# Novel Constrained 2'-Methylribonucleosides: Synthesis and Incorporation into Oligonucleotides

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### **Electronic Supplementary Information**

S1	General information	2
S2	NMR + HRMS: 2'-C-methyl-3',4'-cyclohexylideneuridine (2)	3
S3	<b>NMR</b> + <b>HRMS</b> : 2'- <i>C</i> -methyl-2',3'-cyclohexylidene-4'- <i>C</i> -hydroxymethyluridine ( <b>3</b> )	6-8
S4	NMR + HRMS: 2'-methyl-2',3'-cyclohexylidene-4'-C-methylene-bis(5',5''-	9-11
	mesylate)uridine (4)	
S5	NMR + HRMS: 2'-C-methyl-4'-C-methylene-bis(5',5''-mesylate)uridine (5)	12-14
S6	NMR + HRMS: 2'-C-methyl-2' <i>O</i> -4' <i>C</i> -methylene-5'-mesylateuridine (6a)	15-20
	and 2'-C-methyl-3'O-4'C-methylene-5'-mesylateuridine (6b)	
S7	NMR + HRMS: 2'-C-methyl-2'O-4'C-methyleneuridine (7a)	21-23
S8	NMR + HRMS: 2'-C-methyl-2' <i>O</i> -4' <i>C</i> -methylene-5'- <i>O</i> DMTr-uridine (8a)	24-26
S9	<sup>31</sup> <b>P-NMR + HRMS:</b> Synthesis of 2'- <i>C</i> -methyl-2' <i>O</i> -4' <i>C</i> -methylene-5'- <i>O</i> DMTr-uridine	27-28
	3'-O-cyanoethyl- <i>N</i> , <i>N</i> -diisopropylamine phosphoramidite (9a)	
S10	NMR + HRMS: 2'-C-methyl-3'O-4'C-methyleneuridine (7b)	29-31
S11	NMR + HRMS: 2'-C-methyl-3'O-4'C-methylene-5'-ODMTr-uridine (8b)	32-34
S12	<sup>31</sup> <b>P-NMR + HRMS:</b> Synthesis of 2'- <i>C</i> -methyl-2'3-4' <i>C</i> -methylene-5'- <i>O</i> DMTr-uridine	35-36
	2'-O-cyanoethyl- <i>N</i> , <i>N</i> -diisopropylamine phosphoramidite ( <b>9b</b> )	
S13	NMR + HRMS: 3'O-4'C-methylene-5'-OMs 5-methyluridine (11)	37-39
Figure S1	Synthetic cycle for oligonucleotide synthesis	40
S14	MALDI-TOF and HPLC spectra: ON1 to ON11	41-51
S15	Crystallography: Further descriptions, figures and geometrical data	52-82

#### **General information**

All reagents used were purchased from Sigma-Aldrich, Fluka and used without purification. DNA phosphoramidite monomers, solid supports and additional reagents were purchased from Sigma-Aldrich or Glen Research. Dichloromethane (DCM), N,N-diisopropylethylamine (DIPEA), N,N-dimethylformamide (DMF) and pyridine were dried over activated molecular sieve (3 Å, 8-12 mesh) and their dryness was determined on Karl Fischer titrator (< 15 ppm). Column chromatography was carried out under pressure using Merck Millipore silica gel 60 (0.040-0.063 mm). Thin layer chromatography (TLC) was performed using Merck Kieselgel 60 F254 (0.22 mm thickness, aluminium backed). Compounds were visualized at 254 nm or stained with 10 % sulfuric acid in EtOH. <sup>1</sup>H-NMR spectra were measured at 400 MHz on a Bruker AVANCE III 400 spectrometer. <sup>13</sup>C-NMR spectra were measured at 101 MHz on the same spectrometer. Chemical shifts are given in ppm and J values are given in Hz. All assignments for <sup>1</sup>H-NMR and <sup>13</sup>C-NMR have been confirmed by H-H COSY, HMOC and HMBC. <sup>31</sup>P-NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer at 162 MHz. CD<sub>3</sub>CN was used as solvents. High resolution mass spectra were recorded in acetonitrile or methanol using the electrospray technique on a Bruker APEX III FT-ICR mass spectrometer. HPLC grade CH<sub>3</sub>CN or methanol were used as the solvent.

### S2: <sup>1</sup>H-NMR of (2)



S2: <sup>13</sup>C-NMR of (2)



#### S2: HRMS of (2)

Method

Comment



**Acquisition Parameter** lon Polarity Set Capillary Set End Plate Offset 0.3 Bar 180 °C 4.0 l/min Source Type ESI Positive Set Nebulizer Focus Not active 4200 V Set Dry Heater 50 m/z 1500 m/z Scan Begin -500 V Set Dry Gas Scan End Set Collision Cell RF 325.0 Vpp Set Divert Valve Source Intens. +MS, 21.2-29.3s #(21-29) 361.1373 x104 677.3016 3 2 1037.4323 1 227.1274 1375,5769 1204.5242 902.3632 489.1828 0 200 400 600 800 1000 1200 1400 m/zIntens. +MS, 21.2-29.3s #(21-29) x104 361.1373 3 2 339.1549 1 377.1123 0 320 3<u>0</u>0 340 360 380 m/z N-R Meas. m/z Formula Score m/z Mean mSig rdb # err e<sup>-</sup>

1 C 16 H 22 N 2 Na O 6

361.1373

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[ppm]

-0.8

err

-0.4

[ppm]

ma

2.9

ule

ok

Conf

6.5 even

361.1370

100.00

S3: <sup>1</sup>H-NMR of (3)



S3: <sup>13</sup>C-NMR of (3)



#### S3: HRMS of (3)

# Mass Spectrum SmartFormula Report



8





S4: <sup>13</sup>C-NMR of (4)



#### S4: HRMS of (4)

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Page 1 of 1

S5: <sup>1</sup>H-NMR of (5)



S5: <sup>13</sup>C-NMR of (5)



#### S5: HRMS of (5)

# Mass Spectrum SmartFormula Report

#### Analysis Info

Analysis Name	D:\Data\PKH\ML 05_RA1_01_44963.d
Method	sample pos low.m
Sample Name	ML 05
Comment	

Acquisition Date 12/23/2016 9:31:18 AM

Operator BDAL@DE Instrument / Ser# micrOTOF-Q II 10205



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S6: <sup>1</sup>H-NMR of (6a)





S6: <sup>13</sup>C-NMR of (6a)

#### S6: HRMS of (6a)

### Mass Spectrum SmartFormula Report

#### Analysis Info

Analysis Name	D:\Data\PKH\KIM ML 05T_RB4_01_42045.d
Method	standard pos low.m
Sample Name	KIM ML 05T
Comment	

#### Acquisition Date 6/24/2016 8:09:10 PM

Operator BDAL@DE Instrument / Ser# micrOTOF-Q II 10205



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Page 1 of 1







#### S6: HRMS of (6b)



S7: <sup>1</sup>H-NMR of (7a)



21



#### S7: HRMS of (7a)

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Page 1 of 1

S8:



S8: <sup>1</sup>H-NMR of (8a)



#### S8: HRMS of (8a)

### Mass Spectrum SmartFormula Report



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Page 1 of 1

S9: <sup>31</sup>P-NMR of (9a)



#### S9: HRMS of (9a)

### Mass Spectrum SmartFormula Report



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Page 1 of 1

S10: <sup>1</sup>H-NMR of (7b)





90 80 f1 (ppm)

S10: <sup>13</sup>C-NMR of (7b)

