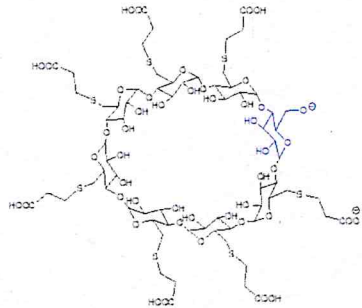




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 Web: www.spincosynthesis.com

CERTIFICATE OF ANALYSIS

Page 1 of 1

NAME OF THE PRODUCT/ IMPURITY (IUPAC Name)		SUGAMMADEX MONOHYDROXY IMPURITY IUPAC NAME: Heptakis(6-deoxy-6-(2-carboxyethyl)thio)-gamma-cyclodextrin
CAS. No.	: 2376607-98-4	STRUCTURE 
BATCH No.	: SRD054/SUG-IMP3-II/019/0622	
MFG. DATE	: JUN-2022	
AR No.	: SRD054/SUG-IMP3-II/019/0622/AR23-3851	
DATE OF ANALYSIS	: 20 JUN 2023	
RETEST DATE	: 19 JUN 2024	
GRADE	: IN-HOUSE	
BATCH QUANTITY	: 10mg	

S. No.	TESTS	SPECIFICATIONS	RESULTS
1	Description	Report the result	White solid
2	Identification by MS	The exact mass of Sugammadex Mono Hydroxy impurity should be confirmed as 1912Da	Mass spectral data confirms the exact mass of Sugammadex Mono Hydroxy impurity as 1912Da
3	Identification by ¹ H & ¹³ C NMR	The molecular structure of Sugammadex Mono Hydroxy impurity should be confirmed by ¹ H & ¹³ C NMR spectra	¹ H & ¹³ C NMR spectra confirms the molecular structure of Sugammadex Mono Hydroxy impurity
4	Purity by HPLC	For Informative	90.09% Area
5	Volatile loss by TGA	For Informative	0.47% w/w
6	Potency	For Informative	89.62% w/w

Storage condition: (i) For long term: Storage at 2 to 8°C.
 (ii) For transport: Ambient temperature.

Remarks: The product **conforms** / **does not conform** to the above specification.

