Strategies for introducing sulfur atom in a sugar ring: Synthesis of 5-thioaldopyranoses and their NMR data

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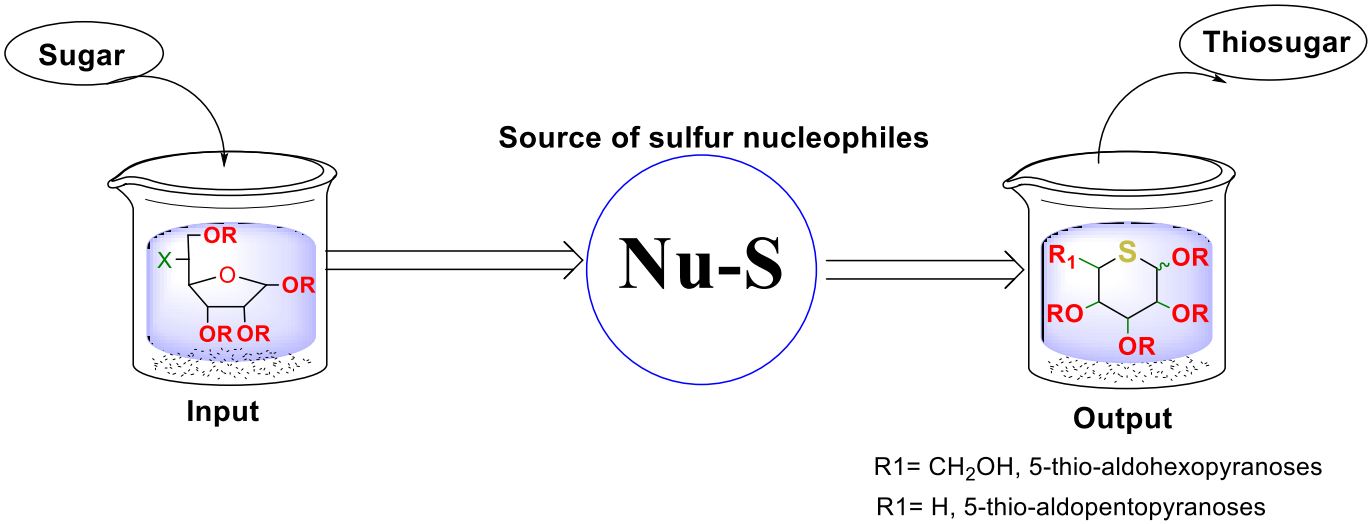
Abstract

Thiosugars containing a sulfur atom as heteroatom in the sugar ring are highly interesting. In particular, 5-thioaldopyranoses have attracted considerable attention because they exhibit biologically important properties as the substrates for glycosidases also can be developed as potential therapeutical agents, such as antineoplastic, anti-diabetic, antiviral, and antithrombotic agents. In this review, we describe and discuss the advances in synthetic strategies for the preparation of 5-thioaldopyranoses (5-thio-D-hexoses, 5-thio-L-hexoses, 5-thio-D-pentoses and 5-thio-L-pentoses), as well as their spectroscopic NMR data as known to date. The study is aiming to give readers an easy understanding of thiosugars chemistry and provide a useful guide for the carbohydrate chemists.



S1. NMR of 5-thio-D-aldohexopyranoses

Table 1. ^{1}H-NMR chemical shifts" and couplings constant (in parenthesis in Hz) of **5-thio-D-aldohexopyranoses**