#### S10: HRMS of (7b)

### Mass Spectrum SmartFormula Report

#### Analysis Info

Analysis Name	D:\Data\PKH\KIMVK ML10_RA3_01_43322.d
Method	standard pos low.m
Sample Name	KIMVK ML10
Commont	

Acquisition Date 9/7/2016 5:30:37 PM

Operator BDAL@DE Instrument / Ser# micrOTOF-Q II 10205



S11: <sup>1</sup>H-NMR of (8b)



S11: <sup>13</sup>C-NMR of (8b)



#### S1: HRMS of (8b)

# Mass Spectrum SmartFormula Report



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Page 1 of 1



# S12: <sup>31</sup>P-NMR of (9b)

#### S12: HRMS of (9b)




S13: <sup>1</sup>H-NMR of (11)



#### S13: HRMS of (11)



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Page 1 of 1

#### Figure S1



**Fig. S1**. The synthetic cycle for incorporation of the 2'-Me-LNA and 2'-Me-ONA phosphoramidites (**9a**) and (**9b**) as monomer **X** and **Y** respectively into oligonucleotides. After coupling the LNA phosphoramidite (**9a**) it is capped and then oxidized. After coupling of the 2'-Me-ONA phosphoramidite (**9b**) it is oxidized using the 0.5 M CSO for 6 minutes and then capped.

# S14 - ON1 to ON11 MALDI-TOF and HPLC spectra

ON1 – IE-HPLC







### ON2 – IE-HPLC







42



**ON3 – MALDI-TOF MS** 











### ON5 – IE-HPLC



**ON5 – MALDI-TOF MS** 



### ON6 – IE-HPLC



ON6 - MALDI-TOF MS



### ON7 – IE-HPLC



**ON7 – MALDI-TOF MS** 



### ON8 – IE-HPLC



**ON8 – MALDI-TOF MS** 



#### ON9 – IE-HPLC



**ON9 – MALDI-TOF MS** 



### ON10 - IE-HPLC



ON10 - MALDI-TOF MS



#### ON11 - IE-HPLC



ON11 - MALDI-TOF MS



# S15 – Crystal data



### Comments

Good quality data obtained on a small crystal. Refined to a final R1 of 4.7% in the chiral space group  $P4_12_12$ , Absolute configuration was determined independently by the X-ray experiment (absolute structure parameter 0.009 (12)).

The molecular structure is as expected (Plot 1, above ), all bond lengths and angles are in the expected ranges.

The molecules form dimeric "base-paired" units by hydrogen bonding between pyrimidine groups (N2 – O4 2.775 (4) Å under symmetry operation y, x, -z+1), see Plot 2. Each molecule also undergoes hydrogen bonding involving the alcohol and second carbonyl (O2 – O5 2.737 (4) Å under symmetry operation -x+3/2, y+1/2, -z+5/4, Plot 3). Overall each molecule is involved in four hydrogen bonds, two as donor and two as acceptor. There are also a number of longer C-H<sup>...</sup>O hydrogen bond interactions, these are less important in controlling the packing but may be responsible for the orientation of the sulfonate.

The two pyrimidine rings in the "base-pair" unit are inclined at an angle of 24.48(12)° to each other . This may be a supramolecular effect as the H-bonded pyrimidines lie in a curved cavity defined by other H-bond interactions (Plot 4). It doesn't appear to be caused by any steric stress directly between the two dimerising molecules.

The methyl group at C7 overhangs the pyrimidine and might be expected to affect any intercalation properties and to restrict rotation of the pyrimidine. It does not appear as if C7 should hinder the pyrimidine from making H-bonds via O4 and N2. However, if steric reasons required the molecule to H-bond via N2 and O5 instead, it could be inhibited.

# Experimental

The data were collected at 150(2)K on a Synergy, Dualflex, AtlasS2 diffractometer using CuK $\alpha$  radiation ( $\lambda = 1.54184$  Å) and the *CrysAlis PRO* 1.171.38.43 suite<sup>1</sup>. Using SHELXT<sup>2</sup> the structure was solved by dual space methods (SHELXT<sup>2</sup>) and refined on F<sup>2</sup> using all the reflections (SHELXL-2016<sup>3</sup>). All the non-hydrogen atoms were refined using anisotropic atomic displacement parameters and hydrogen atoms were inserted at calculated positions using a riding model. Parameters for data collection and refinement are summarised in Table 1.

- 1. CrysAlis PRO 1.171.38.43, Rigaku Oxford Diffraction, 2015.
- 2. C.B. Hübschle, G.M. Sheldrick and B. Dittrich. J. Appl. Cryst., 2011, 44, 1281-1284.
- 3. G.M. Sheldrick, Acta Cryst., 2015, A71, 3-8.
- 4. G.M. Sheldrick, Acta Cryst., 2015, C71, 3-8.

$C_{12}H_{16}N_2O_8S$	$D_{\rm x} = 1.562 {\rm ~Mg} {\rm m}^{-3}$
$M_r = 348.33$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Tetragonal, P4 <sub>1</sub> 2 <sub>1</sub> 2	F(000) = 1456
<i>a</i> = 8.7910 (1) Å	$\mu = 2.39 \text{ mm}^{-1}$
<i>c</i> = 38.3254 (8) Å	T = 100  K
$V = 2961.85 (9) \text{ Å}^3$	Cube, colourless
Z = 8	$0.06 \times 0.04 \times 0.02 \text{ mm}$

### Crystal data JWE1

11649 measured reflections	$h = -8 \rightarrow 10$
3045 independent reflections	$k = -11 \rightarrow 8$
2807 reflections with $I > 2\sigma(I)$	<i>l</i> = -47→35
$R_{\rm int} = 0.036$	210 parameters
<i>S</i> = 1.04	0 restraints
$wR(F^2) = 0.115$	$\Delta$ <sub>max</sub> = 0.38 e Å <sup>-3</sup>
Absolute structure parameter: 0.009 (12)	$\Delta \rangle_{\rm min} = -0.40 \ e \ {\rm \AA}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.047$	

Computing details: Data collection: CrysAlis PRO 1.171.39.12b (Rigaku OD, 2015); cell

refinement: *CrysAlis PRO* 1.171.39.12b (Rigaku OD, 2015); data reduction: *CrysAlis PRO* 1.171.39.12b (Rigaku OD, 2015); program(s) used to solve structure: SHELXT 2014/5 (Sheldrick, 2014); program(s) used to refine structure: *SHELXL2016*/6 (Sheldrick, 2016).

S1—O8	1.423 (3)	N1-C11	1.380 (4)	
S1—O7	1.424 (3)	N1C1	1.485 (4)	
S1—O6	1.571 (3)	N2-C10	1.372 (5)	
S1—C12	1.745 (4)	N2-C11	1.374 (4)	
O1—C1	1.423 (4)	C1—C2	1.548 (5)	
O1—C4	1.455 (4)	C2—C7	1.489 (5)	
O2—C3	1.403 (4)	C2—C3	1.534 (5)	
O3—C6	1.448 (5)	C3—C4	1.523 (5)	
O3—C2	1.453 (4)	C4—C5	1.505 (5)	
O4—C10	1.239 (4)	C4—C6	1.528 (6)	
O5—C11	1.225 (5)	С8—С9	1.345 (5)	
O6—C5	1.466 (5)	C9—C10	1.434 (5)	
N1—C8	1.367 (5)			
O8—S1—O7	119.1 (2)	C3—C2—C1	100.1 (3)	
O8—S1—O6	109.81 (18)	O2—C3—C4	111.9 (3)	
O7—S1—O6	103.42 (18)	O2—C3—C2	114.9 (3)	
O8—S1—C12	109.3 (2)	C4—C3—C2	92.7 (3)	
O7—S1—C12	109.8 (2)	O1—C4—C5	110.2 (3)	
O6—S1—C12	104.36 (19)	O1—C4—C3	101.7 (3)	
C1—O1—C4	107.2 (3)	C5—C4—C3	119.7 (3)	
С6—О3—С2	106.6 (3)	O1—C4—C6	106.3 (3)	
C5—O6—S1	120.2 (2)	C5—C4—C6	116.9 (3)	
C8—N1—C11	120.9 (3)	C3—C4—C6	100.2 (3)	
C8—N1—C1	122.2 (3)	O6—C5—C4	109.4 (3)	
C11—N1—C1	116.6 (3)	O3—C6—C4	103.0 (3)	
C10—N2—C11	126.6 (3)	C9—C8—N1	122.9 (3)	
01—C1—N1	108.3 (3)	C8—C9—C10	119.0 (4)	
O1—C1—C2	103.2 (3)	O4—C10—N2	119.7 (3)	
N1—C1—C2	115.4 (3)	O4—C10—C9	125.1 (4)	
O3—C2—C7	110.2 (3)	N2-C10-C9	115.1 (3)	

