—H24	0.72 (3)	C3—H3A	0.9800
N7—H7	0.8800	C3—H3B	0.9800
N7—C8	1.332 (3)	C3—H3C	0.9800
N7—C5	1.385 (3)	C23—H23	0.9500
N1—C2	1.390 (3)	C23—C24	1.404 (3)
N1—C1	1.476 (3)	C20—C14	1.434 (3)
N1—C6	1.394 (3)	C20—C15	1.422 (3)
N3—C2	1.380 (3)	C13—C14	1.445 (3)
N3—C3	1.468 (3)	C26—H26	0.9500
N3—C4	1.380 (3)	C26—C25	1.385 (3)
N9—C8	1.335 (3)	C24—C25	1.388 (3)
N9—C4	1.363 (3)	C25—H25	0.9500
C21—C12	1.462 (3)	C15—C16	1.384 (4)
C21—C22	1.406 (3)	C16—H16	0.9500
C21—C26	1.398 (3)		

C19—O11—C12	121.92 (18)	N1—C1—H1C	109.5
C17—O17—H17	111 (3)	H1A—C1—H1B	109.5
C13—O13—H13	102 (2)	H1A—C1—H1C	109.5
C15—O15—H15	102 (2)	H1B—C1—H1C	109.5
C24—O24—H24	108 (3)	N3—C3—H3A	109.5
C8—N7—H7	127.3	N3—C3—H3B	109.5
C8—N7—C5	105.44 (18)	N3—C3—H3C	109.5
C5—N7—H7	127.3	H3A—C3—H3B	109.5
C2—N1—C1	116.01 (17)	H3A—C3—H3C	109.5
C2—N1—C6	125.90 (18)	H3B—C3—H3C	109.5
C6—N1—C1	118.07 (18)	N9—C4—N3	126.6 (2)
C2—N3—C3	120.00 (18)	N9—C4—C5	112.38 (19)
C4—N3—C2	118.68 (19)	C5—C4—N3	121.0 (2)
C4—N3—C3	120.99 (18)	C22—C23—H23	120.0
C8—N9—C4	102.13 (18)	C22—C23—C24	120.0 (2)
N7—C8—Br8	119.46 (16)	C24—C23—H23	120.0
N7—C8—N9	114.80 (19)	O6—C6—N1	121.19 (19)
N9—C8—Br8	125.64 (16)	O6—C6—C5	126.8 (2)
C22—C21—C12	119.7 (2)	N1—C6—C5	111.96 (19)
C26—C21—C12	122.5 (2)	C19—C20—C14	119.7 (2)
C26—C21—C22	117.8 (2)	C19—C20—C15	117.2 (2)
O11—C12—C21	110.88 (19)	C15—C20—C14	123.1 (2)

C13—C12—O11	119.4 (2)	O13—C13—C14	116.8 (2)
C13—C12—C21	129.7 (2)	C12—C13—O13	121.1 (2)
N7—C5—C6	130.6 (2)	C12—C13—C14	122.0 (2)
C4—C5—N7	105.24 (19)	C21—C26—H26	119.4
C4—C5—C6	123.9 (2)	C25—C26—C21	121.1 (2)
C21—C22—H22	119.3	C25—C26—H26	119.4
C23—C22—C21	121.4 (2)	O14—C14—C20	123.7 (2)
C23—C22—H22	119.3	O14—C14—C13	120.1 (2)
O2—C2—N1	120.5 (2)	C20—C14—C13	116.2 (2)
O2—C2—N3	121.0 (2)	O24—C24—C23	117.0 (2)
N3—C2—N1	118.44 (19)	O24—C24—C25	123.9 (2)
O17—C17—C18	121.3 (2)	C25—C24—C23	119.1 (2)
O17—C17—C16	117.1 (2)	C26—C25—C24	120.6 (2)
C18—C17—C16	121.5 (2)	C26—C25—H25	119.7
C17—C18—H18	121.0	C24—C25—H25	119.7
C19—C18—C17	118.0 (2)	O15—C15—C20	119.4 (2)
C19—C18—H18	121.0	O15—C15—C16	119.6 (2)
O11—C19—C18	116.04 (19)	C16—C15—C20	120.9 (2)
O11—C19—C20	120.6 (2)	C17—C16—H16	120.5
C18—C19—C20	123.3 (2)	C15—C16—C17	119.1 (2)
N1—C1—H1A	109.5	C15—C16—H16	120.5
N1—C1—H1B	109.5		

O11—C12—C13—O13	−179.2 (2)	C17—C18—C19—O11	179.64 (19)
O11—C12—C13—C14	2.0 (3)	C17—C18—C19—C20	−0.6 (3)
O11—C19—C20—C14	−0.4 (3)	C18—C17—C16—C15	−0.8 (3)
O11—C19—C20—C15	−179.23 (19)	C18—C19—C20—C14	179.8 (2)
O17—C17—C18—C19	−179.9 (2)	C18—C19—C20—C15	1.0 (3)
O17—C17—C16—C15	179.6 (2)	C19—O11—C12—C21	178.10 (18)
O13—C13—C14—O14	−1.0 (3)	C19—O11—C12—C13	−1.4 (3)
O13—C13—C14—C20	179.4 (2)	C19—C20—C14—O14	−178.6 (2)
O15—C15—C16—C17	−179.3 (2)	C19—C20—C14—C13	0.9 (3)
O24—C24—C25—C26	178.9 (2)	C19—C20—C15—O15	179.2 (2)
N7—C5—C4—N3	−178.38 (19)	C19—C20—C15—C16	−1.3 (3)
N7—C5—C4—N9	0.0 (2)	C1—N1—C2—O2	−1.7 (3)
N7—C5—C6—O6	−3.2 (4)	C1—N1—C2—N3	178.16 (19)
N7—C5—C6—N1	174.8 (2)	C1—N1—C6—O6	−0.2 (3)
C8—N7—C5—C4	−0.2 (2)	C1—N1—C6—C5	−178.34 (18)
C8—N7—C5—C6	−174.0 (2)	C3—N3—C2—O2	5.0 (3)
C8—N9—C4—N3	178.5 (2)	C3—N3—C2—N1	−174.92 (19)
C8—N9—C4—C5	0.2 (2)	C3—N3—C4—N9	−1.1 (3)
C21—C12—C13—O13	1.4 (4)	C3—N3—C4—C5	177.0 (2)
C21—C12—C13—C14	−177.4 (2)	C4—N3—C2—O2	178.4 (2)
C21—C22—C23—C24	0.1 (4)	C4—N3—C2—N1	−1.4 (3)
C21—C26—C25—C24	−0.9 (4)	C4—N9—C8—Br8	−176.68 (16)
C12—O11—C19—C18	−179.60 (19)	C4—N9—C8—N7	−0.3 (3)
C12—O11—C19—C20	0.6 (3)	C4—C5—C6—O6	−176.0 (2)
C12—C21—C22—C23	179.6 (2)	C4—C5—C6—N1	2.0 (3)

C12—C21—C26—C25	−179.2 (2)	C23—C24—C25—C26	0.0 (4)
C12—C13—C14—O14	177.8 (2)	C6—N1—C2—O2	179.6 (2)
C12—C13—C14—C20	−1.8 (3)	C6—N1—C2—N3	−0.5 (3)
C5—N7—C8—Br8	176.92 (15)	C6—C5—C4—N3	−4.0 (3)
C5—N7—C8—N9	0.3 (3)	C6—C5—C4—N9	174.3 (2)
C22—C21—C12—O11	2.1 (3)	C20—C15—C16—C17	1.2 (3)
C22—C21—C12—C13	−178.5 (2)	C26—C21—C12—O11	−177.4 (2)
C22—C21—C26—C25	1.3 (3)	C26—C21—C12—C13	2.1 (4)
C22—C23—C24—O24	−178.6 (2)	C26—C21—C22—C23	−0.9 (3)
C22—C23—C24—C25	0.4 (4)	C14—C20—C15—O15	0.4 (3)
C2—N1—C6—O6	178.4 (2)	C14—C20—C15—C16	179.9 (2)
C2—N1—C6—C5	0.3 (3)	C15—C20—C14—O14	0.1 (4)
C2—N3—C4—N9	−174.5 (2)	C15—C20—C14—C13	179.7 (2)
C2—N3—C4—C5	3.6 (3)	C16—C17—C18—C19	0.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O6^{i}	0.88	1.81	2.669 (2)	165
O13—H13···O14	0.76 (3)	2.18 (3)	2.695 (3)	125 (3)
O17—H17···O2	0.72 (3)	2.06 (3)	2.768 (2)	166 (3)
O24—H24···N9^{ii}	0.72 (3)	2.19 (3)	2.878 (3)	160 (4)
O15—H15···O14	0.81 (3)	1.88 (3)	2.649 (3)	158 (3)

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1.