Compound	**H-1**	**H-2**	**H-3**	**H-4**	**H-5**	**H-6a**	**H-6b**	Ref.
	($J_{1,2}$)	($J_{2,3}$)	($J_{3,4}$)	($J_{4,5}$)	($J_{5,6a}$)	($J_{5,6b}$)	($J_{6a,6b}$)	
5-thio-α-D-Glc	5.00	3.78	3.65	3.60	3.23	3.87	3.91	[3, 12]
	(3.1)	(9.4)	(8.9)	(9.1)	(3.0)	(5.1)	(11.4)	
5-thio-β- D-Glc	4.77	3.78	3.65	3.60	2.98	3.87	3.91	[12]
	(9.3)	(9.4)	(8.9)	(9.8)	(3.6)	(6.2)	(11.4)	
5-thio-α-D-Gal	4.99	3.97	3.72-3.81	4.23	3.51	3.72-3.81	3.59-3.65	[6]
	(3.2)	(10.4)		(1.2)	(7.6)			
5-thio-β-D-Gal	4.77	3.72-	3.72-3.81	4.13	3.29	3.59-3.65	3.44	[6]
	(9.2)	(3.81)		(1.2)	(2.6)	(7.6)	(11.1)	
5-thio-α-D-Man	4.77	4.05	3.68	3.77	3.17	3.77	3.89	[2, 7]
	(3.9)	(2.7)	(9.3)	(9.6)	(4.2)	(6.3)	(11.1)	
5-thio-β-D-Man	5.02							[2, 7]
	(1.8)							

solvent: D_2O

Table 2. ^{1}H-NMR of **peracetylated 5-thio-D-aldohexopyranoses**

Compound	**H-1**	**H-2**	**H-3**	**H-4**	**H-5**	**H-6a**	**H-6b**	Other signals	Ref.
	($J_{1,2}$)	($J_{2,3}$)	($J_{3,4}$)	($J_{4,5}$)	($J_{5,6a}$)	($J_{5,6b}$)	($J_{6a,6b}$)		
5-thio-α-D-Glc	6.1	5.24	5.43	5.33	3.59	4.07	4.37	2.18-1.99	[5]
	(2.9)		(9.8)	(10.8)	(2.9)	(4.9)	(12.2)	(5 OAc)	
5-thio-α-D-Gal	6.18	5.45	5.30	5.71	3.80	3.98	4.11	2.17-1.99	[6]
	(3.2)	(10.8)	(2.8)	(1.6)	(6.0)	(8.8)	(11.2)	(5 OAc)	
5-thio-β-D-Gal	5.85	5.43	5.01	5.62	3.43	4.26	4.32	2.15-2.06	[6]
	(6.8)	(6.8)	(2.8)	(2.8)	(6.8)	(8.0)	(11.2)	(5 OAc)	
5-thio-α-D-Man	5.85	5.38	5.28	5.49	3.55	4.09	4.32	2.20-2.02	[2, 7]
	(4.0)	(3.0)	(10.0)	(10.0)	(5.8)	(3.5)	(12.0)	(5 OAc)	
5-thio-β-D-Man	6.02	5.54	5.22	5.40	3.18	4.47	4.54	2.1-2.03	[2, 7]
	(3.4)	(3.4)	(5.1)	(5.1)	(7.0)	(8.4)	(11.5)	(5 OAc)	
5-thio-β-D-Altro	6.16	5.51	5.40	5.66	3.21	4.47	4.47	2.18-2.01	[8]
	(2.5)	(10.5)	(2.0)	(3.5)	(8.0)	(8.0)		(5OAc)	
5-thio-β-D-Allo	6.11	5.29	5.58	5.20	3.66	4.22	4.24	2.16-2.01	[9]
	(9.5)	(2.5)	(2.5)	(10.5)	(4.0)	(5.0)		(5 OAc)	