# Geometric parameters (Å, °) for JWE1

O3—C2—C3	102.5 (3)	O5-C11-N2	121.9 (3)
С7—С2—С3	119.1 (3)	O5-C11-N1	122.9 (3)
O3—C2—C1	103.3 (3)	N2-C11-N1	115.2 (3)
C7—C2—C1	119.3 (3)		
08—S1—O6—C5	-49.4 (3)	02—C3—C4—O1	-172.9 (3)
07—S1—O6—C5	-177.6 (3)	C2-C3-C4-01	-54.7 (3)
C12—S1—O6—C5	67.6 (3)	O2—C3—C4—C5	65.5 (5)
C4—O1—C1—N1	-123.5 (3)	C2—C3—C4—C5	-176.3 (3)
C4—O1—C1—C2	-0.7 (4)	O2—C3—C4—C6	-63.7 (4)
C8—N1—C1—O1	16.5 (5)	C2—C3—C4—C6	54.5 (3)
C11—N1—C1—O1	-157.0 (3)	S1—O6—C5—C4	116.9 (3)
C8—N1—C1—C2	-98.5 (4)	O1—C4—C5—O6	-82.4 (4)
C11—N1—C1—C2	88.0 (4)	C3—C4—C5—O6	34.9 (5)
C6—O3—C2—C7	159.6 (3)	C6—C4—C5—O6	156.1 (3)
C6—O3—C2—C3	31.8 (4)	C2—O3—C6—C4	4.1 (4)
C6-03-C2-C1	-71.9 (3)	O1—C4—C6—O3	67.0 (4)
01—C1—C2—O3	71.1 (3)	C5—C4—C6—O3	-169.5 (3)
N1-C1-C2-O3	-171.0 (3)	C3—C4—C6—O3	-38.5 (3)
O1—C1—C2—C7	-166.3 (3)	C11—N1—C8—C9	-4.1 (6)
N1—C1—C2—C7	-48.3 (5)	C1—N1—C8—C9	-177.3 (4)
O1—C1—C2—C3	-34.5 (3)	N1	1.6 (6)
N1—C1—C2—C3	83.4 (3)	C11—N2—C10—O4	-176.7 (4)
O3—C2—C3—O2	62.5 (4)	C11—N2—C10—C9	1.6 (6)
C7—C2—C3—O2	-59.4 (4)	C8—C9—C10—O4	177.9 (4)
C1—C2—C3—O2	168.7 (3)	C8—C9—C10—N2	-0.2 (6)
O3—C2—C3—C4	-53.1 (3)	C10—N2—C11—O5	176.7 (4)
C7—C2—C3—C4	-175.1 (3)	C10—N2—C11—N1	-3.9 (6)
C1—C2—C3—C4	53.0 (3)	C8—N1—C11—O5	-175.7 (4)
C1—O1—C4—C5	164.2 (3)	C1—N1—C11—O5	-2.1 (5)
C1—O1—C4—C3	36.1 (4)	C8—N1—C11—N2	5.0 (5)
C1C4C6	-68.3 (4)	C1—N1—C11—N2	178.6 (3)

Hydrogen-bond geometry (Å, °) for JWE1

D—H···A	D—H	$H \cdots A$	$D \cdots A$	D—H···A

O2— $H2A$ ···O5 <sup>i</sup>	0.84	2.09	2.737 (4)	134
N2—H2···O4 <sup>ii</sup>	0.88	1.90	2.775 (4)	174
С8—Н8…О8	0.95	2.49	3.365 (5)	154
C7—H7 <i>B</i> ⋯O8 <sup>iii</sup>	0.98	2.58	3.540 (5)	166
C7— $H7C$ ···O3 <sup>i</sup>	0.98	2.50	3.474 (5)	173
C5—H5A…O4 <sup>iv</sup>	0.99	2.39	3.357 (5)	165
C5—H5 $B$ ···O2 <sup>v</sup>	0.99	2.49	3.467 (5)	170
C9—H9…O1 <sup>vi</sup>	0.95	2.44	3.316 (5)	153
C12—H12 $B$ ····O7 <sup>v</sup>	0.98	2.47	3.427 (6)	165
C12—H12 $C$ ···O4 <sup>iv</sup>	0.98	2.49	3.400 (6)	154

Symmetry codes: (i) -x+3/2, y+1/2, -z+5/4; (ii) y, x, -z+1; (iii) x+1, y, z; (iv) -x+1/2, y-1/2, -z+5/4; (v) -y+1, -x+1, -z+3/2; (vi) -x+1/2, y+1/2, -z+5/4.

Document origin: publCIF [Westrip, S. P. (2010). J. Apply. Cryst., 43, 920-925].

### Mean plane calculation

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (\* indicates atom used to define plane)

6.7639 (0.0060) x + 5.2864 (0.0080) y + 8.2552 (0.0545) z = 10.3639 (0.0294)

- \* 0.0368 (0.0026) N1
- \* -0.0145 (0.0027) C8
- \* -0.0181 (0.0031) C9
- \* -0.0006 (0.0026) C10
- \* 0.0240 (0.0027) N2
- \* 0.0031 (0.0032) C11
- \* -0.0308 (0.0023) O5

Rms deviation of fitted atoms = 0.0221

5.2864 (0.0080) x + 6.7639 (0.0060) y - 8.2552 (0.0543) z = 2.1087 (0.0250)

Angle to previous plane (with approximate esd) = 28.483 (0.119)

- \* 0.0368 (0.0027) N1 \$2
- \* -0.0145 (0.0027) C8\_\$2
- \* -0.0181 (0.0031) C9\_\$2
- \* -0.0006 (0.0026) C10\_\$2
- \* 0.0240 (0.0027) N2\_\$2
- \* 0.0032 (0.0032) C11\_\$2
- \* -0.0308 (0.0022) O5\_\$2

Rms deviation of fitted atoms = 0.0221

JWE1 size  $0.06 \times 0.04 \times 0.02$  mm







Plot 1. Showing 50% atomic displacement ellipsoids



Plot 2. Intermolecular "base-pair" H-bonding (dashed red lines)



Plot 3. H-bonding to adjacent molecules



Plot 4. "Slice" to show that the H-bond framework defines curved cavity for the pyrimidine groups i.e the non-planar link may be an effect of the supramolecular H-bond network.



Plot 5. Position of the methyl group relative to the pyrimidine ring.



Plot 6. Position of the methyl group relative to the H-bonded dimer (carbons of  $2^{nd}$  molecule shown in pale blue).