Prepared By (Sign& Date)	<i>Gokul S</i> 21 Jun 23	Reviewed By (Sign& Date)	<i>L. L. M</i> 21 Jun 2023	Approved By (Sign& Date)	<i>N. Aashish</i> 21 Jun 2023
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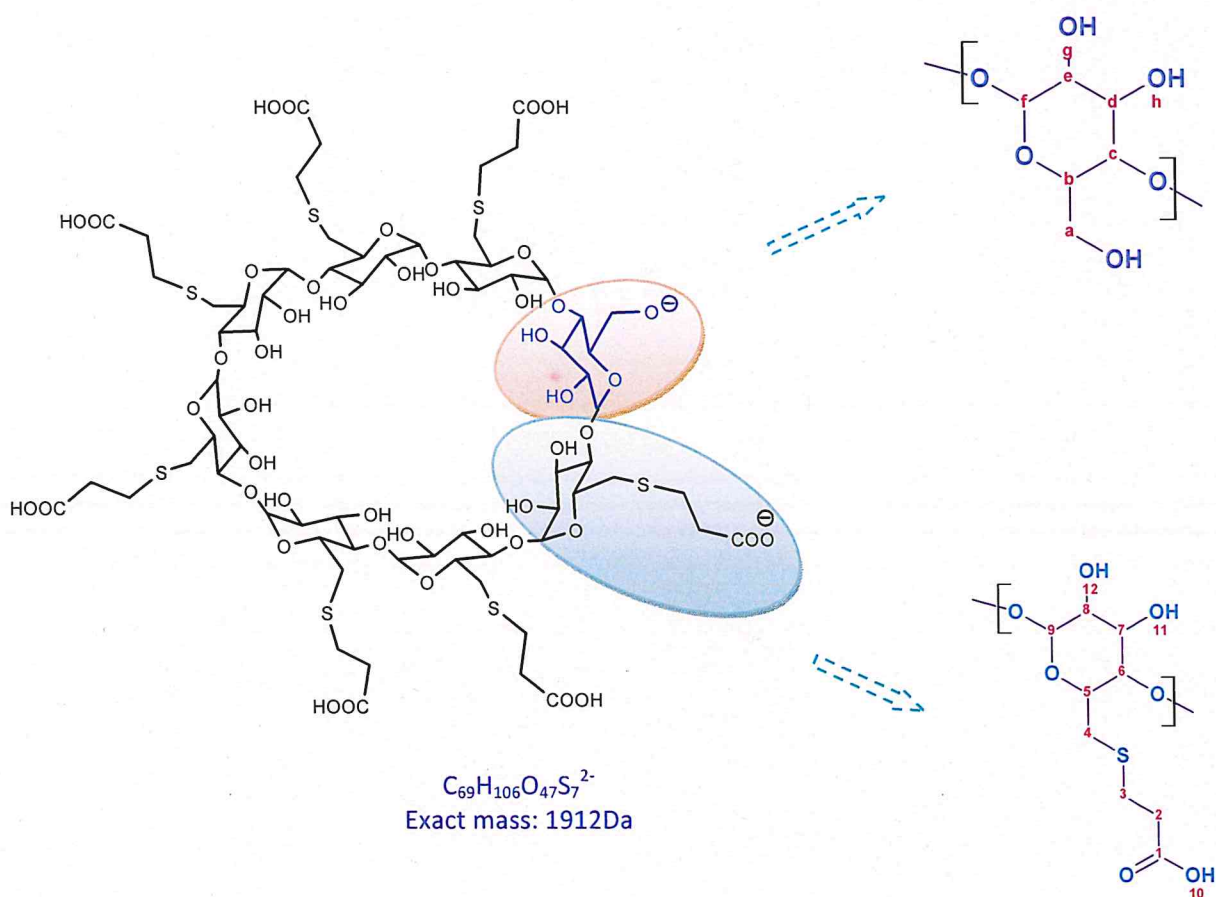
^1H & ^{13}C NMR Spectral data

Conditions: 200MHz, DMSO- d_6 (^1H NMR)

Conditions: 500MHz, DMSO- d_6 (^{13}C NMR)

The structure of Sugammadex Monohydroxy Impurity (SRD054/SUG-IMP3-II/019/0622) is confirmed with the signals of the ^1H & ^{13}C spectra and their respective interpretation.

Sugammadex Monohydroxy Impurity



Gokul
21 Jun 23

L. L. L.
21 Jun 2023

The assignment of ^1H and ^{13}C NMR signals of Sugammadex Monohydroxy Impurity

Position ^a	^1H	^1H δ (ppm)	Multiplicity ^b	^{13}C δ (ppm)	^{13}C
1	--	--	--	173.113,173.044	COOH
2 ^c	14H	2.48-2.511	m	33.130	CH ₂
3	14H	2.703-2.768	t	27.921	CH ₂
4	7H	2.842-2.934	m	34.568	CH ₂
	7H	3.006-3.069	d		
a	2H	3.686	brs	59.899	CH ₂
5	7H	3.332-3.413	m	72.519	CH
b	1H	3.332-3.413	m	72.393	CH
6	7H	3.762-3.805	t	71.385	CH
c	1H	3.762-3.805	t	71.472	CH
7	7H	3.522-3.609	t	72.612	CH
d	1H	3.522-3.609	t	72.393	CH
8	7H	3.332-3.413	m	84.022-84.243	CH
e	1H	3.332-3.413	m	80.700	CH
9	7H	4.902	brs	102.371,102.069	CH
f	1H	4.902	brs	101.427	CH
10	7H	12.158	brs	--	COOH
11 & 12	14H	5.823-5.912	brs	--	OH
g & h	2H	5.823-5.912	brs	--	OH