solvent: $CDCl_3$

Table 3. ^{13}C-NMR chemical shifts" of **5-thio-D-aldohexopyranoses**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	**C-6**	Ref.
5-thio-α-D-Glc	75.4	77.6	75.9	75.9	45.4	62.6	[3, 12]
5-thio-β-D-Glc	76.4	78.5	76.8	76.9	46.3	63.4	[3, 12]
5-thio-α-D-Gal	97.2	73.0	71.7	70.6	39.8	60.9	[6]
5-thio-β-D-Gal	73.3	71.1	70.5	70.0	43.3	61.1	[6]
5-thio-α-D-Man	77.8	74.7	73.5	72.0	45.6	62.9	[2, 7]
5-thio-α-D-Allo	76.0	74.1	72.3	72.3	39.9	61.4	[9]
5-thio-β-D-Allo	76.3	75.8	73.2	72.2	45.3	62.1	[9]
5-thio-α-D-Altro	75.4	74.0	73.3	70.0	44.9	62.8	[8]
5-thio-β-D-Altro	75.1	72.2	71.6	69.1	48.8	64.7	[8]

solvent: D_2O

Table 4. ^{13}C-NMR chemical shifts" of **methyl 5-thio-D-aldohexopyranosides**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	**C-6**	Other signals	Ref.
Methyl 5-thio-α-D-Allo	85.4	75.3	72.6	72.3	39.1	61.5	57.8 (OMe)	[9]
Methyl 5-thio-β-D-Allo	84.2	74.9	72.8	71.8	45.7	62.7	59.0 (OMe)	[9]
Methyl 5-thio-α-D-Altro	86.0	73.4	72.8	69.6	42.8	62.6	58.0 (OMe)	[8]
Methyl 5-thio-β-D-Altro	86.2	71.7	71.6	68.3	49.8	64.7	57.3 (OMe)	[8]

solvent: D_2O

Table 5. ^{13}C-NMR chemical shifts" of **peracetylated of 5-thio-D-aldohexopyranosides**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	**C-6**	Ref.
5-thio-α-D-Glc	97.2	73.0	71.7	70.6	39.8	60.9	[5]
5-thio-α-D-Man	72.6	69.6	68.5	70.0	40.2	61.6	[2, 7]
5-thio-β-D-Man	69.7	68.4	68.2	68.0	40.0	64.1	[2, 7]

solvent: $CDCl_3$

S2. NMR of 5-thio-L-aldohexopyranoses

Table 6. ^{1}H-NMR chemical shifts" and couplings (in parenthesis in Hz) of **5-thio-L-aldohexopyranoses** and some of their **methyl glycosides**

Compound	**H-1** ($J_{1,2}$)	**H-2** ($J_{2,3}$)	**H-3** ($J_{3,4}$)	**H-4** ($J_{4,5}$)	**H-5** ($J_{5,6a}$)	**H-6a** ($J_{5,6b}$)	**H-6b** ($J_{6a,6b}$)	Other signals	Ref.
5-thio-α-L-Ido	4.96	3.61	4.04	3.56	3.12	3.90	3.80		[11]
	(7.1)	t, (8.6)	t, (9.7)	t, (8.7)			(11.5)		
5-thio-β- L-Ido	4.95	3.78	4.02	3.88	3.20	4.01	3.92		[11]
	(3.4)	(9.3)	t, (9.1)	t, (9.2)	(6.7)	(4.7)	(11.5)		
Methyl 5-thio-α-L-Ido	4.57	3.83	4.03	3.87	3.06	4.04	3.80	3.43 (OMe)	[10]
	(3.5)	(9.5)	(9.5)	(5.0)	(9.0)	(5.9)	(11.0)		
Methyl 5-thio-β- L-Ido	4.67	3.69	4.00	3.60	3.19	3.90	3.77	3.05 (OMe)	[10]
	(7.5)	(8.5)	(8.0)	(4.7)	(8.0)	(4.7)	(12.0)		
5-thio-α-L-Gal	5.10	4.09	3.95-3.85	4.35	3.62	3.95-3.85	3.74		[4]
	(3.3)	(10.2)	(2.8)	(1.3)	(7.3)	(6.8)	(11.3)		
5-thio-β-L-Gal	4.9	3.95-3.85	3.55	4.25	3.39	3.95-3.85	3.72		[4]
		(9.5)	(3.0)	(1.4)	(7.6)	(7.3)	(11.6)		
5-thio-α-L-Fuco	4.90	3.93	3.80	4.04	3.49	1.09			[1]
	(3.2)	(10.2)	(2.9)		(7.0)	d, 3H, CH_3 (J=7.0)			
5-thio-β-L-Fuco	4.45	3.47	3.10	3.64	3.01	1.05			[1]
	(8.8)	(10.0)	(2.9)	(1.6)	(7.0)	d, 3H, CH_3 (J=7.0)			

