

Substrate flexibility of a 2,6-dideoxyglycosyltransferase

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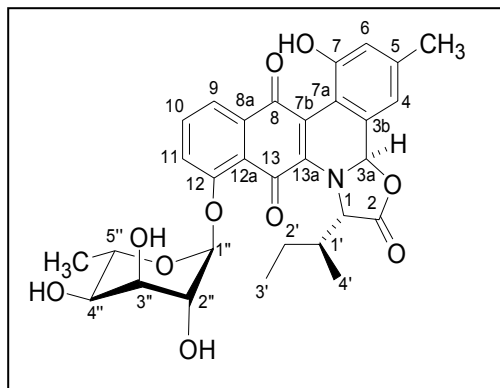
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Spectroscopic data

ILEVS1080 3aR (35%)

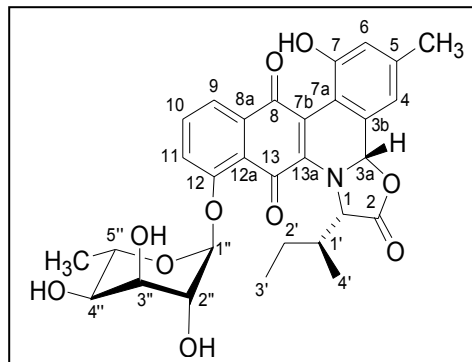
m/z (ESI) = 566.2 (M+H)⁺; MS/MS = 420.1 [aglycone, (M+H)⁺], 306.0 [phenanthroviridin, (M+H)⁺];
mass calcd for C₂₇H₂₆NO₉ (M+Na)⁺ 588.1846; found 588.1848.
UV (methanol): $\lambda_{\max}$ (ϵ) = 210 (35312), 317 (18890), 519 (2887); R_f = 0.34 (DCM:MeOH:H₂O, 85.9:4:0.1)
Solvent: acetone-d₆



Position	δ ¹ H [ppm]	Multiplicity (J[Hz])	δ ¹³ C [ppm]	HMBC	COSY	NOESY
1	5.43	d (3.5)	62.42		H-1'	H-1', 4'-CH ₃
2						
3a	6.74	s	87.64			4'-CH ₃
3b						
4	6.78	s	113.62		5-CH ₃	5-CH ₃
5			142.10			
5-CH ₃	2.35	s	20.41	4, 5, 6	4, 6	4, 6
6	6.81	s	120.15		5-CH ₃	5-CH ₃
7						
7-OH	10.90	s				
7a						
7b						
8						
8a						
9	7.91	m*	121.20		10	10
10	7.85	t (8.0)	135.30		9, 11	9, 11
11	7.64	d (8.5)	120.60		10	1'', 10
12						
12a						
13						
13a						
1'	2.17	m			H-2'a, H-2'b, 4'-CH ₃	H-1, 4'-CH ₃
2'a	1.86	m			H-1', H-2'b, 3'-CH ₃	H-2'b, 3'-CH ₃
2'b	1.53	m			H-1', H-2'a, 3'-CH ₃	H-2'a, 3'-CH ₃
3'	1.10	t (7.0)	11.31		H-2'a, H-2'b	H-2'a, H-2'b
4'	1.07	d (7.0)	14.14		H-1'	H-1, H-1', H-3a
1''	5.80	d (0.5)	97.95		H-2''	H-2'', H-11
2''	4.20	m	70.15		H-1'', 2''-OH, H-3''	H-1'', 2''-OH, H-3''
2''-OH	4.77	brd (5.0)			H-2''	H-2''
3''	3.94	dt (3.0, 11.0)	70.90		H-2'', 3''-OH, H-4''	H-2'', H-4''
3''-OH	4.50	d (10.5)			H-3''	H-3'', H-5''
4''	3.56	ddd (3.0, 10.0)	69.39		H-3'', 4''-OH, H-5''	H-3'', 4''-OH, 5''-CH ₃
4''-OH	3.36	d (10.0)			H-4''	H-4''
5''	3.85	dq (6.0, 10.0)	65.96		H-4'', 5''-CH ₃	3''-OH, 5''-CH ₃
5''-CH ₃	1.21	d (6.0)	17.27		H-5''	H-4'', H-5''