^a Carbon, Hydrogen and nitrogen numbering - only for ^1H & ^{13}C NMR spectral data assignment,

^b Resonance multiplicities, coupling constants, and coupled partners in parentheses: (t) Triplet, (m) Multiplet, (brs) Singlet Broad, (s) Singlet,

(d) Doublet,

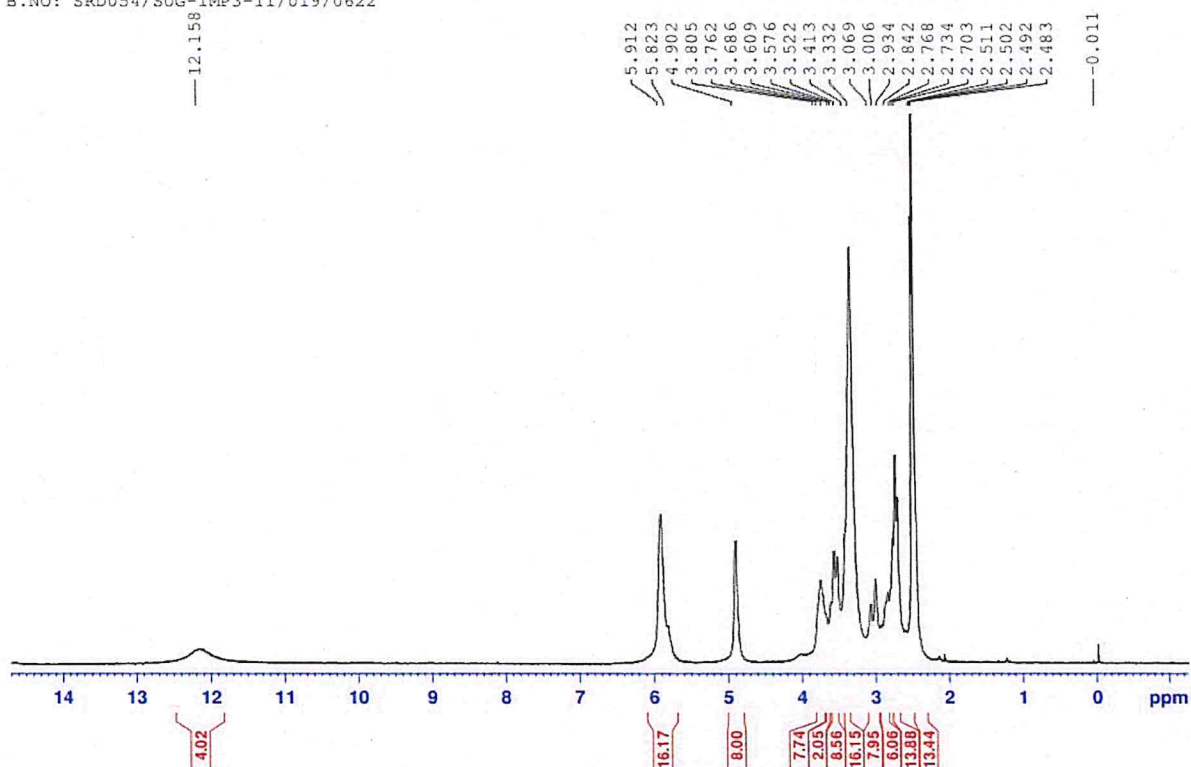
^c At 2nd position of CH₂ protons are overlapped with the NMR solvent of DMSO-d₆.