Plot 7. Unit cell plot viewed down the *a* axis



Plot 8. Unit cell plot showing H-bonding



Plot 9. Unit cell plot viewed down the *c* axis (4-fold screw axis)



 $0.09 \times 0.04 \times 0.02 \text{ mm}$ 

### Comments

Reasonably good quality data were obtained on a small, colourless crystal. The structure refined to a final R1 of 5.4% in the chiral space group P1 with four independent molecules in the asymmetric unit, the absolute configuration was determined independently by the X-ray experiment (absolute structure parameter -0.005 (18))

The molecular structure of one of the molecules is shown above (Plot), there are three other independent molecules (i.e. not related by symmetry)in the asymmetric unit, these have the same labelling system but with suffixes B, C and D. All four molecules have the same absolute configuration, the differences between them are small conformational changes, probably due to packing in the solid state.

In molecule C the methyl sulfonate is disordered by rotation about the O6C – S1C bond. Molecule B has a slight rotational disorder of the pyrimidine ring, most noticeable at the methyl group. These have each been modelled as 56:44% occupancy of two positions related by rotation. There do not seem to be any unusual bond lengths or angles. (To have more than one independent molecule in the asymmetric unit is unusual, however I am satisfied it is genuine in this case – rather than due to missed symmetry.)

The molecules form dimeric "base-paired" units by hydrogen bonding between pyrimidine groups (involving N2and O4 on each molecule (see H-bond Table and PPT). In contrast to the previous structure JWE1, the pyrimidine pairs in this structure are almost coplanar (interplanar

angles 12.5(9)° and 4.8(3)° for the pyrimidine rings of molecules A & B and C & D, respectively).

The alcohol group (O2) of each molecule also undergoes hydrogen bonding to the carbonyls O5 and O4 of a neighbouring "base pair" (H-bond table). For molecule A these two H-bonds are approximately equivalent but for the other molecules they are more asymmetric with the O2<sup>...</sup>O5 interaction more convincing. The net effect of the intramolecular hydrogen bonding is to link the molecules into 2D sheets running parallel to the (101) plane.

In comparison to JWE1, the current compound appears to have better access around the pyrimidine ring (see Plot 11).

## Experimental

The data were collected at 100(2)K on a Synergy, Dualflex, AtlasS2 diffractometer using CuK $\alpha$  radiation ( $\lambda = 1.54184$  Å) and the *CrysAlis PRO* 1.171.39.12b suite<sup>1</sup>. Using SHELXLE<sup>2</sup> the structure was solved by dual space methods (SHELXT<sup>2</sup>) and refined on F<sup>2</sup> using all the reflections (SHELXL-2016<sup>3</sup>). All the non-hydrogen atoms were refined using anisotropic atomic displacement parameters. Hydrogen atoms bonded to carbon were inserted at calculated positions using a riding model; those bonded to oxygen or nitrogen were located from difference maps and their coordinates refined. The methylsulfonate group of one molecule (C) was disordered, this was modelled as 56:44% occupancy of two conformations related by rotation about the C6C – S1C bond. Part of the pyrimidine group of molecule B was also disordered; since this section is close to the disordered methylsulfonate it was modelled with the same 56:44% occupancy of two sites. Parameters for data collection and refinement are summarised in Table 1.

- 1. CrysAlis PRO 1.171.39.12b , Rigaku Oxford Diffraction, 2015.
- 2. C.B. Hübschle, G.M. Sheldrick and B. Dittrich. J. Appl. Cryst., 2011, 44, 1281-1284.
- 3. G.M. Sheldrick, Acta Cryst., 2015, A71, 3-8.
- 4. G.M. Sheldrick, Acta Cryst., 2015, C71, 3-8.

$C_{12}H_{16}N_2O_8S$	$V = 1480.64 (7) \text{ Å}^3$
$M_r = 348.33$	<i>Z</i> = 4
Triclinic, P1	$D_{\rm x} = 1.563 {\rm ~Mg} {\rm m}^{-3}$
a = 9.0609 (3)  Å	Cu K $\alpha$ radiation, $\lambda = 1.54184$ Å
<i>b</i> = 10.3679 (2) Å	F(000) = 728
c = 16.4856 (4) Å	$\mu = 2.39 \text{ mm}^{-1}$

### Crystal data JWE2

$\alpha = 82.494 \ (2)^{\circ}$	T = 100  K
$\beta = 85.478 \ (2)^{\circ}$	Block, colourless
$\gamma = 74.878 \ (2)^{\circ}$	

32404 measured reflections	$h = -10 \rightarrow 10$
10353 independent reflections	$k = -12 \rightarrow 12$
9332 reflections with $I > 2\sigma(I)$	$l = -19 \rightarrow 19$
$R_{\rm int} = 0.051$	916 parameters
<i>S</i> = 1.05	82 restraints
$wR(F^2) = 0.146$	$(\Delta/\sigma)_{\rm max} < 0.001$
Absolute structure parameter: -0.005 (18)	$\Delta \rangle_{\text{max}} = 0.55 \text{ e} \text{ Å}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.054$	$\Delta \rangle_{\rm min} = -0.43 \ {\rm e} \ {\rm \AA}^{-3}$

**Computing details:** Data collection: *CrysAlis PRO* 1.171.39.12b (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* 1.171.39.12b (Rigaku OD, 2015); data reduction: *CrysAlis PRO* 1.171.39.12b (Rigaku OD, 2015); program(s) used to solve structure: SHELXT 2014/5 (Sheldrick, 2014); program(s) used to refine structure: *SHELXL2016*/6 (Sheldrick, 2016); molecular graphics: *Mercury* (Macrae at al., 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

*Geometric parameters (Å, °) for JWE2*.(Atoms with suffixes X or Y are the minor components of disorder on molecules C and B, respectively)

S1A—08A	1.413 (5)	С8Ү—С9Ү	1.517 (14)
S1A—07A	1.423 (6)	S1C—07X	1.382 (13)
S1A—O6A	1.576 (5)	S1C—07C	1.403 (10)
S1A—C12A	1.728 (8)	S1C—O8X	1.437 (11)
O1A—C1A	1.430 (7)	S1C—O8C	1.439 (9)
O1A—C4A	1.457 (8)	S1C—O6C	1.575 (5)
O2A—C3A	1.390 (9)	S1C—C12X	1.653 (18)
O3A—C2A	1.430 (8)	S1C—C12C	1.774 (11)
O3A—C6A	1.446 (9)	01C—C1C	1.423 (7)
O4A—C10A	1.227 (7)	O1C—C4C	1.453 (9)
O5A—C11A	1.236 (7)	O2C—C3C	1.396 (8)