Solvent: D_2O

Table 7. ^{1}H-NMR chemical shifts of **peracetylated 5-thio-L-aldohexopyranoses**

Compound	**H-1** ($J_{1,2}$)	**H-2** ($J_{2,3}$)	**H-3** ($J_{3,4}$)	**H-4** ($J_{4,5}$)	**H-5** ($J_{5,6a}$)	**H-6a** ($J_{5,6b}$)	**H-6b** ($J_{6a,6b}$)	Other signals	Ref
5-thio-α-L-Allo	6.08 (3.5) Hz	5.18 (3.0) Hz	5.60 (3.1) Hz	5.15 (11.0) Hz	3.79 (5.0) Hz	4.38 (3.0) Hz	4.19 (12.0)	2.16-1.99 (5 OAc)	[13]
5-thio-β-L-Allo	6.12 (9.0) Hz	5.30 (2..6) Hz	5.59 (2.5) Hz	5.20 (10.5) Hz	3.67 (5.5) Hz	4.28 (3.5) Hz	4.20 (12.0)	2.14-1.99 (5 OAc)	[13]
5-thio-α-L-Fuco	6.11 (3.3)	5.46 (10.9)	5.31 (3.0)	5.55 (1.6)	3.64 (6.9)	1.16 d, 3H, CH_3 (J=6.9)		2.19-2.00 (4 OAc)	[1]
5-thio-β-L-Fuco	6.06 (2.8)	5.43 (11.2)	5.26 (3.0)	5.54 (1.5)	3.64 (7.0)	1.17 d, 3H, CH_3 (J=7.0)		2.18-1.97 (4 OAc)	[1]
5-thio-α-L-Gal	6.15 (2.7)	5.39 (10.6)	5.29 (2.6)	5.70 (1.6)		4.4-3.8 (3H)		2.2-2.0 (5 OAc)	[4]
5-thio-β-L-Gal	5.85 (7.0)	5.20 (7.6)	5.02 (3.0)	5.61 (3.0)		4.4-3.80 (3H)		2.2-2.0 (5 OAc)	[4]

solvent: $CDCl_3$

Table 8. ^{13}C-NMR Data for **5-thio-L-aldohexopyranoses**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	**C-6**	**Ref.**
5-thio-α-L-Ido	72.3	77.6	73.3	73.0	45.8	59.5	[10, 11]
5-thio-β-L-Ido	73.5	75.6	73.6	70.2	46.1	61.2	[10, 11]
5-thio-α-L-Allo	74.9	73.3	70.9	71.1	38.2	60.2	[13]
5-thio-β-L-Allo	75.2	74.4	70.8	71.9	44.1	60.9	[13]
5-thio-α-L-Fuco	75.6	75.1	72.2	71.5	37.4	17.0 (CH_3)	[1, 11]
5-thio-α-L-Gal	73.8	71.6	70.9	70.5	43.8	61.5	[4]