Spk
21 Jun 23

L. J. J.
21 Jun 2017

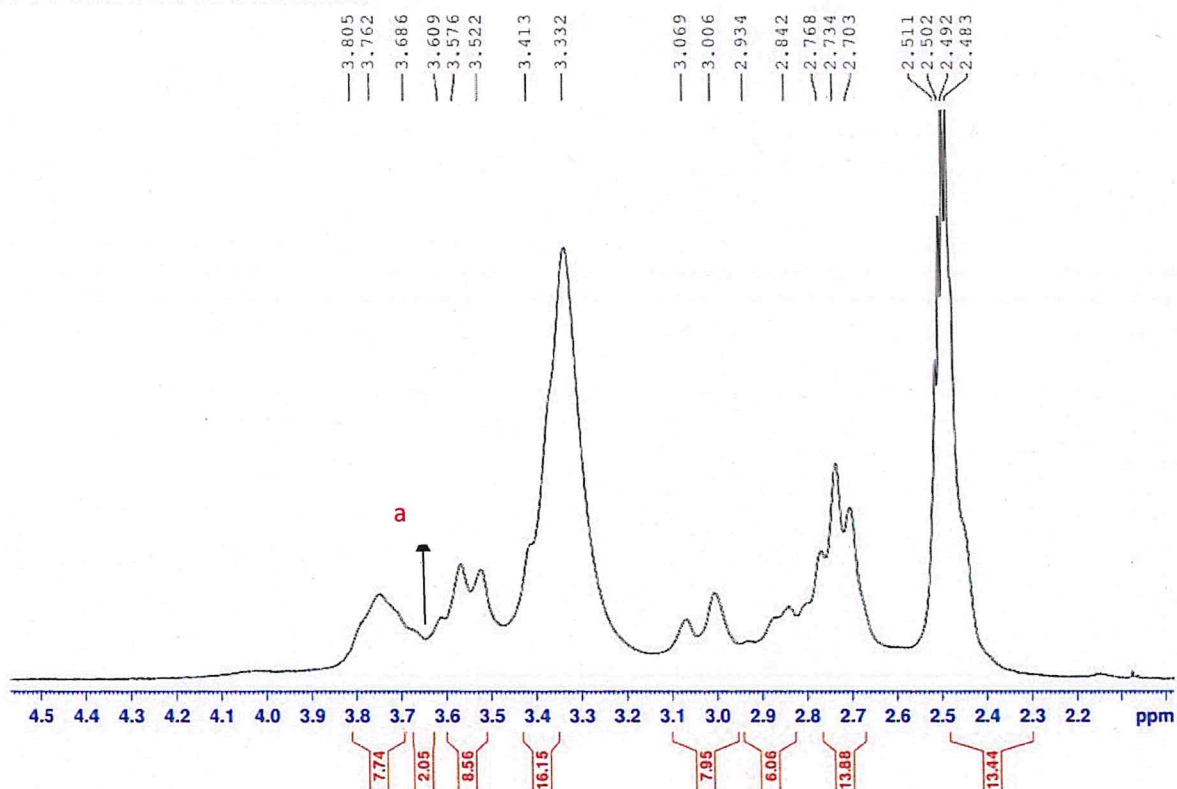
¹H NMR spectrum of Sugammadex Monohydroxy Impurity

B.NO: SRD054/SUG-IMP3-II/019/0622



¹H NMR spectrum of Sugammadex Monohydroxy Impurity (Zoomed 1)