O6A—C5A	1.454 (8)	O3C—C2C	1.428 (8)
N1A—C11A	1.373 (7)	O3C—C6C	1.460 (8)
N1A—C7A	1.383 (7)	O4C—C10C	1.224 (8)
N1A—C1A	1.488 (7)	05C—C11C	1.237 (7)
N2A—C11A	1.360 (7)	O6C—C5C	1.447 (8)
N2A—C10A	1.388 (7)	N1C—C11C	1.382 (8)
C1A—C2A	1.520 (10)	N1C—C7C	1.391 (8)
C2A—C3A	1.529 (9)	N1C—C1C	1.467 (8)
C3A—C4A	1.536 (9)	N2C—C11C	1.346 (8)
C4A—C5A	1.495 (9)	N2C—C10C	1.380 (8)
C4A—C6A	1.525 (9)	C1C—C2C	1.529 (9)
C7A—C8A	1.347 (8)	C2C—C3C	1.529 (8)
C8A—C10A	1.437 (8)	C3C—C4C	1.543 (9)
С8А—С9А	1.504 (8)	C4C—C5C	1.498 (9)
S1B—O7B	1.411 (6)	C4C—C6C	1.518 (9)
S1B—O8B	1.433 (5)	C7C—C8C	1.327 (9)
S1B—O6B	1.575 (5)	C8C—C10C	1.449 (9)
S1B—C12B	1.733 (7)	C8C—C9C	1.497 (9)
O1B—C1B	1.411 (7)	S1D—07D	1.420 (6)
O1B—C4B	1.454 (8)	S1D—O8D	1.427 (5)
O2B—C3B	1.396 (8)	S1D—O6D	1.583 (5)
O3B—C2B	1.440 (7)	S1D—C12D	1.733 (7)
O3B—C6B	1.446 (8)	01D—C1D	1.430 (8)
O4B—C10Y	1.229 (7)	O1D—C4D	1.466 (8)
O4B—C10B	1.229 (7)	O2D—C3D	1.392 (8)
O5B—C11B	1.227 (7)	O3D—C2D	1.435 (7)
O6B—C5B	1.457 (8)	O3D—C6D	1.450 (8)
N1B—C11B	1.362 (8)	O4D—C10D	1.233 (8)
N1B—C7Y	1.36 (4)	O5D—C11D	1.235 (8)
N1B—C7B	1.44 (4)	O6D—C5D	1.455 (8)
N1B—C1B	1.493 (7)	N1D—C11D	1.365 (8)
N2B—C11B	1.369 (7)	N1D—C7D	1.395 (8)
N2B—C10B	1.386 (8)	N1D—C1D	1.487 (8)
N2B—C10Y	1.386 (8)	N2D—C11D	1.358 (9)
C1B—C2B	1.524 (10)	N2D—C10D	1.372 (8)
C2B—C3B	1.525 (8)	C1D—C2D	1.528 (9)
C3B—C4B	1.535 (9)	C2D—C3D	1.535 (8)

C4B—C5B	1.497 (9)	C3D—C4D	1.522 (9)
C4B—C6B	1.520 (8)	C4D—C5D	1.504 (9)
С7В—С8В	1.330 (18)	C4D—C6D	1.518 (9)
C8B—C10B	1.456 (18)	C7D—C8D	1.332 (9)
C8B—C9B	1.519 (18)	C8D—C10D	1.450 (9)
C7Y—C8Y	1.330 (15)	C8D—C9D	1.511 (9)
C8Y—C10Y	1.447 (15)		
08A—S1A—07A	118.9 (3)	O5B—C11B—N2B	122.1 (5)
08A—S1A—O6A	109.7 (3)	N1B—C11B—N2B	115.6 (5)
07A—S1A—O6A	107.7 (4)	07X—S1C—O8X	117.2 (9)
08A—S1A—C12A	110.0 (3)	O7C—S1C—O8C	119.8 (6)
O7A—S1A—C12A	109.5 (4)	O7X—S1C—O6C	104.4 (6)
O6A—S1A—C12A	99.2 (4)	07C—S1C—O6C	109.9 (5)
C1A—O1A—C4A	106.2 (5)	O8X—S1C—O6C	104.0 (5)
C2A—O3A—C6A	106.8 (5)	08C—S1C—O6C	111.0 (4)
C5A—O6A—S1A	119.3 (4)	07X—S1C—C12X	114.6 (10)
C11A—N1A—C7A	121.0 (5)	08X—S1C—C12X	111.9 (9)
C11A—N1A—C1A	115.9 (5)	O6C—S1C—C12X	102.8 (7)
C7A—N1A—C1A	123.1 (5)	07C—S1C—C12C	108.5 (6)
C11A—N2A—C10A	126.3 (5)	O8C—S1C—C12C	107.3 (5)
O1A—C1A—N1A	109.1 (5)	O6C—S1C—C12C	98.0 (4)
O1A—C1A—C2A	103.1 (5)	C1C—O1C—C4C	106.1 (5)
N1A—C1A—C2A	112.9 (5)	C2C—O3C—C6C	105.7 (5)
O3A—C2A—C1A	105.8 (6)	C5C—O6C—S1C	118.2 (4)
O3A—C2A—C3A	103.8 (5)	C11C—N1C—C7C	120.2 (5)
C1A—C2A—C3A	100.4 (5)	C11C—N1C—C1C	116.0 (5)
O2A—C3A—C2A	113.8 (6)	C7C—N1C—C1C	123.6 (5)
O2A—C3A—C4A	115.9 (6)	C11C—N2C—C10C	127.0 (6)
C2A—C3A—C4A	90.8 (5)	O1C—C1C—N1C	110.5 (5)
O1A—C4A—C5A	110.6 (5)	01C—C1C—C2C	103.1 (5)
O1A—C4A—C6A	106.4 (6)	N1C—C1C—C2C	115.2 (5)
C5A—C4A—C6A	114.2 (5)	O3C—C2C—C1C	106.3 (5)
O1A—C4A—C3A	101.9 (5)	O3C—C2C—C3C	104.4 (5)
С5А—С4А—С3А	121.1 (6)	C1C—C2C—C3C	100.5 (5)
С6А—С4А—С3А	101.1 (5)	O2C—C3C—C2C	111.8 (5)
O6A—C5A—C4A	106.9 (5)	O2C—C3C—C4C	114.4 (5)

O3A—C6A—C4A	102.0 (5)	C2C—C3C—C4C	90.1 (5)
C8A—C7A—N1A	122.5 (5)	O1C—C4C—C5C	110.6 (6)
C7A—C8A—C10A	118.8 (5)	O1C—C4C—C6C	108.2 (6)
С7А—С8А—С9А	123.4 (6)	C5C—C4C—C6C	114.6 (5)
C10A—C8A—C9A	117.6 (5)	O1C—C4C—C3C	101.7 (5)
O4A—C10A—N2A	120.1 (5)	C5C—C4C—C3C	119.5 (6)
O4A—C10A—C8A	124.6 (5)	C6C—C4C—C3C	100.9 (5)
N2A—C10A—C8A	115.3 (5)	O6C—C5C—C4C	107.7 (5)
O5A—C11A—N2A	121.9 (5)	O3C—C6C—C4C	102.3 (5)
O5A—C11A—N1A	122.0 (5)	C8C—C7C—N1C	123.5 (6)
N2A—C11A—N1A	116.0 (5)	C7C—C8C—C10C	118.1 (6)
O7B—S1B—O8B	118.7 (3)	C7C—C8C—C9C	124.6 (6)
O7B—S1B—O6B	108.5 (4)	C10C—C8C—C9C	117.1 (6)
O8B—S1B—O6B	109.6 (3)	O4C-C10C-N2C	120.0 (6)
O7B—S1B—C12B	109.6 (4)	O4C—C10C—C8C	124.6 (6)
O8B—S1B—C12B	110.4 (3)	N2C-C10C-C8C	115.4 (6)
O6B—S1B—C12B	98.2 (3)	O5C—C11C—N2C	123.2 (6)
C1B—O1B—C4B	106.5 (5)	O5C—C11C—N1C	121.1 (6)
C2B—O3B—C6B	106.1 (4)	N2C—C11C—N1C	115.7 (5)
C5B—O6B—S1B	117.4 (4)	O7D—S1D—O8D	119.1 (3)
C11B—N1B—C7Y	120.6 (10)	O7D—S1D—O6D	107.9 (4)
C11B—N1B—C7B	120.8 (12)	O8D—S1D—O6D	108.9 (3)
C11B—N1B—C1B	116.7 (5)	O7D—S1D—C12D	109.8 (4)
C7Y—N1B—C1B	122.4 (11)	O8D—S1D—C12D	110.3 (4)
C7B—N1B—C1B	121.7 (12)	O6D—S1D—C12D	98.9 (3)
C11B—N2B—C10B	126.3 (5)	C1D—O1D—C4D	105.7 (5)
C11B—N2B—C10Y	126.3 (5)	C2D—O3D—C6D	106.9 (4)
O1B—C1B—N1B	109.1 (5)	C5D—O6D—S1D	118.5 (4)
O1B—C1B—C2B	103.4 (5)	C11D—N1D—C7D	121.2 (5)
N1B—C1B—C2B	113.0 (5)	C11D—N1D—C1D	116.5 (5)
O3B—C2B—C1B	105.2 (5)	C7D—N1D—C1D	122.1 (5)
O3B—C2B—C3B	104.0 (5)	C11D—N2D—C10D	126.7 (6)
C1B—C2B—C3B	100.2 (5)	O1D—C1D—N1D	109.1 (5)
O2B—C3B—C2B	111.6 (5)	O1D—C1D—C2D	103.1 (5)
O2B—C3B—C4B	115.1 (5)	N1D—C1D—C2D	113.3 (5)
C2B—C3B—C4B	90.8 (5)	O3D—C2D—C1D	105.7 (5)
O1B—C4B—C5B	110.6 (6)	O3D—C2D—C3D	103.6 (5)