8-Bromo-1,3-dimethyl-7*H*-purine-2,6-dione–\ 3,5,7-trihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one (3c-ywj230_cu100k_auto)

Crystal data

$C_7H_7BrN_4O_2·C_{15}H_{10}O_8$
M_r = 577.30
Triclinic, $P\overline{1}$
a = 7.7841 (3) Å
b = 10.7050 (5) Å
c = 13.4516 (6) Å
α = 78.741 (4)°
β = 81.454 (4)°
γ = 81.657 (4)°
V = 1079.31 (8) Å^3
Z = 2
F(000) = 584
D_x = 1.776 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 3815 reflections
θ = 3.4–74.5°
μ = 3.25 mm^{-1}
T = 100 K
Block, colourless
0.09 × 0.06 × 0.05 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3577 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)
T_{min} = 0.899, T_{max} = 1.000
6335 measured reflections
4197 independent reflections
3614 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 74.9°, θ_{min} = 3.4°
h = −9→6
k = −11→13
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.097$
$S = 1.04$
4197 reflections
342 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.7157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.08$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2507 (3)	0.7598 (2)	0.83167 (17)	0.0145 (4)
C1	0.3504 (3)	0.6455 (2)	0.8854 (2)	0.0171 (5)
H1A	0.363325	0.659124	0.953741	0.026*
H1B	0.466295	0.630615	0.846844	0.026*
H1C	0.287795	0.570772	0.891399	0.026*
O2	0.3597 (3)	0.6902 (2)	0.68281 (15)	0.0218 (4)
C2	0.2674 (3)	0.7713 (3)	0.7249 (2)	0.0169 (5)
N3	0.1753 (3)	0.8760 (2)	0.67180 (17)	0.0190 (5)
C3	0.1903 (5)	0.8923 (4)	0.5599 (2)	0.0340 (8)
H3A	0.073303	0.912576	0.538075	0.051*
H3B	0.247595	0.812796	0.537966	0.051*
H3C	0.259975	0.962557	0.529203	0.051*
C4	0.0730 (3)	0.9628 (3)	0.7231 (2)	0.0157 (5)
C5	0.0602 (3)	0.9506 (2)	0.82806 (19)	0.0138 (5)
O6	0.1319 (3)	0.82084 (18)	0.98406 (14)	0.0188 (4)
C6	0.1460 (3)	0.8442 (2)	0.8893 (2)	0.0142 (5)
N7	−0.0594 (3)	1.0519 (2)	0.85167 (17)	0.0154 (4)
H7	−0.097161	1.070590	0.912645	0.019*
Br8	−0.26068 (4)	1.26566 (3)	0.75069 (2)	0.02122 (10)
C8	−0.1061 (3)	1.1163 (2)	0.7614 (2)	0.0161 (5)
N9	−0.0315 (3)	1.0667 (2)	0.68071 (18)	0.0182 (5)
O11	0.9718 (2)	0.40130 (16)	0.34283 (13)	0.0117 (3)
C12	1.0415 (3)	0.4232 (2)	0.24229 (18)	0.0105 (4)
O13	1.0704 (2)	0.56286 (17)	0.08178 (13)	0.0150 (4)
H13	1.069302	0.642533	0.063085	0.023*
C13	0.9962 (3)	0.5371 (2)	0.18070 (18)	0.0112 (4)
O14	0.8240 (2)	0.73771 (17)	0.15912 (13)	0.0150 (4)
C14	0.8692 (3)	0.6345 (2)	0.21817 (19)	0.0115 (5)
O15	0.6427 (3)	0.81863 (17)	0.31971 (14)	0.0161 (4)
H15	0.690131	0.823066	0.259023	0.024*

C15	0.6917 (3)	0.7013 (2)	0.37416 (19)	0.0124 (5)
C16	0.6322 (3)	0.6739 (2)	0.4760 (2)	0.0144 (5)
H16	0.556503	0.735977	0.508463	0.017*
O17	0.6262 (3)	0.52055 (18)	0.63232 (14)	0.0194 (4)
H17	0.556569	0.581774	0.650794	0.029*
C17	0.6849 (3)	0.5516 (2)	0.53234 (19)	0.0143 (5)
C18	0.7995 (3)	0.4611 (2)	0.48758 (19)	0.0143 (5)
H18	0.836199	0.380077	0.526424	0.017*
C19	0.8592 (3)	0.4918 (2)	0.38487 (18)	0.0120 (5)
C20	0.8062 (3)	0.6101 (2)	0.32468 (19)	0.0109 (5)
C21	1.1635 (3)	0.3136 (2)	0.21596 (18)	0.0105 (4)
C22	1.2410 (3)	0.2275 (2)	0.29392 (19)	0.0120 (5)
H22	1.213874	0.241525	0.362402	0.014*
O23	1.4327 (3)	0.04403 (18)	0.35031 (14)	0.0166 (4)
H23	1.479710	−0.024368	0.331020	0.025*
C23	1.3564 (3)	0.1226 (2)	0.27253 (19)	0.0113 (5)
O24	1.5122 (2)	−0.00381 (17)	0.15509 (14)	0.0154 (4)
H24	1.516502	−0.013395	0.094230	0.023*
C24	1.3946 (3)	0.1001 (2)	0.17208 (19)	0.0111 (5)
O25	1.3538 (3)	0.14938 (18)	−0.00024 (14)	0.0177 (4)
H25	1.285561	0.193852	−0.039911	0.026*
C25	1.3128 (3)	0.1829 (2)	0.09459 (18)	0.0111 (4)
C26	1.2004 (3)	0.2907 (2)	0.11472 (18)	0.0106 (4)
H26	1.149142	0.348390	0.060999	0.013*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0147 (10)	0.0144 (10)	0.0140 (10)	−0.0004 (8)	0.0000 (8)	−0.0036 (8)
C1	0.0155 (12)	0.0135 (12)	0.0201 (13)	0.0017 (10)	−0.0021 (10)	−0.0006 (10)
O2	0.0180 (9)	0.0278 (11)	0.0201 (10)	0.0043 (8)	−0.0003 (8)	−0.0121 (8)
C2	0.0136 (12)	0.0211 (13)	0.0165 (12)	−0.0012 (10)	0.0006 (10)	−0.0067 (10)
N3	0.0188 (11)	0.0249 (12)	0.0118 (10)	0.0011 (9)	−0.0006 (8)	−0.0035 (9)
C3	0.0382 (18)	0.0441 (19)	0.0118 (13)	0.0185 (15)	−0.0007 (12)	−0.0050 (12)
C4	0.0146 (12)	0.0176 (12)	0.0143 (12)	−0.0035 (10)	−0.0004 (9)	−0.0010 (9)
C5	0.0143 (12)	0.0134 (11)	0.0131 (12)	−0.0009 (9)	−0.0006 (9)	−0.0023 (9)
O6	0.0294 (11)	0.0137 (9)	0.0119 (8)	0.0012 (8)	−0.0021 (8)	−0.0020 (7)
C6	0.0148 (12)	0.0130 (12)	0.0150 (12)	−0.0018 (9)	−0.0006 (9)	−0.0036 (9)
N7	0.0198 (11)	0.0109 (10)	0.0152 (10)	0.0003 (8)	−0.0016 (8)	−0.0031 (8)
Br8	0.02409 (16)	0.01388 (15)	0.02499 (16)	0.00142 (10)	−0.00728 (11)	−0.00128 (10)
C8	0.0161 (12)	0.0118 (11)	0.0196 (13)	−0.0006 (9)	−0.0036 (10)	−0.0008 (9)
N9	0.0179 (11)	0.0194 (11)	0.0159 (11)	0.0005 (9)	−0.0027 (9)	−0.0014 (9)
O11	0.0144 (8)	0.0082 (8)	0.0097 (8)	0.0045 (6)	0.0013 (6)	−0.0014 (6)
C12	0.0121 (11)	0.0100 (11)	0.0095 (11)	−0.0014 (9)	−0.0003 (9)	−0.0029 (9)
O13	0.0236 (10)	0.0087 (8)	0.0094 (8)	0.0023 (7)	0.0029 (7)	−0.0001 (6)
C13	0.0137 (11)	0.0104 (11)	0.0087 (11)	0.0000 (9)	0.0001 (9)	−0.0020 (9)
O14	0.0206 (9)	0.0107 (8)	0.0112 (8)	0.0045 (7)	−0.0025 (7)	−0.0003 (6)
C14	0.0120 (11)	0.0092 (11)	0.0133 (12)	0.0002 (9)	−0.0022 (9)	−0.0025 (9)