Solvent: D_2O

Table 9. ^{13}C-NMR Data for **peracetylated of 5-thio-L-aldohexopyranoses**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	**C-6**	Other signals	Ref.
5-thio-α-L-Allo	71.4	69.7	69.3	69.6	40.2	61.4	170.4-168.9 (5 CO), 20.6-20.5 (5 OAc)	[13]
5-thio-β-L-Allo	68.8	70.1	69.8	70.0	35.4	60.9	170.6-169.2 (5 CO), 20.9-20.4 (5 OAc)	[13]
5-thio-α-L-Fuco	72.2	71.9	69.2	69.0	35.6	15.7 (CH_3)	170.5-169.1 (4 CO), 20.8-20.5 (4 OAc)	[1]
5-thio-α-L-Gal	71.3	69.4	68.7	68.1	39.6	61.1	170.1, 169.9, 169.8, 169.0 (5 CO), 20.9-20.6 (5 OAc)	[4]
Methyl 5-thio-α-L-Ido	87.1	77.6	72.4	76.6	49.1	63.7	58.6 (OMe)	[10]
Methyl 5-thio-β-L-Ido	85.5	77.5	75.1	75.0	46.6	62.2	60.3 (OMe)	[10]

solvent: $CDCl_3$

S3. NMR of 5-thio-D-aldopentopyranoses

Table 10. ^{1}H-NMR chemical shifts" and couplings constant (in parenthesis in Hz) for **5-thio-D-aldopentopyranoses**

Compound	**H-1** ($J_{1,2}$)	**H-2** ($J_{2,3}$)	**H-3** ($J_{3,4}$)	**H-4** ($J_{4,5a}$)	**H-5a** ($J_{4,5b}$)	**H-5b** ($J_{5a,5b}$)	Ref.
5-thio-α-D-Xylo	4.92	3.76	3.58	3.73	2.59	2.88	[20]
	(2.2)	(9.7)	(9.7)	(11.3)	(3.0)	(13.2)	
5-thio-β-D-Xylo	4.80	3.63	3.48	3.63	2.75	2.47	[16]
	(9.1)	(9.3)	(9.3)	(11.2)	(4.3)	(13.0)	
5-thio-β-D-Arab	4.96	3.98	3.78	4.28	3.19	2.58	[17]
	(3.0)	(9.5)	(2.5)	(4.5)	(2.5)	(14.5)	
5-thio-α-D-Ribo	4.73	3.80	3.96	3.93	2.79	2.61	[19]
	(3.1)	(3.1)	(2.5)	(3.5)	(9.0)	(13.6)	
5-thio-β-D-Ribo	4.82	3.70	3.88	3.85	2.88	2.37	[19]
	(7.6)	(2.6)	2.45	(4.0)	(11.0)	(13.2)	
5-Thio-α-D-Lyxo	4.84	4.12	3.66	3.90	2.80	2.67	[17]
	(4.0)	(3.0)	(9.0)	(5.0)	(9.5)	(13.0)	

solvent: D_2O

Table 11. ^{1}H-NMR Data for **Methyl 5-thio-D-aldopentopyranosides**

Compound	**H-1** ($J_{1,2}$)	**H-2** ($J_{2,3}$)	**H-3** ($J_{3,4}$)	**H-4** ($J_{4,5a}$)	**H-5a** ($J_{4,5b}$)	**H-5b** ($J_{5a,5b}$)	Other signals	Ref.
5-thio-α-D-Arab	4.42 (7.0)	3.97 (7.0)	3.60 (3.0)	4.17 (5.5)	2.79 (3.5)	2.73 (14.0)	3.49 (OMe)	[17]
5-thio-β-D-Arab	4.56 (3.0)	4.02 (10.0)	3.73 (3.0)	4.26 (1.5)	3.02 (4.0)	2.50 (14.5)	3.43 (OMe)	[17]
5-Thio-α-D-Lyxo	4.52 (4.0)	4.19 (3.0)	3.56 (9.5)	3.91 (5.5)	2.62 (9.0)	2.54 (13.0)	3.44 (OMe)	[17]
5-Thio-β-D-Lyxo	4.62 (2.5)	4.18 (3.0)	3.68 (6.0)	4.08 (3.0)	3.00 (6.0)	2.42 (14.4)	3.47 (OMe)	[17]