B.NO: SRD054/SUG-IMP3-II/019/0622

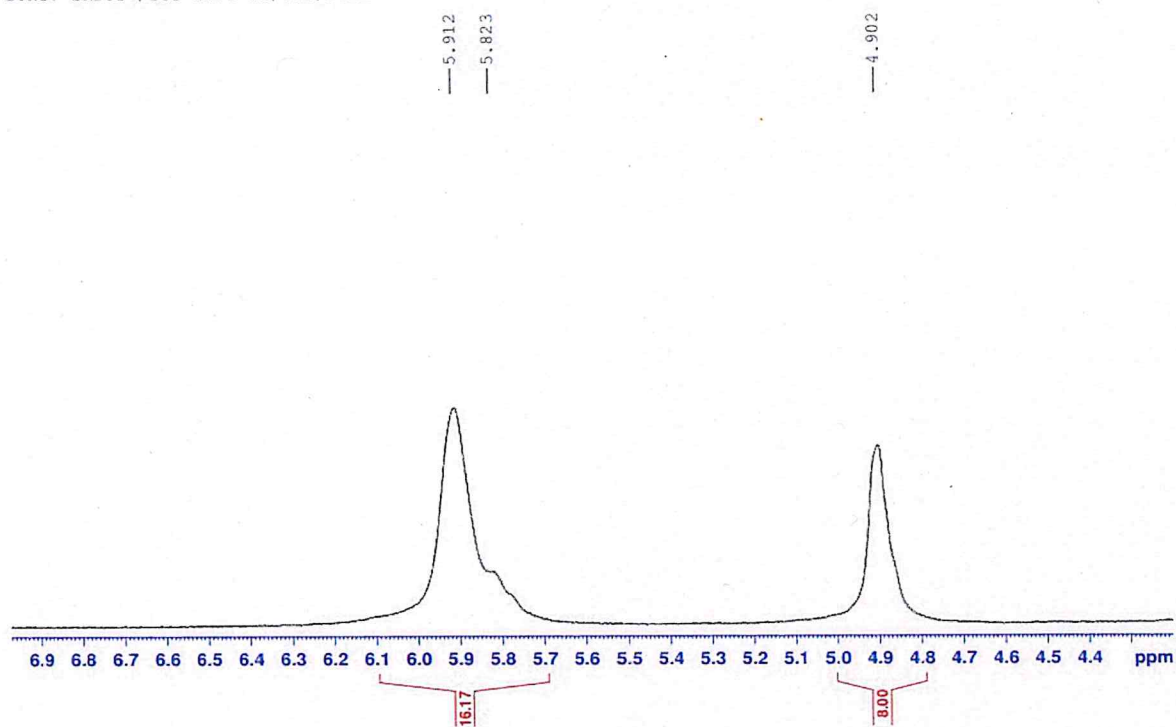


Gopikrishna
21 Jun 23

L. L. L.
21 Jun 23

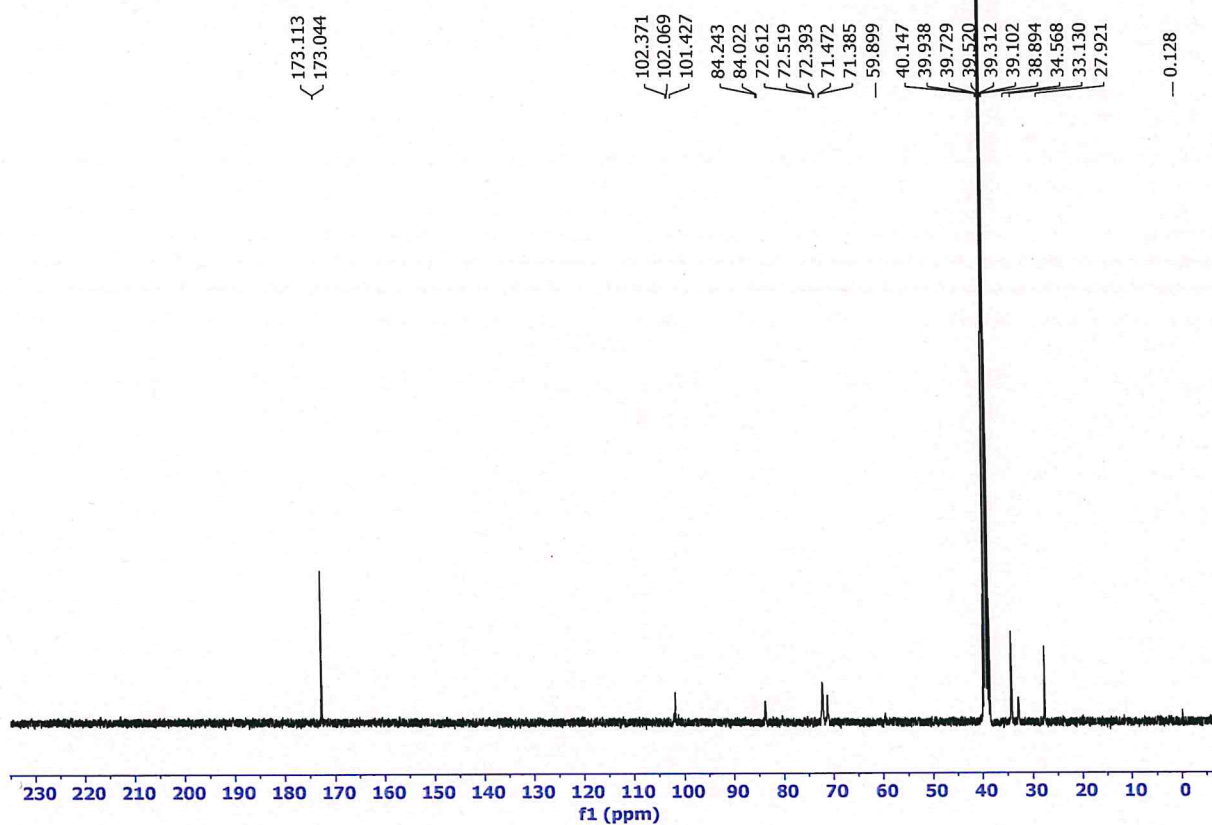
¹H NMR spectrum of Sugammadex Monohydroxy Impurity (Zoomed 2)