*signal obscured by peaks from major diastereoisomer

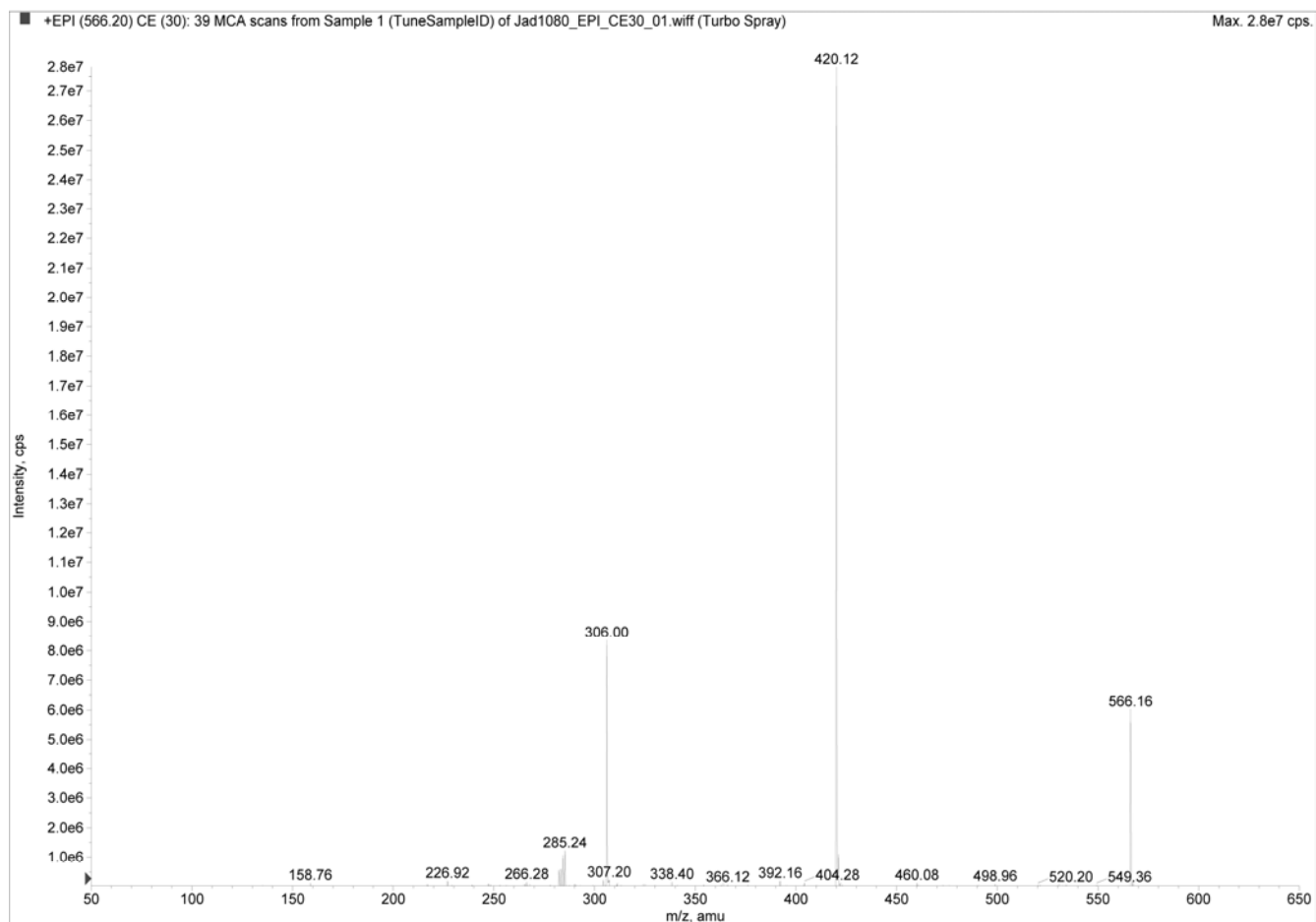
ILEVS1080 3aS (65%)