Solvent: $CDCl_3$

Table 12. ^{1}H-NMR Data for **peracetylated 5-thio-D-aldopentopyranosides**

Compound	**H-1** ($J_{1,2}$)	**H-2** ($J_{2,3}$)	**H-3** ($J_{3,4}$)	**H-4** ($J_{4,5a}$)	**H-5a** ($J_{4,5b}$)	**H-5b** ($J_{5a,5b}$)	Other signals	Ref.
5-thio-α-D-Xylo	6.08 (3.1)	5.21 (10.2)	5.44 (9.9)	5.12 (11.3)	3.00 (4.6)	2.80 (13.1)	2.18- 2.03 (4 OAc)	[15]
5-thio-α-D-Arab	5.67 (4.5)	5.29 (5.5)	5.09 (2.5)	5.36 (9.5)	3.18 (3.0)	2.58 (13.0)	2.13- 2.09 (4 OAc)	[17]
5-thio-β-D-Arab	6.11 (3.0)	5.50 (10.5)	5.29 (3.0)	5.52 (1.5)	3.29 (4.5)	2.75 (14.5)	2.18-2.01 (4 OAc)	[14,17]
5-thio-α-D-Ribo	5.90 (4.70)	5.12 (2.0)	5.52 (2.5)	5.10 (3.4)	3.16 (11.9)	2.44 (12.9)	2.10-1.90 (4 OAc)	[18]
5-thio-β-D-Ribo	5.97 (8.5)	5.12 (2.0)	5.45 (2.5)	5.10 (3.6)	3.04 (9.8)	2.71 (13.4)	2.10-1.90 (4 OAc)	[18]

solvent: $CDCl_3$

Table 13. ^{13}C-NMR Data for **5-thio-D-aldopentopyranoses**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	Ref.
5-thio-α-D-Xylo	74.6	78.0	76.9	73.5	30.0	[16, 20]
5-thio-β-D-Xylo	73.5	75.8	74.0	73.5	27.4	[16]
5-thio-β-D-Arab	74.9	72.4	70.9	70.7	29.9	[17]
5-thio-α-D-Ribo	73.9	74.1	71.6	70.6	28.5	[19]
5-thio-β-D-Ribo	73.0	74.5	71.3	70.2	22.7	[19]

solvent: D_2O

Table 14. ^{13}C-NMR Data for **Methyl 5-thio-D-aldopentopyranosides**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	Other signals	Ref.
Methyl 5-thio-α-D-Xylo	84.6	76.3	75.2	74.2	27.9	57.2 (OMe)	[17]
Methyl 5-thio-β-D-Xylo	85.4	77.5	77.4	73.7	30.2	59.4 (OMe)	[17]
Methyl 5-thio-α-D-Arab	85.6	74.6	73.8	69.0	29.0	58.6 (OMe)	[17]
Methyl 5-thio-β-D-Arab	85.4	72.2	71.2	70.6	29.8	57.4 (OMe)	[17]
Methyl 5-Thio-α-D-Lyxo	87.1	73.5	73.2	69.9	28.6	57.0 (OMe)	[17]
Methyl 5-Thio-β-D-Lyxo	85.0	74.1	70.6	70.6	26.5	58.3 (OMe)	[17]

solvent: $CDCl_3$

Table 15. ^{13}C-NMR Data for **peracetylated 5-thio-D-aldopentopyranoses**

Compound	**C-1**	**C-2**	**C-3**	**C-4**	**C-5**	Other signals	Ref.
5-thio-α-D-Xylo	73.2	72.4	70.8	69.9	26.8		[15]
5-thio-β-D-Arab	71.9	69.0	68.3	69.9	27.8	170.4-169.3 (4 CO), 21.0-20.6 (4 Ac)	[17]
5-thio-α-D-Ribo	70.1	70.0	68.8	68.4	21.8	170.0-169.0 (4 CO), 20.8-20.2 (4 Ac)	[18]

solvent: $CDCl_3$

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