B.NO: SRD054/SUG-IMP3-II/019/0622



¹³C NMR spectrum of Sugammadex Monohydroxy Impurity

B.NO : SRD054/SUG-IMP3-II/019/0622

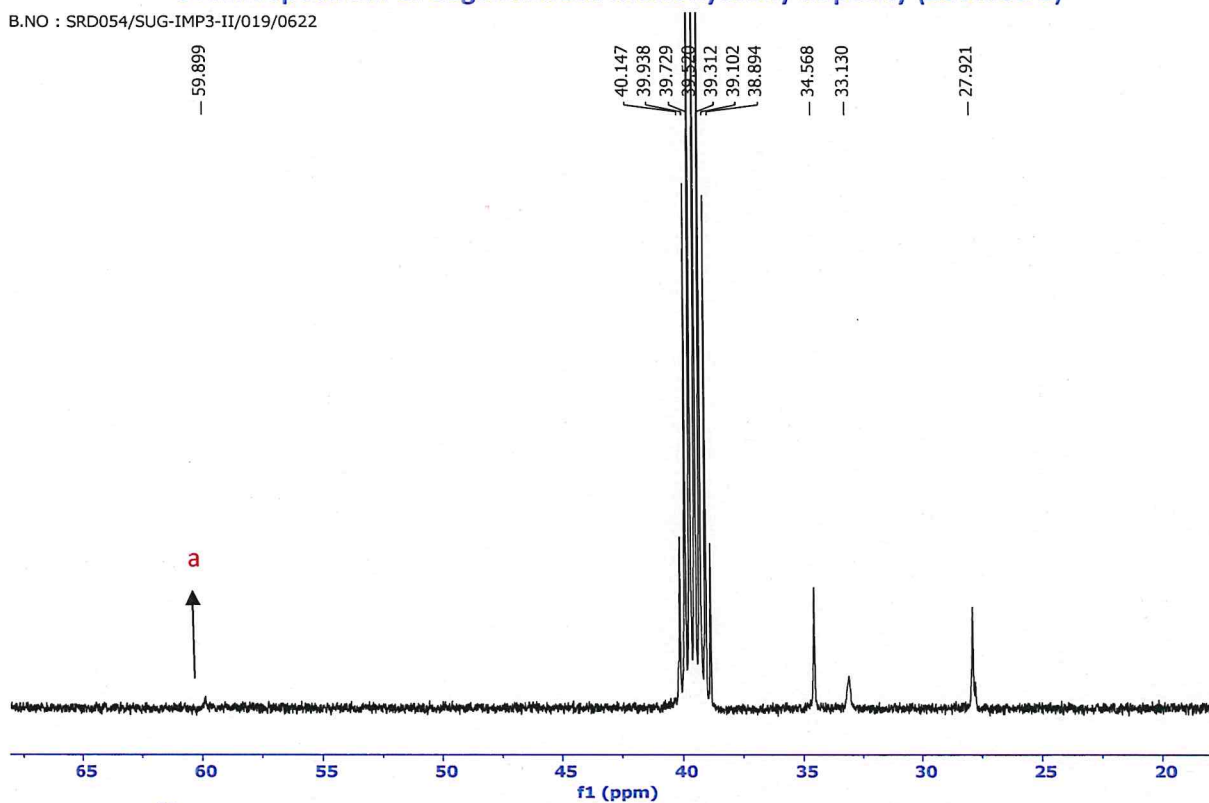