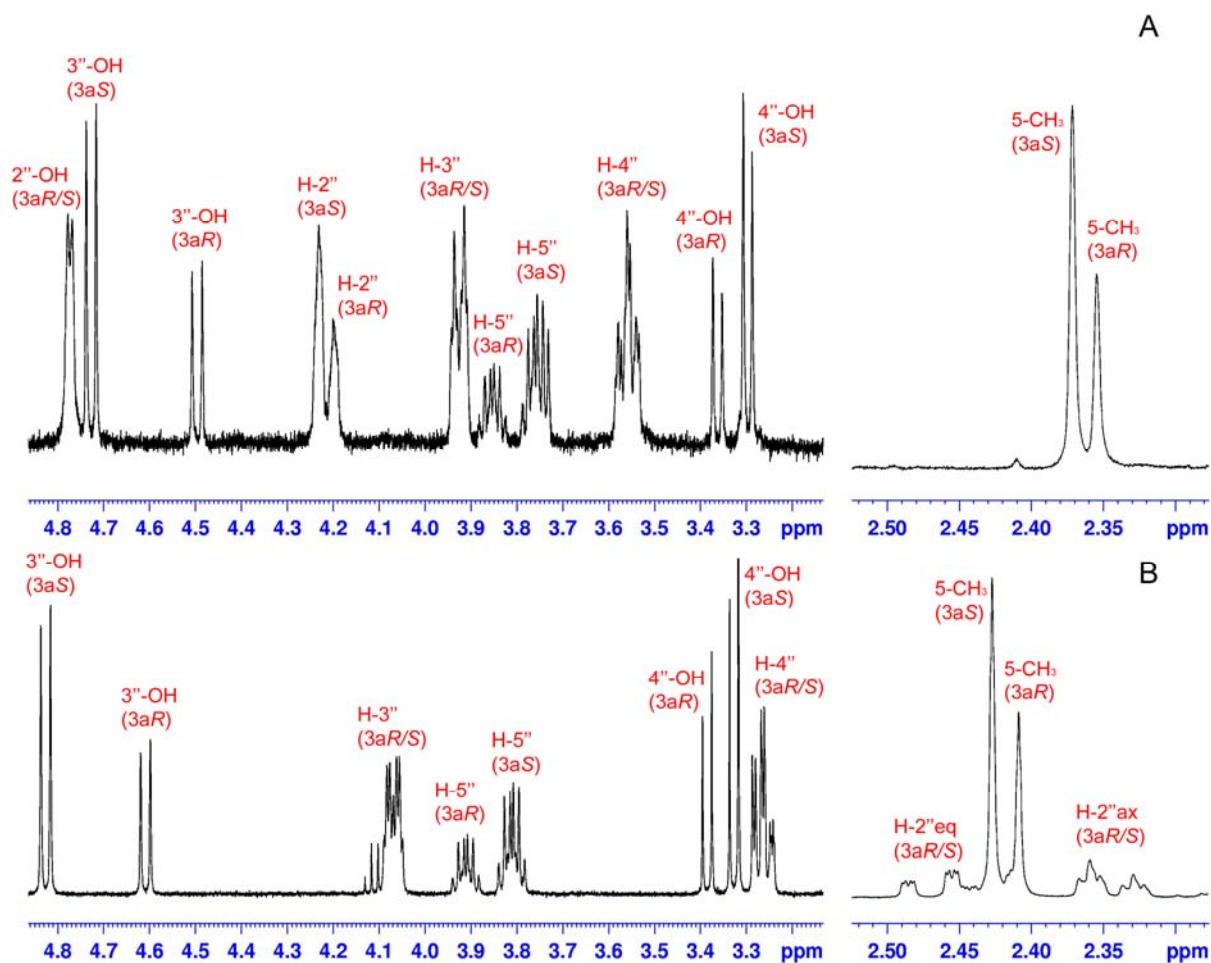
Position	$\delta^1\text{H}$ [ppm]	Multiplicity (J[Hz])	$\delta^{13}\text{C}$ [ppm]	HMBC	COSY	NOESY
1	5.41	d (3.5)	62.42		H-1'	H-1', 4'-CH ₃
2						
3a	6.34	s	87.21			
3b						
4	6.79	s	113.62		5-CH ₃	5-CH ₃
5			142.10			
5-CH ₃	2.37	s	20.41	4, 5, 6	4, 6	4, 6
6	6.84	s	120.15		5-CH ₃	5-CH ₃
7						
7-OH	10.25	s				
7a						
7b						
8						
8a						
9	7.97	d (7.5)	121.20		10	10
10	7.89	t (8.5)	135.30		9, 11	9, 11
11	7.70	d (8.5)	120.60		10	1'', 10
12						
12a						
13						
13a						
1'a	1.99	m			H-2'a, H-2'b, 4'-CH ₃	H-1, 4'-CH ₃
2'a	1.86	m			H-1', H-2'b, 3'-CH ₃	H-2'b, 3'-CH ₃
2'b	1.53	m			H-1', H-2'a, 3'-CH ₃	H-2'a, 3'-CH ₃
3'	1.07	t (7.5)	11.31		H-2'a, H-2'b	H-2'a, H-2'b
4'	0.48	d (7.0)	13.69		H-1'	H-1'
1''	5.88	brs	97.52		H-2''	H-2'', H-11
2''	4.23	m	70.15		H-1'', 2''-OH, H-3''	H-1'', 2''-OH, H-3''
2''-OH	4.77	brd (5.0)			H-2''	H-2''
3''	3.94	dt (3.0, 11.0)	70.90		H-2'', 3''-OH, H-4''	H-2'', H-4''
3''-OH	4.73	d (11.0)			H-3''	H-3'', H-5''
4''	3.56	ddd (3.0, 10.0)	69.39		H-3'', 4''-OH, H-5''	H-3'', 4''-OH, 5''-CH ₃
4''-OH	3.30	d (10.0)			H-4''	H-4''
5''	3.75	dq (6.0, 10.0)	65.96		H-4'', 5''-CH ₃	3''-OH, 5''-CH ₃
5''-CH ₃	1.18	d (6.0)	17.27		H-5''	H-4'', H-5''

Figure 1S. EPI scan of ILEVS1080

The sample for MS analysis was prepared as described previously.¹



Expanded Figure 2



References

- (1) Jakeman, D. L.; Graham, C. L.; Reid, T. R. *Bioorg. Med. Chem. Lett.* **2005**, *15*, 5280-5283.