O15	0.0218 (9)	0.0094 (8)	0.0136 (8)	0.0080 (7)	−0.0018 (7)	−0.0009 (7)
C15	0.0131 (11)	0.0094 (11)	0.0143 (12)	0.0035 (9)	−0.0043 (9)	−0.0026 (9)
C16	0.0151 (12)	0.0112 (11)	0.0159 (12)	0.0051 (9)	−0.0013 (9)	−0.0056 (9)
O17	0.0258 (10)	0.0160 (9)	0.0112 (9)	0.0066 (8)	0.0046 (7)	−0.0015 (7)
C17	0.0176 (12)	0.0144 (12)	0.0099 (11)	0.0012 (9)	−0.0010 (9)	−0.0021 (9)
C18	0.0190 (12)	0.0102 (11)	0.0115 (11)	0.0034 (9)	−0.0015 (10)	−0.0005 (9)
C19	0.0138 (11)	0.0102 (11)	0.0112 (11)	0.0035 (9)	−0.0011 (9)	−0.0041 (9)
C20	0.0106 (11)	0.0095 (11)	0.0127 (11)	0.0016 (9)	−0.0026 (9)	−0.0034 (9)
C21	0.0113 (11)	0.0081 (10)	0.0113 (11)	0.0005 (9)	−0.0003 (9)	−0.0019 (8)
C22	0.0153 (12)	0.0093 (11)	0.0109 (11)	−0.0002 (9)	−0.0014 (9)	−0.0022 (9)
O23	0.0224 (10)	0.0128 (8)	0.0119 (8)	0.0088 (7)	−0.0039 (7)	−0.0020 (7)
C23	0.0132 (11)	0.0084 (11)	0.0112 (11)	0.0009 (9)	−0.0021 (9)	−0.0002 (8)
O24	0.0202 (9)	0.0122 (8)	0.0110 (8)	0.0094 (7)	−0.0019 (7)	−0.0038 (7)
C24	0.0121 (11)	0.0082 (11)	0.0123 (11)	0.0023 (9)	0.0003 (9)	−0.0037 (9)
O25	0.0257 (10)	0.0152 (9)	0.0096 (8)	0.0106 (7)	−0.0035 (7)	−0.0048 (7)
C25	0.0147 (11)	0.0093 (11)	0.0088 (11)	0.0006 (9)	0.0005 (9)	−0.0032 (8)
C26	0.0131 (11)	0.0080 (10)	0.0096 (11)	0.0012 (9)	−0.0011 (9)	−0.0008 (8)

Geometric parameters (Å, º)

N1—C1	1.472 (3)	O14—C14	1.266 (3)
N1—C2	1.406 (3)	C14—C20	1.430 (3)
N1—C6	1.397 (3)	O15—H15	0.8400
C1—H1A	0.9800	O15—C15	1.358 (3)
C1—H1B	0.9800	C15—C16	1.368 (4)
C1—H1C	0.9800	C15—C20	1.420 (3)
O2—C2	1.216 (3)	C16—H16	0.9500
C2—N3	1.374 (4)	C16—C17	1.416 (3)
N3—C3	1.469 (4)	O17—H17	0.8400
N3—C4	1.359 (4)	O17—C17	1.347 (3)
C3—H3A	0.9800	C17—C18	1.384 (3)
C3—H3B	0.9800	C18—H18	0.9500
C3—H3C	0.9800	C18—C19	1.382 (3)
C4—C5	1.382 (4)	C19—C20	1.406 (3)
C4—N9	1.361 (4)	C21—C22	1.401 (3)
C5—C6	1.413 (4)	C21—C26	1.411 (3)
C5—N7	1.378 (3)	C22—H22	0.9500
O6—C6	1.242 (3)	C22—C23	1.381 (3)
N7—H7	0.8800	O23—H23	0.8400
N7—C8	1.346 (3)	O23—C23	1.360 (3)
Br8—C8	1.852 (3)	C23—C24	1.400 (3)
C8—N9	1.321 (4)	O24—H24	0.8400
O11—C12	1.369 (3)	O24—C24	1.367 (3)
O11—C19	1.359 (3)	C24—C25	1.397 (3)
C12—C13	1.366 (3)	O25—H25	0.8400
C12—C21	1.463 (3)	O25—C25	1.372 (3)
O13—H13	0.8400	C25—C26	1.389 (3)
O13—C13	1.362 (3)	C26—H26	0.9500

C13—C14	1.441 (3)		
C2—N1—C1	115.4 (2)	O14—C14—C13	120.5 (2)
C6—N1—C1	118.7 (2)	O14—C14—C20	123.0 (2)
C6—N1—C2	125.9 (2)	C20—C14—C13	116.4 (2)
N1—C1—H1A	109.5	C15—O15—H15	109.5
N1—C1—H1B	109.5	O15—C15—C16	119.4 (2)
N1—C1—H1C	109.5	O15—C15—C20	119.4 (2)
H1A—C1—H1B	109.5	C16—C15—C20	121.2 (2)
H1A—C1—H1C	109.5	C15—C16—H16	120.5
H1B—C1—H1C	109.5	C15—C16—C17	119.1 (2)
O2—C2—N1	120.3 (2)	C17—C16—H16	120.5
O2—C2—N3	122.4 (3)	C17—O17—H17	109.5
N3—C2—N1	117.3 (2)	O17—C17—C16	120.6 (2)
C2—N3—C3	119.1 (2)	O17—C17—C18	117.8 (2)
C4—N3—C2	119.7 (2)	C18—C17—C16	121.5 (2)
C4—N3—C3	121.1 (2)	C17—C18—H18	120.9
N3—C3—H3A	109.5	C19—C18—C17	118.2 (2)
N3—C3—H3B	109.5	C19—C18—H18	120.9
N3—C3—H3C	109.5	O11—C19—C18	117.0 (2)
H3A—C3—H3B	109.5	O11—C19—C20	120.5 (2)
H3A—C3—H3C	109.5	C18—C19—C20	122.5 (2)
H3B—C3—H3C	109.5	C15—C20—C14	122.8 (2)
N3—C4—C5	122.0 (2)	C19—C20—C14	119.7 (2)
N3—C4—N9	125.7 (2)	C19—C20—C15	117.4 (2)
N9—C4—C5	112.2 (2)	C22—C21—C12	118.9 (2)
C4—C5—C6	122.2 (2)	C22—C21—C26	119.5 (2)
N7—C5—C4	105.0 (2)	C26—C21—C12	121.6 (2)
N7—C5—C6	132.5 (2)	C21—C22—H22	119.6
N1—C6—C5	112.7 (2)	C23—C22—C21	120.9 (2)
O6—C6—N1	120.8 (2)	C23—C22—H22	119.6
O6—C6—C5	126.5 (2)	C23—O23—H23	109.5
C5—N7—H7	127.4	C22—C23—C24	119.9 (2)
C8—N7—C5	105.2 (2)	O23—C23—C22	118.7 (2)
C8—N7—H7	127.4	O23—C23—C24	121.4 (2)
N7—C8—Br8	122.4 (2)	C24—O24—H24	109.5
N9—C8—N7	115.3 (2)	O24—C24—C23	117.4 (2)
N9—C8—Br8	122.3 (2)	O24—C24—C25	123.1 (2)
C8—N9—C4	102.3 (2)	C25—C24—C23	119.5 (2)
C19—O11—C12	121.64 (19)	C25—O25—H25	109.5
O11—C12—C21	111.5 (2)	O25—C25—C24	114.9 (2)
C13—C12—O11	120.3 (2)	O25—C25—C26	124.1 (2)
C13—C12—C21	128.2 (2)	C26—C25—C24	121.1 (2)
C13—O13—H13	109.5	C21—C26—H26	120.4
C12—C13—C14	121.3 (2)	C25—C26—C21	119.1 (2)
O13—C13—C12	121.1 (2)	C25—C26—H26	120.4
O13—C13—C14	117.7 (2)		