Gokul
21-08-23

L. L. L.
21-08-2023

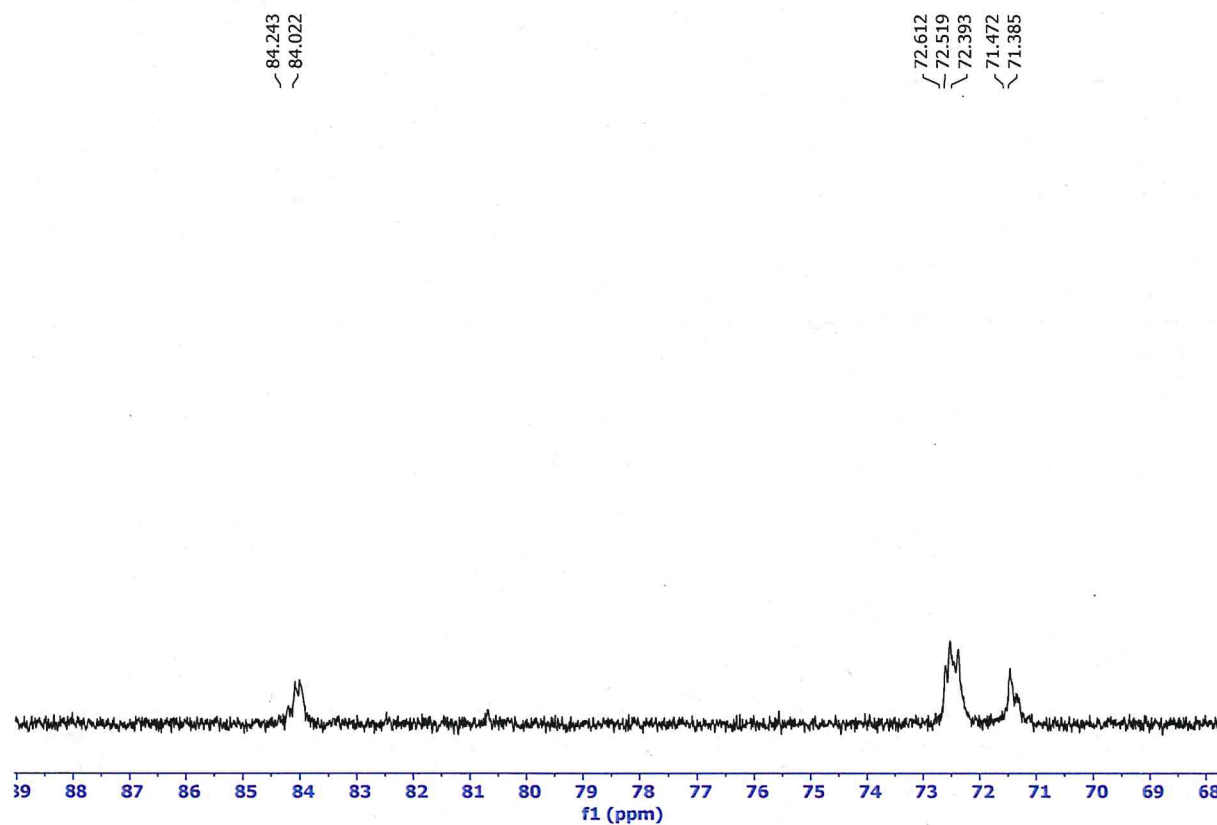
¹³C NMR spectrum of Sugammadex Monohydroxy Impurity (Zoomed 1)