O1B—C4B—C6B	106.3 (5)	C1D—C2D—C3D	99.9 (5)
C5B—C4B—C6B	115.7 (5)	O2D—C3D—C4D	114.1 (5)
O1B—C4B—C3B	102.1 (5)	O2D—C3D—C2D	112.1 (5)
C5B—C4B—C3B	119.7 (6)	C4D—C3D—C2D	90.7 (5)
C6B—C4B—C3B	100.8 (5)	O1D-C4D-C5D	109.6 (5)
O6B—C5B—C4B	106.9 (5)	O1D-C4D-C6D	106.8 (5)
O3B—C6B—C4B	102.5 (5)	C5D—C4D—C6D	115.0 (5)
C8B—C7B—N1B	122 (3)	O1D—C4D—C3D	102.7 (5)
C7B—C8B—C10B	118 (2)	C5D—C4D—C3D	119.8 (5)
С7В—С8В—С9В	122 (2)	C6D—C4D—C3D	101.5 (5)
C10B—C8B—C9B	118.8 (19)	O6DC5DC4D	107.7 (5)
O4B—C10B—N2B	118.9 (5)	O3D—C6D—C4D	101.5 (5)
O4B—C10B—C8B	124.6 (12)	C8D—C7D—N1D	122.5 (6)
N2B—C10B—C8B	115.3 (12)	C7D—C8D—C10D	118.3 (6)
C8Y—C7Y—N1B	124 (2)	C7D—C8D—C9D	123.9 (6)
C7Y—C8Y—C10Y	117.8 (18)	C10D—C8D—C9D	117.7 (5)
С7Ү—С8Ү—С9Ү	123.6 (18)	O4D-C10D-N2D	120.6 (6)
C10Y—C8Y—C9Y	118.2 (14)	O4D-C10D-C8D	123.8 (6)
O4B—C10Y—N2B	118.9 (5)	N2D-C10D-C8D	115.6 (5)
O4B—C10Y—C8Y	126.1 (10)	O5D—C11D—N2D	121.9 (6)
N2B—C10Y—C8Y	114.5 (9)	O5D—C11D—N1D	122.4 (6)
O5B—C11B—N1B	122.4 (5)	N2D—C11D—N1D	115.6 (5)
08A—S1A—O6A—C5A	-21.6 (8)	C7B—N1B—C11B—N2B	8 (3)
O7A—S1A—O6A—C5A	109.1 (7)	C1B—N1B—C11B—N2B	178.4 (5)
C12A—S1A—O6A—C5A	-136.8 (7)	C10B—N2B—C11B—O5B	-177.6 (6)
C4A—O1A—C1A—N1A	-118.4 (5)	C10Y—N2B—C11B—O5B	-177.6 (6)
C4A—O1A—C1A—C2A	1.9 (6)	C10B—N2B—C11B—N1B	2.7 (9)
C11A—N1A—C1A—O1A	-171.6 (5)	C10Y—N2B—C11B—N1B	2.7 (9)
C7A—N1A—C1A—O1A	9.8 (8)	O7X—S1C—O6C—C5C	173.8 (10)
C11A—N1A—C1A—C2A	74.5 (7)	O7C—S1C—O6C—C5C	110.2 (8)
C7A—N1A—C1A—C2A	-104.2 (7)	O8X—S1C—O6C—C5C	50.5 (8)
C6A—O3A—C2A—C1A	-71.8 (7)	O8C—S1C—O6C—C5C	-24.7 (8)
C6A—O3A—C2A—C3A	33.4 (8)	C12X—S1C—O6C—C5C	-66.3 (10)
01A—C1A—C2A—O3A	69.5 (6)	C12C—S1C—O6C—C5C	-136.8 (7)
N1A—C1A—C2A—O3A	-173.0 (5)	C4C—O1C—C1C—N1C	-123.5 (5)
O1A—C1A—C2A—C3A	-38.3 (6)	C4C—O1C—C1C—C2C	0.1 (6)

N1A—C1A—C2A—C3A	79.3 (6)	C11C—N1C—C1C—O1C	-168.3 (5)
O3A—C2A—C3A—O2A	65.2 (8)	C7C—N1C—C1C—O1C	15.7 (8)
C1A—C2A—C3A—O2A	174.5 (6)	C11C—N1C—C1C—C2C	75.5 (7)
O3A—C2A—C3A—C4A	-53.6 (6)	C7C—N1C—C1C—C2C	-100.5 (7)
C1A—C2A—C3A—C4A	55.6 (5)	C6C—O3C—C2C—C1C	-71.7 (6)
C1A—O1A—C4A—C5A	165.1 (5)	C6C—O3C—C2C—C3C	34.0 (7)
C1A—O1A—C4A—C6A	-70.3 (6)	O1C—C1C—C2C—O3C	71.1 (6)
C1A—O1A—C4A—C3A	35.2 (6)	N1C—C1C—C2C—O3C	-168.5 (5)
O2A—C3A—C4A—O1A	-172.4 (6)	O1C—C1C—C2C—C3C	-37.4 (6)
C2A—C3A—C4A—O1A	-55.3 (5)	N1C—C1C—C2C—C3C	83.0 (6)
O2A—C3A—C4A—C5A	64.5 (8)	O3C—C2C—C3C—O2C	61.9 (6)
C2A—C3A—C4A—C5A	-178.5 (6)	C1C—C2C—C3C—O2C	171.9 (5)
O2A—C3A—C4A—C6A	-62.8 (7)	O3C—C2C—C3C—C4C	-54.6 (5)
C2A—C3A—C4A—C6A	54.3 (6)	C1C—C2C—C3C—C4C	55.5 (5)
S1A—O6A—C5A—C4A	171.1 (6)	C1C—O1C—C4C—C5C	165.0 (5)
01A—C4A—C5A—O6A	-59.9 (8)	C1C—O1C—C4C—C6C	-68.7 (6)
C6A—C4A—C5A—O6A	-179.9 (7)	C1C—O1C—C4C—C3C	37.0 (6)
C3A—C4A—C5A—O6A	59.0 (8)	O2C—C3C—C4C—O1C	-170.6 (5)
C2A—O3A—C6A—C4A	3.2 (8)	C2C—C3C—C4C—O1C	-56.5 (5)
O1A—C4A—C6A—O3A	67.8 (7)	O2C—C3C—C4C—C5C	67.4 (8)
C5A—C4A—C6A—O3A	-169.9 (6)	C2C—C3C—C4C—C5C	-178.5 (6)
СЗА—С4А—С6А—ОЗА	-38.3 (7)	O2C—C3C—C4C—C6C	-59.2 (7)
C11A—N1A—C7A—C8A	-0.6 (10)	C2C—C3C—C4C—C6C	54.9 (6)
C1A—N1A—C7A—C8A	178.0 (6)	S1C—O6C—C5C—C4C	176.6 (5)
N1A—C7A—C8A—C10A	-1.3 (10)	O1C—C4C—C5C—O6C	-59.4 (8)
N1A—C7A—C8A—C9A	-177.8 (7)	C6C—C4C—C5C—O6C	177.9 (6)
C11A—N2A—C10A—O4A	-179.6 (6)	C3C—C4C—C5C—O6C	58.1 (8)
C11A—N2A—C10A—C8A	-2.4 (9)	C2C—O3C—C6C—C4C	3.5 (7)
C7A—C8A—C10A—O4A	179.7 (7)	O1C—C4C—C6C—O3C	67.4 (7)
C9A—C8A—C10A—O4A	-3.6 (10)	C5C—C4C—C6C—O3C	-168.6 (6)
C7A—C8A—C10A—N2A	2.6 (9)	C3C—C4C—C6C—O3C	-38.9 (7)
C9A—C8A—C10A—N2A	179.4 (6)	C11C—N1C—C7C—C8C	1.3 (9)
C10A—N2A—C11A—O5A	-178.7 (6)	C1C—N1C—C7C—C8C	177.2 (6)
C10A—N2A—C11A—N1A	0.6 (9)	N1C-C7C-C8C-C10C	0.7 (9)
C7A—N1A—C11A—O5A	-179.7 (6)	N1C—C7C—C8C—C9C	-174.6 (6)
C1A—N1A—C11A—O5A	1.6 (9)	C11C—N2C—C10C—O4C	179.9 (7)
C7A—N1A—C11A—N2A	1.0 (8)	C11C—N2C—C10C—C8C	-1.9 (10)