N1—C2—N3—C3	179.5 (3)	C12—C21—C22—C23	179.8 (2)
N1—C2—N3—C4	−0.2 (4)	C12—C21—C26—C25	−177.9 (2)
C1—N1—C2—O2	−0.2 (4)	O13—C13—C14—O14	2.9 (3)
C1—N1—C2—N3	179.4 (2)	O13—C13—C14—C20	−174.1 (2)
C1—N1—C6—C5	179.0 (2)	C13—C12—C21—C22	156.8 (3)
C1—N1—C6—O6	−2.7 (4)	C13—C12—C21—C26	−25.1 (4)
O2—C2—N3—C3	−1.0 (4)	C13—C14—C20—C15	174.8 (2)
O2—C2—N3—C4	179.3 (3)	C13—C14—C20—C19	−4.6 (3)
C2—N1—C6—C5	−2.9 (4)	O14—C14—C20—C15	−2.1 (4)
C2—N1—C6—O6	175.4 (2)	O14—C14—C20—C19	178.5 (2)
C2—N3—C4—C5	1.1 (4)	O15—C15—C16—C17	−179.6 (2)
C2—N3—C4—N9	−176.5 (3)	O15—C15—C20—C14	−1.9 (4)
N3—C4—C5—C6	−3.1 (4)	O15—C15—C20—C19	177.5 (2)
N3—C4—C5—N7	−177.5 (2)	C15—C16—C17—O17	−178.8 (2)
N3—C4—N9—C8	177.9 (3)	C15—C16—C17—C18	2.2 (4)
C3—N3—C4—C5	−178.6 (3)	C16—C15—C20—C14	179.1 (2)
C3—N3—C4—N9	3.8 (4)	C16—C15—C20—C19	−1.5 (4)
C4—C5—C6—N1	3.7 (4)	C16—C17—C18—C19	−1.5 (4)
C4—C5—C6—O6	−174.5 (3)	O17—C17—C18—C19	179.5 (2)
C4—C5—N7—C8	−0.8 (3)	C17—C18—C19—O11	179.9 (2)
C5—C4—N9—C8	0.1 (3)	C17—C18—C19—C20	−0.8 (4)
C5—N7—C8—Br8	−177.87 (19)	C18—C19—C20—C14	−178.3 (2)
C5—N7—C8—N9	1.0 (3)	C18—C19—C20—C15	2.3 (4)
C6—N1—C2—O2	−178.3 (2)	C19—O11—C12—C13	−1.3 (3)
C6—N1—C2—N3	1.3 (4)	C19—O11—C12—C21	177.9 (2)
C6—C5—N7—C8	−174.4 (3)	C20—C15—C16—C17	−0.6 (4)
N7—C5—C6—N1	176.4 (3)	C21—C12—C13—O13	−2.2 (4)
N7—C5—C6—O6	−1.8 (5)	C21—C12—C13—C14	178.2 (2)
N7—C8—N9—C4	−0.7 (3)	C21—C22—C23—O23	177.9 (2)
Br8—C8—N9—C4	178.14 (19)	C21—C22—C23—C24	−1.1 (4)
N9—C4—C5—C6	174.8 (2)	C22—C21—C26—C25	0.2 (4)
N9—C4—C5—N7	0.4 (3)	C22—C23—C24—O24	178.8 (2)
O11—C12—C13—O13	176.9 (2)	C22—C23—C24—C25	−1.2 (4)
O11—C12—C13—C14	−2.7 (4)	O23—C23—C24—O24	−0.2 (4)
O11—C12—C21—C22	−22.4 (3)	O23—C23—C24—C25	179.8 (2)
O11—C12—C21—C26	155.7 (2)	C23—C24—C25—O25	−177.4 (2)
O11—C19—C20—C14	0.9 (4)	C23—C24—C25—C26	3.1 (4)
O11—C19—C20—C15	−178.5 (2)	O24—C24—C25—O25	2.5 (4)
C12—O11—C19—C18	−178.5 (2)	O24—C24—C25—C26	−176.9 (2)
C12—O11—C19—C20	2.2 (3)	C24—C25—C26—C21	−2.6 (4)
C12—C13—C14—O14	−177.5 (2)	O25—C25—C26—C21	178.0 (2)
C12—C13—C14—C20	5.5 (3)	C26—C21—C22—C23	1.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O6^{i}	0.88	1.94	2.764 (3)	154
O13—H13···O6^{ii}	0.84	2.08	2.892 (3)	162

O15—H15···O14	0.84	1.87	2.623 (3)	148
O17—H17···O2	0.84	1.83	2.652 (3)	164
O23—H23···O15^{iii}	0.84	1.97	2.776 (3)	161
O24—H24···O25^{iv}	0.84	2.16	2.829 (3)	137
O25—H25···O14^{v}	0.84	1.90	2.707 (3)	161

Symmetry codes: (i) −*x*, −*y*+2, −*z*+2; (ii) *x*+1, *y*, *z*−1; (iii) *x*+1, *y*−1, *z*; (iv) −*x*+3, −*y*, −*z*; (v) −*x*+2, −*y*+1, −*z*.

8-Bromo-1,3-dimethyl-7*H*-purine-2,6-dione–\ 3,5,7-trihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one–\ methanol (1/1/1) (3a_prime_ywj75e_mo100k_auto)

Crystal data

$C_7H_7BrN_4O_2·C_{15}H_{10}O_6·CH_4O$
M_r = 577.35
Triclinic, $P\overline{1}$
a = 9.9847 (5) Å
b = 10.6290 (4) Å
c = 12.1537 (4) Å
α = 67.509 (4)°
β = 81.274 (3)°
γ = 73.675 (4)°
V = 1142.27 (9) Å^3
Z = 2
F(000) = 588
D_x = 1.679 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5099 reflections
θ = 2.6–29.4°
μ = 1.86 mm^{-1}
T = 100 K
Plate, yellow
0.3 × 0.2 × 0.1 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.3577 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2023)
T_{min} = 0.615, T_{max} = 1.000
7430 measured reflections
4532 independent reflections
4048 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.2°, θ_{min} = 2.7°
h = −10→12
k = −13→13
l = −15→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.066
S = 1.05
4532 reflections
358 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0292P)^2 + 0.1784P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 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}
Br8	1.11654 (2)	0.02429 (2)	−0.35283 (2)	0.01863 (7)
O2	0.83970 (14)	0.48202 (14)	0.00210 (11)	0.0166 (3)
O6	0.93850 (14)	0.61998 (14)	−0.40003 (11)	0.0143 (3)
N1	0.90362 (16)	0.54782 (16)	−0.19711 (13)	0.0114 (3)
N3	0.92455 (16)	0.30951 (16)	−0.07344 (13)	0.0113 (3)
N7	1.02727 (16)	0.30289 (17)	−0.36065 (14)	0.0111 (3)
H7	1.037 (2)	0.343 (2)	−0.440 (2)	0.026 (6)*
N9	1.01693 (16)	0.14336 (17)	−0.17544 (13)	0.0130 (3)
C1	0.8756 (2)	0.6934 (2)	−0.20379 (17)	0.0149 (4)
H1A	0.920977	0.747677	−0.276856	0.022*
H1B	0.912381	0.695403	−0.134356	0.022*
H1C	0.774514	0.734389	−0.204615	0.022*
C2	0.88648 (19)	0.4483 (2)	−0.08491 (16)	0.0112 (4)
C3	0.8923 (2)	0.2045 (2)	0.04003 (15)	0.0150 (4)
H3A	0.791646	0.227575	0.059684	0.022*
H3B	0.943263	0.204081	0.103069	0.022*
H3C	0.920342	0.111547	0.033338	0.022*
C4	0.97289 (19)	0.2756 (2)	−0.17358 (15)	0.0108 (4)
C5	0.97805 (19)	0.3762 (2)	−0.28415 (15)	0.0107 (4)
C6	0.94075 (19)	0.5218 (2)	−0.30441 (15)	0.0104 (4)
C8	1.0483 (2)	0.1672 (2)	−0.29070 (16)	0.0130 (4)
O1	0.61742 (13)	0.65416 (13)	0.50867 (10)	0.0114 (3)
O13	0.63675 (14)	0.37551 (15)	0.79999 (11)	0.0137 (3)
H13	0.652 (3)	0.292 (3)	0.817 (2)	0.036 (8)*
O14	0.71277 (14)	0.22705 (14)	0.65371 (11)	0.0157 (3)
O15	0.76914 (16)	0.21985 (15)	0.43815 (13)	0.0182 (3)
H15	0.764 (3)	0.197 (3)	0.503 (2)	0.038 (9)*
O17	0.71225 (15)	0.66086 (15)	0.11745 (11)	0.0176 (3)
H17	0.744 (2)	0.609 (3)	0.080 (2)	0.026 (7)*
O24	0.44251 (14)	0.98867 (14)	0.85334 (11)	0.0153 (3)
H24	0.420278	0.952132	0.925945	0.023*
C12	0.60988 (19)	0.5871 (2)	0.63070 (15)	0.0111 (4)
C13	0.64077 (19)	0.4444 (2)	0.67962 (16)	0.0109 (4)
C14	0.68414 (19)	0.3588 (2)	0.60589 (16)	0.0112 (4)
C15	0.73520 (19)	0.3614 (2)	0.39756 (16)	0.0118 (4)
C16	0.74224 (19)	0.4354 (2)	0.27721 (16)	0.0124 (4)
H16	0.770622	0.387439	0.222566	0.015*
C17	0.70701 (19)	0.5833 (2)	0.23544 (16)	0.0124 (4)
C18	0.66444 (19)	0.6572 (2)	0.31290 (16)	0.0116 (4)
H18	0.640347	0.756936	0.283937	0.014*
C19	0.65865 (18)	0.5793 (2)	0.43401 (15)	0.0099 (4)
C20	0.69271 (19)	0.4326 (2)	0.47999 (16)	0.0114 (4)
C21	0.56797 (19)	0.6887 (2)	0.69200 (16)	0.0111 (4)
C22	0.52574 (19)	0.6490 (2)	0.81327 (16)	0.0129 (4)
H22	0.524493	0.553830	0.858000	0.016*