C1A—N1A—C11A—N2A	-177.7 (5)	C7C—C8C—C10C—O4C	177.6 (7)
O7B—S1B—O6B—C5B	93.0 (7)	C9C—C8C—C10C—O4C	-6.7 (10)
O8B—S1B—O6B—C5B	-37.9 (7)	C7C—C8C—C10C—N2C	-0.5 (9)
C12B—S1B—O6B—C5B	-153.1 (6)	C9C—C8C—C10C—N2C	175.2 (6)
C4B—O1B—C1B—N1B	-118.2 (5)	C10C—N2C—C11C—O5C	-176.4 (7)
C4B—O1B—C1B—C2B	2.3 (6)	C10C—N2C—C11C—N1C	3.8 (10)
C11B—N1B—C1B—O1B	-167.9 (5)	C7C—N1C—C11C—O5C	176.9 (6)
C7Y—N1B—C1B—O1B	18 (2)	C1C—N1C—C11C—O5C	0.7 (9)
C7B—N1B—C1B—O1B	2 (3)	C7C—N1C—C11C—N2C	-3.4 (8)
C11B—N1B—C1B—C2B	77.7 (7)	C1C—N1C—C11C—N2C	-179.5 (5)
C7Y—N1B—C1B—C2B	-96 (2)	O7D—S1D—O6D—C5D	99.2 (6)
C7B—N1B—C1B—C2B	-112 (3)	O8D—S1D—O6D—C5D	-31.4 (7)
C6B—O3B—C2B—C1B	-72.2 (6)	C12D—S1D—O6D—C5D	-146.5 (6)
C6B—O3B—C2B—C3B	32.7 (7)	C4D—O1D—C1D—N1D	-117.2 (5)
O1B—C1B—C2B—O3B	69.3 (5)	C4D—O1D—C1D—C2D	3.5 (6)
N1B—C1B—C2B—O3B	-172.9 (5)	C11D—N1D—C1D—O1D	-173.3 (5)
O1B—C1B—C2B—C3B	-38.4 (6)	C7D—N1D—C1D—O1D	12.0 (8)
N1B—C1B—C2B—C3B	79.5 (6)	C11D—N1D—C1D—C2D	72.5 (7)
O3B—C2B—C3B—O2B	64.0 (7)	C7D—N1D—C1D—C2D	-102.2 (7)
C1B—C2B—C3B—O2B	172.6 (5)	C6D—O3D—C2D—C1D	-72.9 (6)
O3B—C2B—C3B—C4B	-53.6 (5)	C6D—O3D—C2D—C3D	31.7 (6)
C1B—C2B—C3B—C4B	55.1 (5)	O1D-C1D-C2D-O3D	68.0 (5)
C1B—O1B—C4B—C5B	162.9 (5)	N1D—C1D—C2D—O3D	-174.2 (5)
C1B—O1B—C4B—C6B	-70.7 (6)	O1D-C1D-C2D-C3D	-39.3 (5)
C1B—O1B—C4B—C3B	34.5 (5)	N1D—C1D—C2D—C3D	78.5 (6)
O2B—C3B—C4B—O1B	-169.2 (5)	O3D—C2D—C3D—O2D	63.5 (6)
C2B—C3B—C4B—O1B	-54.8 (5)	C1D—C2D—C3D—O2D	172.5 (5)
O2B—C3B—C4B—C5B	68.3 (7)	O3D—C2D—C3D—C4D	-53.0 (5)
C2B—C3B—C4B—C5B	-177.2 (6)	C1D—C2D—C3D—C4D	56.0 (5)
O2B—C3B—C4B—C6B	-59.7 (6)	C1D—O1D—C4D—C5D	162.5 (5)
C2B—C3B—C4B—C6B	54.7 (5)	C1D—O1D—C4D—C6D	-72.2 (6)
S1B—O6B—C5B—C4B	177.0 (5)	C1D—O1D—C4D—C3D	34.1 (6)
O1B—C4B—C5B—O6B	-57.0 (8)	O2D—C3D—C4D—O1D	-169.8 (5)
C6B—C4B—C5B—O6B	-178.0 (6)	C2D—C3D—C4D—O1D	-55.2 (5)
C3B—C4B—C5B—O6B	61.1 (8)	O2D—C3D—C4D—C5D	68.4 (7)
C2B—O3B—C6B—C4B	4.2 (7)	C2D—C3D—C4D—C5D	-176.9 (6)
O1B—C4B—C6B—O3B	67.0 (6)	O2D—C3D—C4D—C6D	-59.4 (6)