C23	0.4857 (2)	0.7474 (2)	0.86873 (16)	0.0133 (4)
H23	0.458730	0.719119	0.951302	0.016*
C24	0.48494 (19)	0.8869 (2)	0.80368 (16)	0.0116 (4)
C25	0.5295 (2)	0.9277 (2)	0.68374 (16)	0.0142 (4)
H25	0.532081	1.022662	0.639782	0.017*
C26	0.5700 (2)	0.8295 (2)	0.62897 (16)	0.0135 (4)
H26	0.599721	0.858035	0.546997	0.016*
O1S	0.36466 (15)	0.90690 (14)	1.08470 (11)	0.0151 (3)
H1S	0.429 (3)	0.910 (3)	1.120 (2)	0.040 (8)*
C2S	0.2464 (2)	1.0199 (2)	1.09085 (18)	0.0192 (4)
H2SA	0.171479	1.025029	1.044579	0.029*
H2SB	0.274730	1.108831	1.057990	0.029*
H2SC	0.212850	1.002421	1.174170	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br8	0.03113 (14)	0.01030 (11)	0.01296 (11)	−0.00209 (9)	0.00381 (8)	−0.00645 (8)
O2	0.0240 (8)	0.0155 (8)	0.0093 (7)	−0.0018 (6)	0.0034 (5)	−0.0071 (6)
O6	0.0218 (8)	0.0096 (7)	0.0097 (7)	−0.0033 (6)	−0.0001 (5)	−0.0022 (6)
N1	0.0161 (9)	0.0084 (8)	0.0090 (8)	−0.0011 (7)	0.0002 (6)	−0.0038 (7)
N3	0.0156 (9)	0.0106 (9)	0.0061 (7)	−0.0017 (7)	0.0017 (6)	−0.0033 (6)
N7	0.0153 (9)	0.0090 (8)	0.0091 (8)	−0.0025 (7)	0.0018 (6)	−0.0044 (7)
N9	0.0170 (9)	0.0104 (9)	0.0108 (8)	−0.0020 (7)	0.0018 (6)	−0.0048 (7)
C1	0.0227 (11)	0.0099 (10)	0.0139 (9)	−0.0028 (8)	0.0004 (8)	−0.0075 (8)
C2	0.0114 (10)	0.0113 (10)	0.0098 (9)	−0.0009 (8)	−0.0015 (7)	−0.0035 (8)
C3	0.0216 (11)	0.0111 (10)	0.0093 (9)	−0.0031 (8)	0.0013 (8)	−0.0019 (8)
C4	0.0100 (10)	0.0107 (10)	0.0113 (9)	−0.0004 (8)	−0.0010 (7)	−0.0049 (8)
C5	0.0120 (10)	0.0124 (10)	0.0082 (9)	−0.0031 (8)	0.0018 (7)	−0.0050 (8)
C6	0.0094 (10)	0.0135 (10)	0.0090 (9)	−0.0027 (8)	0.0005 (7)	−0.0052 (8)
C8	0.0173 (10)	0.0095 (10)	0.0124 (9)	−0.0029 (8)	0.0009 (7)	−0.0049 (8)
O1	0.0178 (7)	0.0082 (7)	0.0078 (6)	−0.0019 (6)	0.0003 (5)	−0.0037 (5)
O13	0.0226 (8)	0.0082 (8)	0.0085 (6)	−0.0023 (6)	0.0001 (5)	−0.0024 (6)
O14	0.0240 (8)	0.0082 (7)	0.0120 (7)	−0.0005 (6)	−0.0006 (5)	−0.0029 (6)
O15	0.0329 (9)	0.0094 (7)	0.0096 (7)	−0.0015 (6)	0.0010 (6)	−0.0039 (6)
O17	0.0312 (9)	0.0131 (8)	0.0071 (7)	−0.0037 (6)	0.0035 (6)	−0.0049 (6)
O24	0.0247 (8)	0.0113 (7)	0.0110 (7)	−0.0030 (6)	0.0025 (6)	−0.0073 (6)
C12	0.0115 (10)	0.0137 (10)	0.0086 (9)	−0.0042 (8)	0.0002 (7)	−0.0040 (8)
C13	0.0112 (10)	0.0128 (10)	0.0088 (9)	−0.0041 (8)	0.0001 (7)	−0.0034 (8)
C14	0.0090 (10)	0.0110 (10)	0.0138 (9)	−0.0014 (8)	−0.0014 (7)	−0.0051 (8)
C15	0.0111 (10)	0.0087 (10)	0.0164 (9)	−0.0017 (8)	0.0000 (7)	−0.0060 (8)
C16	0.0139 (10)	0.0132 (10)	0.0128 (9)	−0.0037 (8)	0.0029 (7)	−0.0086 (8)
C17	0.0126 (10)	0.0151 (10)	0.0096 (9)	−0.0042 (8)	0.0007 (7)	−0.0042 (8)
C18	0.0127 (10)	0.0096 (10)	0.0127 (9)	−0.0026 (8)	0.0002 (7)	−0.0045 (8)
C19	0.0091 (9)	0.0123 (10)	0.0107 (9)	−0.0022 (8)	0.0011 (7)	−0.0074 (8)
C20	0.0104 (10)	0.0129 (10)	0.0124 (9)	−0.0024 (8)	0.0006 (7)	−0.0069 (8)
C21	0.0108 (10)	0.0110 (10)	0.0124 (9)	−0.0017 (8)	−0.0014 (7)	−0.0057 (8)
C22	0.0163 (10)	0.0103 (10)	0.0134 (9)	−0.0046 (8)	−0.0001 (7)	−0.0049 (8)

C23	0.0155 (10)	0.0144 (10)	0.0104 (9)	−0.0033 (8)	0.0006 (7)	−0.0054 (8)
C24	0.0123 (10)	0.0113 (10)	0.0135 (9)	0.0004 (8)	−0.0026 (7)	−0.0085 (8)
C25	0.0196 (11)	0.0081 (10)	0.0136 (9)	−0.0024 (8)	−0.0023 (8)	−0.0028 (8)
C26	0.0179 (10)	0.0127 (10)	0.0094 (9)	−0.0029 (8)	−0.0012 (7)	−0.0038 (8)
O1S	0.0204 (8)	0.0114 (7)	0.0143 (7)	−0.0036 (6)	0.0004 (6)	−0.0061 (6)
C2S	0.0225 (11)	0.0119 (11)	0.0215 (10)	−0.0012 (9)	0.0026 (8)	−0.0076 (9)

Geometric parameters (Å, º)

Br8—C8	1.8655 (18)	O17—C17	1.355 (2)
O2—C2	1.229 (2)	O24—H24	0.8400
O6—C6	1.228 (2)	O24—C24	1.371 (2)
N1—C1	1.465 (2)	C12—C13	1.362 (3)
N1—C2	1.391 (2)	C12—C21	1.471 (2)
N1—C6	1.409 (2)	C13—C14	1.450 (2)
N3—C2	1.372 (2)	C14—C20	1.430 (3)
N3—C3	1.463 (2)	C15—C16	1.372 (3)
N3—C4	1.380 (2)	C15—C20	1.424 (2)
N7—H7	0.90 (2)	C16—H16	0.9500
N7—C5	1.383 (2)	C16—C17	1.409 (3)
N7—C8	1.339 (2)	C17—C18	1.393 (3)
N9—C4	1.358 (2)	C18—H18	0.9500
N9—C8	1.328 (2)	C18—C19	1.387 (3)
C1—H1A	0.9800	C19—C20	1.397 (3)
C1—H1B	0.9800	C21—C22	1.401 (3)
C1—H1C	0.9800	C21—C26	1.401 (3)
C3—H3A	0.9800	C22—H22	0.9500
C3—H3B	0.9800	C22—C23	1.390 (3)
C3—H3C	0.9800	C23—H23	0.9500
C4—C5	1.365 (3)	C23—C24	1.386 (3)
C5—C6	1.417 (3)	C24—C25	1.392 (3)
O1—C12	1.379 (2)	C25—H25	0.9500
O1—C19	1.371 (2)	C25—C26	1.382 (3)
O13—H13	0.80 (3)	C26—H26	0.9500
O13—C13	1.363 (2)	O1S—H1S	0.83 (3)
O14—C14	1.258 (2)	O1S—C2S	1.444 (2)
O15—H15	0.73 (3)	C2S—H2SA	0.9800
O15—C15	1.348 (2)	C2S—H2SB	0.9800
O17—H17	0.81 (2)	C2S—H2SC	0.9800
C2—N1—C1	116.42 (14)	C12—C13—C14	121.38 (17)
C2—N1—C6	125.97 (16)	O14—C14—C13	119.93 (16)
C6—N1—C1	117.57 (15)	O14—C14—C20	123.73 (16)
C2—N3—C3	118.73 (15)	C20—C14—C13	116.34 (17)
C2—N3—C4	118.88 (16)	O15—C15—C16	119.69 (16)
C4—N3—C3	121.69 (15)	O15—C15—C20	119.69 (17)
C5—N7—H7	124.3 (15)	C16—C15—C20	120.61 (17)
C8—N7—H7	130.4 (15)	C15—C16—H16	120.3

C8—N7—C5	105.17 (15)	C15—C16—C17	119.39 (16)
C8—N9—C4	102.09 (15)	C17—C16—H16	120.3
N1—C1—H1A	109.5	O17—C17—C16	121.50 (16)
N1—C1—H1B	109.5	O17—C17—C18	116.61 (17)
N1—C1—H1C	109.5	C18—C17—C16	121.89 (17)
H1A—C1—H1B	109.5	C17—C18—H18	121.3
H1A—C1—H1C	109.5	C19—C18—C17	117.34 (18)
H1B—C1—H1C	109.5	C19—C18—H18	121.3
O2—C2—N1	121.71 (17)	O1—C19—C18	116.37 (16)
O2—C2—N3	120.41 (17)	O1—C19—C20	120.63 (16)
N3—C2—N1	117.89 (15)	C18—C19—C20	123.01 (16)
N3—C3—H3A	109.5	C15—C20—C14	122.09 (17)
N3—C3—H3B	109.5	C19—C20—C14	120.14 (16)
N3—C3—H3C	109.5	C19—C20—C15	117.77 (16)
H3A—C3—H3B	109.5	C22—C21—C12	121.84 (17)
H3A—C3—H3C	109.5	C26—C21—C12	119.93 (16)
H3B—C3—H3C	109.5	C26—C21—C22	118.23 (16)
N9—C4—N3	125.42 (17)	C21—C22—H22	119.7
N9—C4—C5	112.67 (16)	C23—C22—C21	120.61 (18)
C5—C4—N3	121.89 (17)	C23—C22—H22	119.7
N7—C5—C6	131.85 (17)	C22—C23—H23	120.0
C4—C5—N7	105.09 (16)	C24—C23—C22	120.07 (16)
C4—C5—C6	123.05 (16)	C24—C23—H23	120.0
O6—C6—N1	120.19 (17)	O24—C24—C23	122.33 (16)
O6—C6—C5	128.02 (16)	O24—C24—C25	117.59 (17)
N1—C6—C5	111.80 (16)	C23—C24—C25	120.08 (16)
N7—C8—Br8	121.70 (13)	C24—C25—H25	120.1
N9—C8—Br8	123.33 (14)	C26—C25—C24	119.70 (18)
N9—C8—N7	114.98 (16)	C26—C25—H25	120.1
C19—O1—C12	121.11 (15)	C21—C26—H26	119.4
C13—O13—H13	111.6 (17)	C25—C26—C21	121.26 (17)
C15—O15—H15	106 (2)	C25—C26—H26	119.4
C17—O17—H17	109.7 (17)	C2S—O1S—H1S	107.1 (18)
C24—O24—H24	109.5	O1S—C2S—H2SA	109.5
O1—C12—C21	111.26 (16)	O1S—C2S—H2SB	109.5
C13—C12—O1	120.39 (16)	O1S—C2S—H2SC	109.5
C13—C12—C21	128.35 (17)	H2SA—C2S—H2SB	109.5
O13—C13—C14	117.05 (16)	H2SA—C2S—H2SC	109.5
C12—C13—O13	121.54 (16)	H2SB—C2S—H2SC	109.5
N3—C4—C5—N7	−178.19 (16)	O13—C13—C14—C20	−177.79 (16)
N3—C4—C5—C6	3.0 (3)	O14—C14—C20—C15	0.0 (3)
N7—C5—C6—O6	3.4 (3)	O14—C14—C20—C19	179.82 (18)
N7—C5—C6—N1	−176.32 (18)	O15—C15—C16—C17	−179.82 (17)
N9—C4—C5—N7	0.5 (2)	O15—C15—C20—C14	−0.2 (3)
N9—C4—C5—C6	−178.34 (17)	O15—C15—C20—C19	179.93 (17)
C1—N1—C2—O2	5.6 (3)	O17—C17—C18—C19	−179.94 (16)
C1—N1—C2—N3	−173.95 (16)	O24—C24—C25—C26	−178.40 (17)