C5B—C4B—C6B—O3B	-169.7 (6)	C2D—C3D—C4D—C6D	55.2 (5)
C3B—C4B—C6B—O3B	-39.1 (6)	S1D-06D-C5D-C4D	166.7 (5)
C11B—N1B—C7B—C8B	-8 (6)	O1D-C4D-C5D-O6D	-55.3 (7)
C1B—N1B—C7B—C8B	-178 (3)	C6D—C4D—C5D—O6D	-175.7 (6)
N1B—C7B—C8B—C10B	-3 (6)	C3D-C4D-C5D-06D	62.9 (8)
N1B—C7B—C8B—C9B	-176 (3)	C2D—O3D—C6D—C4D	4.8 (7)
C11B—N2B—C10B—O4B	178.6 (6)	O1D-C4D-C6D-O3D	67.3 (6)
C11B—N2B—C10B—C8B	-13.3 (17)	C5D—C4D—C6D—O3D	-170.8 (6)
C7B—C8B—C10B—O4B	-180 (3)	C3D—C4D—C6D—O3D	-39.9 (6)
C9B—C8B—C10B—O4B	-7 (3)	C11D—N1D—C7D—C8D	-1.2 (10)
C7B—C8B—C10B—N2B	13 (4)	C1D—N1D—C7D—C8D	173.1 (6)
C9B—C8B—C10B—N2B	-174 (2)	N1D-C7D-C8D-C10D	-0.3 (9)
C11B—N1B—C7Y—C8Y	4 (5)	N1D-C7D-C8D-C9D	-178.1 (6)
C1B—N1B—C7Y—C8Y	178 (3)	C11D—N2D—C10D—O4D	178.3 (7)
N1B—C7Y—C8Y—C10Y	5 (5)	C11D—N2D—C10D—C8D	-4.1 (9)
N1B—C7Y—C8Y—C9Y	179 (3)	C7D—C8D—C10D—O4D	-179.8 (7)
C11B—N2B—C10Y—O4B	178.6 (6)	C9D—C8D—C10D—O4D	-1.8 (10)
C11B—N2B—C10Y—C8Y	5.7 (14)	C7D—C8D—C10D—N2D	2.7 (9)
C7Y—C8Y—C10Y—O4B	178 (2)	C9D—C8D—C10D—N2D	-179.3 (6)
C9Y—C8Y—C10Y—O4B	4 (3)	C10D—N2D—C11D—O5D	-177.2 (6)
C7Y—C8Y—C10Y—N2B	-9 (3)	C10D—N2D—C11D—N1D	2.7 (10)
C9Y—C8Y—C10Y—N2B	176.8 (14)	C7D—N1D—C11D—O5D	-180.0 (6)
C7Y—N1B—C11B—O5B	173 (2)	C1D—N1D—C11D—O5D	5.4 (10)
C7B—N1B—C11B—O5B	-171 (3)	C7D—N1D—C11D—N2D	0.2 (9)
C1B—N1B—C11B—O5B	-1.3 (9)	C1D—N1D—C11D—N2D	-174.5 (6)
C7Y—N1B—C11B—N2B	-8 (2)		

# Hydrogen-bond geometry (Å, °) for (jwe2)

<i>D</i> —H··· <i>A</i>	<i>D</i> —Н	H···A	$D \cdots A$	D—H···A
N2 <i>A</i> —H2 <i>A</i> ⋯O4 <i>B</i>	0.94 (5)	1.88 (6)	2.816 (7)	174 (6)
N2 <i>B</i> —H2 <i>B</i> ⋯O4 <i>A</i>	0.94 (5)	1.82 (6)	2.754 (6)	174 (6)
N2 <i>C</i> —H2 <i>C</i> ⋯O4 <i>D</i>	0.85 (8)	1.96 (9)	2.805 (7)	171 (8)
N2 <i>D</i> —H2 <i>D</i> ⋯O4 <i>C</i>	0.71 (8)	2.14 (8)	2.825 (7)	162 (9)
$O2A$ — $H22A$ ···O4 $C^{i}$	0.82 (6)	2.37 (9)	3.039 (8)	140 (11)
$O2A$ — $H22A$ ···O5 $D^{i}$	0.82 (6)	2.50 (11)	2.938 (9)	115 (10)
$O2B$ — $H22B$ ···O4 $D^{ii}$	0.81(5)	2.88(10)	3.163(7)	103(8)
$O2B$ —H22 $B$ ···O5 $C^{ii}$	0.81 (5)	1.99 (6)	2.774 (7)	161 (10)
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O2 <i>C</i> —H22 <i>C</i> ⋯O4 <i>A</i>	0.82 (5)	2.72 (9)	3.168 (6)	117 (8)
O2 <i>C</i> —H22 <i>C</i> ⋯O5 <i>B</i>	0.82 (5)	2.09 (7)	2.815 (7)	148 (9)
$O2D$ — $H22D$ ···· $O4B^{iii}$	0.82(5)	2.92(9)	3.141(6)	98(6)
$O2D$ — $H22D$ ···· $O5A^{iii}$	0.82 (5)	1.96 (6)	2.765 (6)	166 (9)

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

*Note:*  $D \cdots A$  *distances*  $> \sim 3.0$  Å *and*  $D - H \cdots A$  *angles*  $< \sim 110^{\circ}$  *are a bit suspect but included for* 

completeness.Least-squares planes (x,y,z in crystal coordinates) and deviations from them

## (\* indicates atom used to define plane)

7.6182 (0.0127) x + 0.5271 (0.0275) y + 9.1298 (0.0354) z = 7.0292 (0.0346)

- \* 0.0092 (0.0042) N1A
- \* -0.0005 (0.0046) C7A
- \* -0.0113 (0.0047) C8A
- \* 0.0151 (0.0043) C10A
- \* -0.0076 (0.0042) N2A \* -0.0049 (0.0040) C11A
- 0.0028 (0.0090) O4A

Rms deviation of fitted atoms = 0.0093

7.3866 (0.0362) x - 1.5814 (0.1364) y + 7.5064 (0.1735) z = 5.2045 (0.1267)

## Angle to previous plane (with approximate esd) = 12.531 ( 0.899 )

- \* 0.0695 (0.0197) N1B \* -0.0240 (0.0314) C7B \* -0.0507 (0.0258) C8B \* 0.0854 (0.0110) C10B \* -0.0470 (0.0103) N2B \* -0.0331 (0.0066) C11B
- 0.0798 (0.0195) O4B

Rms deviation of fitted atoms = 0.0556

7.9669 (0.0114) x + 0.1149 (0.0263) y + 7.5607 (0.0383) z = 10.4615 (0.0234)

- \* 0.0116 (0.0041) N1C
- \* 0.0029 (0.0042) C7C
- \* -0.0095 (0.0045) C8C
- \* 0.0012 (0.0045) C10C
- \* 0.0136 (0.0045) N2C
- \* -0.0197 (0.0042) C11C

0.0334 (0.0100) 04C

Rms deviation of fitted atoms = 0.0116

7.5840 (0.0126) x - 0.5106 (0.0272) y + 8.2675 (0.0365) z = 9.6143 (0.0317)

## Angle to previous plane (with approximate esd) = 4.848 ( 0.329 )

- \* -0.0099 (0.0043) N1D
- \* 0.0070 (0.0043) C7D
- \* 0.0068 (0.0045) C8D
- \* -0.0191 (0.0041) C10D
- \* 0.0178 (0.0044) N2D
- \* -0.0027 (0.0045) C11D -0.0260 (0.0091) O4D

Rms deviation of fitted atoms = 0.0121



Plot 10. The four independent molecules in the unit cell of **11** shown in similar orientations. Minor components of disorder shown with dashed bonds.



Plot 11. One complete asymmetric unit. Hydrogen bonds shownas dashed red bonds, minor components of disorder shown as dashed black lines.



Plot 12. Perspective view of molecules A and B. Non-hydrogen atoms drawn with 50% probability ellipsoids, hydrogen bonds shown as dashed red bonds. Minor component of disorder omitted for clarity.



Plot 13. Perspective view of molecules C and D. Non-hydrogen atoms drawn with 50% probability ellipsoids, hydrogen bonds shown as dashed red bonds. Minor component of disorder omitted for clarity.



Plot 14. Section of hydrogen bonded sheet. In the lower plot the carbon atoms are colour-coded to indicate the different independent molecules



Plot 15. Unit cell packing plot viewed down the *b* axis. Hydrogen bonds shown in red.



Plot 16. Unit cell packing plot viewed down the *a* axis. Hydrogen bonds shown in red.



Plot 17. Unit cell packing plot viewed down a body diagonal to show 2D H-bonded layers. Hydrogen bonds shown in red.



Plot 18. Comparison of shown in red.ed down a body diagonal to s