C1—N1—C6—O6	−5.5 (3)	C12—O1—C19—C18	−179.13 (15)
C1—N1—C6—C5	174.31 (16)	C12—O1—C19—C20	0.7 (3)
C2—N1—C6—O6	172.27 (17)	C12—C13—C14—O14	179.59 (18)
C2—N1—C6—C5	−7.9 (3)	C12—C13—C14—C20	0.1 (3)
C2—N3—C4—N9	178.52 (17)	C12—C21—C22—C23	−179.52 (17)
C2—N3—C4—C5	−2.9 (3)	C12—C21—C26—C25	179.18 (18)
C3—N3—C2—O2	7.6 (3)	C13—C12—C21—C22	−13.8 (3)
C3—N3—C2—N1	−172.89 (16)	C13—C12—C21—C26	166.01 (19)
C3—N3—C4—N9	−11.2 (3)	C13—C14—C20—C15	179.40 (17)
C3—N3—C4—C5	167.36 (17)	C13—C14—C20—C19	−0.7 (3)
C4—N3—C2—O2	178.16 (16)	C15—C16—C17—O17	−179.93 (17)
C4—N3—C2—N1	−2.3 (3)	C15—C16—C17—C18	−0.3 (3)
C4—N9—C8—Br8	179.92 (14)	C16—C15—C20—C14	179.96 (17)
C4—N9—C8—N7	−0.1 (2)	C16—C15—C20—C19	0.1 (3)
C4—C5—C6—O6	−178.04 (18)	C16—C17—C18—C19	0.4 (3)
C4—C5—C6—N1	2.2 (3)	C17—C18—C19—O1	179.57 (15)
C5—N7—C8—Br8	−179.60 (13)	C17—C18—C19—C20	−0.3 (3)
C5—N7—C8—N9	0.4 (2)	C18—C19—C20—C14	−179.84 (17)
C6—N1—C2—O2	−172.19 (17)	C18—C19—C20—C15	0.0 (3)
C6—N1—C2—N3	8.3 (3)	C19—O1—C12—C13	−1.3 (3)
C8—N7—C5—C4	−0.6 (2)	C19—O1—C12—C21	178.07 (15)
C8—N7—C5—C6	178.2 (2)	C20—C15—C16—C17	0.0 (3)
C8—N9—C4—N3	178.40 (17)	C21—C12—C13—O13	−0.6 (3)
C8—N9—C4—C5	−0.3 (2)	C21—C12—C13—C14	−178.39 (17)
O1—C12—C13—O13	178.71 (15)	C21—C22—C23—C24	1.1 (3)
O1—C12—C13—C14	0.9 (3)	C22—C21—C26—C25	−1.0 (3)
O1—C12—C21—C22	166.84 (16)	C22—C23—C24—O24	178.09 (17)
O1—C12—C21—C26	−13.3 (2)	C22—C23—C24—C25	−2.5 (3)
O1—C19—C20—C14	0.3 (3)	C23—C24—C25—C26	2.2 (3)
O1—C19—C20—C15	−179.79 (15)	C24—C25—C26—C21	−0.4 (3)
O13—C13—C14—O14	1.7 (3)	C26—C21—C22—C23	0.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O6^{i}	0.90 (2)	1.82 (2)	2.6982 (19)	165 (2)
O13—H13···O1*S*^{ii}	0.80 (3)	2.03 (3)	2.788 (2)	157 (2)
O15—H15···O14	0.73 (3)	1.95 (3)	2.6208 (18)	154 (3)
O17—H17···O2	0.81 (2)	1.90 (2)	2.7085 (18)	173 (2)
O24—H24···O1*S*	0.84	1.84	2.6670 (17)	169
O1*S*—H1*S*···O24^{iii}	0.83 (3)	2.03 (3)	2.791 (2)	152 (3)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*−1; (ii) −*x*+1, −*y*+1, −*z*+2; (iii) −*x*+1, −*y*+2, −*z*+2.

3,5,7-Trihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one monohydrate (aH2O-ywj219_ga100k)

Crystal data

$C_{15}H_{10}O_6 \cdot H_2O$
$M_r = 304.25$
Monoclinic, $C2/c$
$a = 27.7113$ (11) Å
$b = 3.7151$ (2) Å
$c = 24.7282$ (11) Å
$\beta = 98.208$ (2)°
$V = 2519.7$ (2) Å^3
$Z = 8$
$F(000) = 1264$
$D_x = 1.604$ Mg m^{-3}
Ga $K\alpha$ radiation, $\lambda = 1.34139$ Å
Cell parameters from 3925 reflections
$\theta = 3.1$–$57.0°$
$\mu = 0.70$ mm^{-1}
$T = 100$ K
Plate, colourless
0.03 × 0.03 × 0.03 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.658$, $T_{max} = 0.751$
11000 measured reflections
2557 independent reflections
1890 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 57.1°$, $\theta_{min} = 2.8°$
$h = -34 \rightarrow 34$
$k = -4 \rightarrow 4$
$l = -30 \rightarrow 30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 1.06$
2557 reflections
224 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 1.2901P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: SHELXL2019 (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0011 (2)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.58690 (4)	0.6718 (4)	0.34033 (5)	0.0335 (3)
C2	0.57465 (5)	0.5485 (5)	0.38888 (7)	0.0293 (4)
O3	0.59658 (4)	0.4356 (4)	0.48301 (5)	0.0412 (4)
H3	0.6254 (9)	0.381 (7)	0.5100 (10)	0.071 (7)*
C3	0.60818 (5)	0.5532 (5)	0.43461 (7)	0.0310 (4)
O4	0.68735 (4)	0.7029 (4)	0.47689 (5)	0.0448 (4)
C4	0.65677 (5)	0.6936 (5)	0.43342 (7)	0.0323 (4)
O5	0.74976 (4)	0.9812 (4)	0.41816 (5)	0.0443 (4)
H5	0.7370 (10)	0.880 (7)	0.4468 (11)	0.080 (9)*

C5	0.71407 (5)	0.9602 (5)	0.37437 (7)	0.0313 (4)
C6	0.72268 (5)	1.0775 (5)	0.32454 (7)	0.0309 (4)
H6	0.753554	1.175666	0.320214	0.037*
O7	0.69644 (4)	1.1721 (4)	0.23138 (5)	0.0387 (4)
H7	0.6693 (9)	1.156 (7)	0.2059 (10)	0.068 (7)*
C7	0.68598 (6)	1.0531 (5)	0.27980 (7)	0.0320 (4)
C8	0.64032 (6)	0.9152 (5)	0.28529 (7)	0.0350 (4)
H8	0.615375	0.899677	0.254754	0.042*
C9	0.63225 (5)	0.8018 (5)	0.33623 (7)	0.0297 (4)
C10	0.66777 (5)	0.8168 (5)	0.38221 (7)	0.0289 (4)
C11	0.52368 (5)	0.4282 (5)	0.38199 (7)	0.0296 (4)
C12	0.49528 (6)	0.4614 (5)	0.33073 (7)	0.0356 (4)
H12	0.509611	0.554955	0.300993	0.043*
C13	0.44697 (6)	0.3611 (6)	0.32254 (7)	0.0398 (5)
H13	0.428270	0.387768	0.287464	0.048*
O14	0.37744 (4)	0.1259 (4)	0.35477 (6)	0.0463 (4)
H14	0.3670 (9)	0.038 (7)	0.3829 (10)	0.064 (8)*
C14	0.42553 (5)	0.2214 (6)	0.36530 (7)	0.0365 (4)
C15	0.45285 (6)	0.1836 (5)	0.41624 (7)	0.0352 (4)
H15	0.438335	0.086564	0.445601	0.042*
C16	0.50138 (6)	0.2870 (5)	0.42460 (7)	0.0351 (4)
H16	0.519796	0.261439	0.459861	0.042*
O1W	0.33632 (4)	−0.1296 (5)	0.43835 (5)	0.0435 (4)
H1WA	0.3282 (10)	0.033 (9)	0.4619 (12)	0.087 (10)*
H1WB	0.3115 (10)	−0.254 (8)	0.4281 (11)	0.071 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0185 (5)	0.0563 (8)	0.0270 (6)	−0.0092 (5)	0.0082 (4)	−0.0009 (6)
C2	0.0207 (7)	0.0406 (10)	0.0286 (9)	−0.0055 (7)	0.0107 (6)	−0.0054 (8)
O3	0.0245 (6)	0.0757 (10)	0.0248 (6)	−0.0184 (6)	0.0077 (5)	−0.0021 (6)
C3	0.0227 (7)	0.0466 (11)	0.0256 (9)	−0.0093 (7)	0.0105 (6)	−0.0070 (8)
O4	0.0260 (5)	0.0836 (10)	0.0254 (7)	−0.0221 (6)	0.0060 (5)	−0.0062 (7)
C4	0.0231 (7)	0.0478 (11)	0.0272 (9)	−0.0096 (7)	0.0073 (6)	−0.0093 (8)
O5	0.0244 (6)	0.0808 (11)	0.0283 (7)	−0.0235 (6)	0.0059 (5)	−0.0060 (7)
C5	0.0215 (7)	0.0442 (11)	0.0297 (9)	−0.0075 (7)	0.0090 (6)	−0.0079 (8)
C6	0.0207 (7)	0.0415 (10)	0.0330 (10)	−0.0068 (7)	0.0128 (6)	−0.0079 (8)
O7	0.0232 (5)	0.0643 (9)	0.0304 (7)	−0.0037 (6)	0.0107 (5)	0.0048 (6)
C7	0.0254 (7)	0.0428 (11)	0.0304 (10)	−0.0011 (7)	0.0128 (7)	−0.0014 (8)
C8	0.0206 (7)	0.0559 (12)	0.0294 (9)	−0.0041 (7)	0.0067 (6)	−0.0021 (8)
C9	0.0175 (7)	0.0407 (10)	0.0328 (9)	−0.0051 (6)	0.0103 (6)	−0.0060 (8)
C10	0.0212 (7)	0.0392 (10)	0.0283 (9)	−0.0074 (7)	0.0105 (6)	−0.0074 (8)
C11	0.0195 (7)	0.0402 (10)	0.0303 (9)	−0.0061 (7)	0.0075 (6)	−0.0065 (8)
C12	0.0224 (7)	0.0532 (12)	0.0326 (10)	−0.0068 (7)	0.0088 (7)	0.0001 (9)
C13	0.0229 (8)	0.0639 (13)	0.0322 (10)	−0.0077 (8)	0.0028 (7)	0.0030 (9)
O14	0.0196 (6)	0.0854 (11)	0.0340 (8)	−0.0170 (6)	0.0047 (5)	0.0037 (7)
C14	0.0176 (7)	0.0557 (12)	0.0375 (10)	−0.0084 (7)	0.0079 (7)	−0.0039 (9)

C15	0.0240 (7)	0.0553 (12)	0.0282 (9)	−0.0105 (7)	0.0108 (7)	−0.0058 (8)
C16	0.0229 (7)	0.0571 (12)	0.0264 (9)	−0.0089 (7)	0.0073 (6)	−0.0065 (8)
O1W	0.0253 (6)	0.0756 (11)	0.0312 (7)	−0.0222 (7)	0.0090 (5)	−0.0085 (7)

Geometric parameters (Å, º)

O1—C2	1.372 (2)	C8—H8	0.9500
O1—C9	1.3636 (17)	C8—C9	1.376 (2)
C2—C3	1.357 (2)	C9—C10	1.395 (2)
C2—C11	1.468 (2)	C11—C12	1.399 (2)
O3—H3	0.99 (3)	C11—C16	1.398 (2)
O3—C3	1.355 (2)	C12—H12	0.9500
C3—C4	1.448 (2)	C12—C13	1.376 (2)
O4—C4	1.271 (2)	C13—H13	0.9500
C4—C10	1.420 (2)	C13—C14	1.385 (3)
O5—H5	0.92 (3)	O14—H14	0.85 (3)
O5—C5	1.3604 (19)	O14—C14	1.3676 (18)
C5—C6	1.360 (2)	C14—C15	1.381 (2)
C5—C10	1.428 (2)	C15—H15	0.9500
C6—H6	0.9500	C15—C16	1.386 (2)
C6—C7	1.395 (2)	C16—H16	0.9500
O7—H7	0.91 (3)	O1W—H1WA	0.89 (3)
O7—C7	1.346 (2)	O1W—H1WB	0.84 (3)
C7—C8	1.389 (2)		

C9—O1—C2	121.95 (12)	O1—C9—C10	120.28 (15)
O1—C2—C11	110.75 (13)	C8—C9—C10	123.09 (14)
C3—C2—O1	119.94 (13)	C4—C10—C5	123.55 (15)
C3—C2—C11	129.31 (15)	C9—C10—C4	119.86 (14)
C3—O3—H3	113.2 (15)	C9—C10—C5	116.59 (15)
C2—C3—C4	121.04 (15)	C12—C11—C2	119.07 (15)
O3—C3—C2	120.73 (14)	C16—C11—C2	123.25 (15)
O3—C3—C4	118.21 (14)	C16—C11—C12	117.66 (14)
O4—C4—C3	120.14 (15)	C11—C12—H12	119.4
O4—C4—C10	122.93 (14)	C13—C12—C11	121.26 (16)
C10—C4—C3	116.92 (14)	C13—C12—H12	119.4
C5—O5—H5	106.0 (17)	C12—C13—H13	119.9
O5—C5—C10	118.59 (15)	C12—C13—C14	120.22 (16)
C6—C5—O5	120.09 (14)	C14—C13—H13	119.9
C6—C5—C10	121.32 (15)	C14—O14—H14	112.2 (16)
C5—C6—H6	120.1	O14—C14—C13	117.67 (16)
C5—C6—C7	119.71 (14)	O14—C14—C15	122.66 (16)
C7—C6—H6	120.1	C15—C14—C13	119.67 (14)
C7—O7—H7	109.4 (16)	C14—C15—H15	119.9
O7—C7—C6	117.31 (14)	C14—C15—C16	120.14 (16)
O7—C7—C8	121.53 (15)	C16—C15—H15	119.9
C8—C7—C6	121.16 (16)	C11—C16—H16	119.5
C7—C8—H8	120.9	C15—C16—C11	121.04 (16)

C9—C8—C7	118.13 (15)	C15—C16—H16	119.5
C9—C8—H8	120.9	H1WA—O1W—H1WB	107 (2)
O1—C9—C8	116.64 (14)		

O1—C2—C3—O3	179.38 (15)	C5—C6—C7—O7	−179.82 (16)
O1—C2—C3—C4	1.2 (3)	C5—C6—C7—C8	0.9 (3)
O1—C2—C11—C12	−1.8 (2)	C6—C5—C10—C4	−179.46 (17)
O1—C2—C11—C16	179.53 (16)	C6—C5—C10—C9	0.4 (3)
O1—C9—C10—C4	0.5 (3)	C6—C7—C8—C9	−0.2 (3)
O1—C9—C10—C5	−179.34 (15)	O7—C7—C8—C9	−179.44 (16)
C2—O1—C9—C8	179.52 (16)	C7—C8—C9—O1	179.26 (16)
C2—O1—C9—C10	−0.8 (3)	C7—C8—C9—C10	−0.4 (3)
C2—C3—C4—O4	178.50 (18)	C8—C9—C10—C4	−179.80 (18)
C2—C3—C4—C10	−1.4 (3)	C8—C9—C10—C5	0.3 (3)
C2—C11—C12—C13	−178.32 (18)	C9—O1—C2—C3	−0.1 (3)
C2—C11—C16—C15	178.74 (17)	C9—O1—C2—C11	179.37 (15)
O3—C3—C4—O4	0.3 (3)	C10—C5—C6—C7	−1.0 (3)
O3—C3—C4—C10	−179.63 (16)	C11—C2—C3—O3	0.0 (3)
C3—C2—C11—C12	177.65 (18)	C11—C2—C3—C4	−178.14 (17)
C3—C2—C11—C16	−1.1 (3)	C11—C12—C13—C14	−0.5 (3)
C3—C4—C10—C5	−179.61 (16)	C12—C11—C16—C15	0.0 (3)
C3—C4—C10—C9	0.5 (3)	C12—C13—C14—O14	−179.91 (18)
O4—C4—C10—C5	0.5 (3)	C12—C13—C14—C15	0.1 (3)
O4—C4—C10—C9	−179.37 (17)	C13—C14—C15—C16	0.4 (3)
O5—C5—C6—C7	179.63 (16)	O14—C14—C15—C16	−179.62 (18)
O5—C5—C10—C4	−0.1 (3)	C14—C15—C16—C11	−0.4 (3)
O5—C5—C10—C9	179.77 (16)	C16—C11—C12—C13	0.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1*W*^{i}	0.99 (3)	1.80 (3)	2.7385 (19)	158 (2)
O5—H5···O4	0.92 (3)	1.78 (3)	2.6241 (17)	152 (2)
O7—H7···O14^{ii}	0.91 (3)	1.84 (3)	2.7413 (17)	169 (2)
O14—H14···O1*W*	0.85 (3)	1.82 (3)	2.6727 (19)	172 (2)
O1*W*—H1*WA*···O4^{iii}	0.89 (3)	1.90 (3)	2.780 (2)	168 (3)
O1*W*—H1*WB*···O5^{iv}	0.84 (3)	1.96 (3)	2.7842 (17)	169 (3)

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