B.NO : SRD054/SUG-IMP3-II/019/0622



¹³C NMR spectrum of Sugammadex Monohydroxy Impurity (Zoomed 2)

B.NO : SRD054/SUG-IMP3-II/019/0622

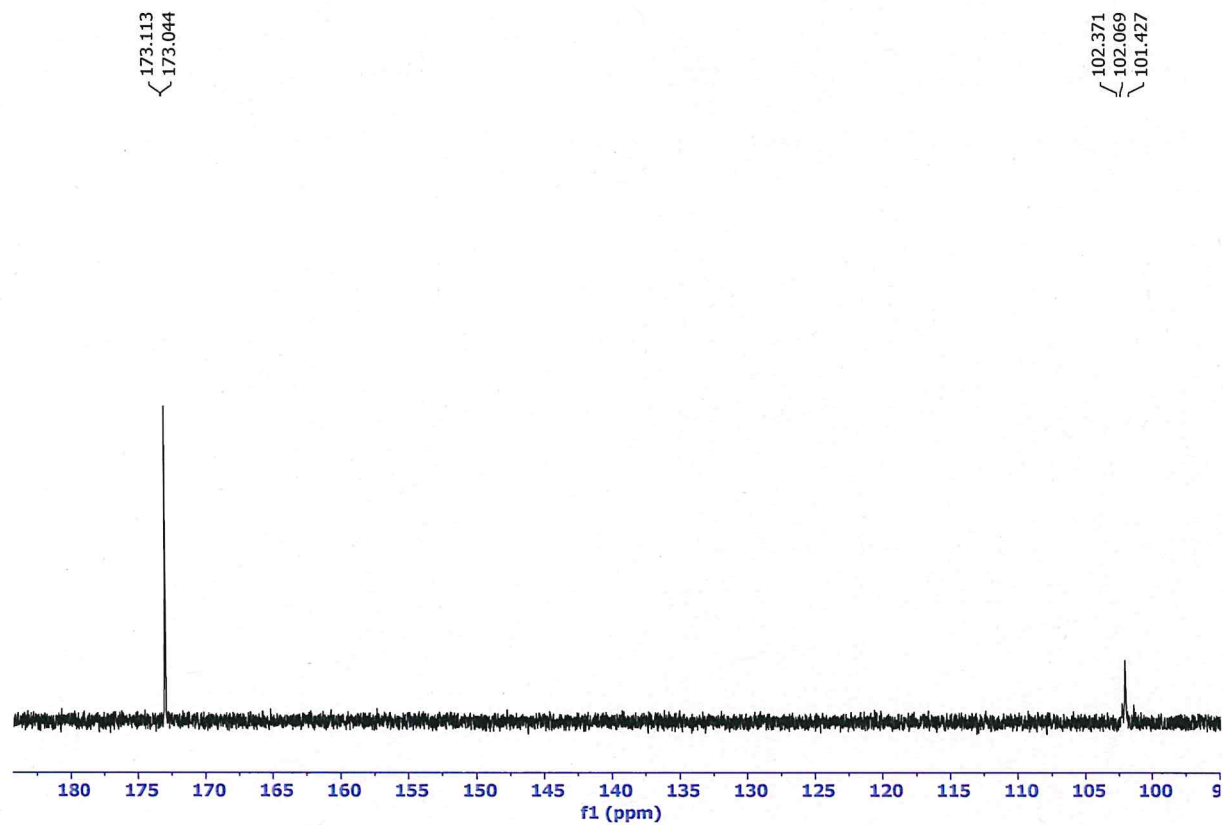


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21 Jun 2023

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21 Jun 2023

¹³C NMR spectrum of Sugammadex Monohydroxy Impurity (Zoomed 3)

B.NO : SRD054/SUG-IMP3-II/019/0622



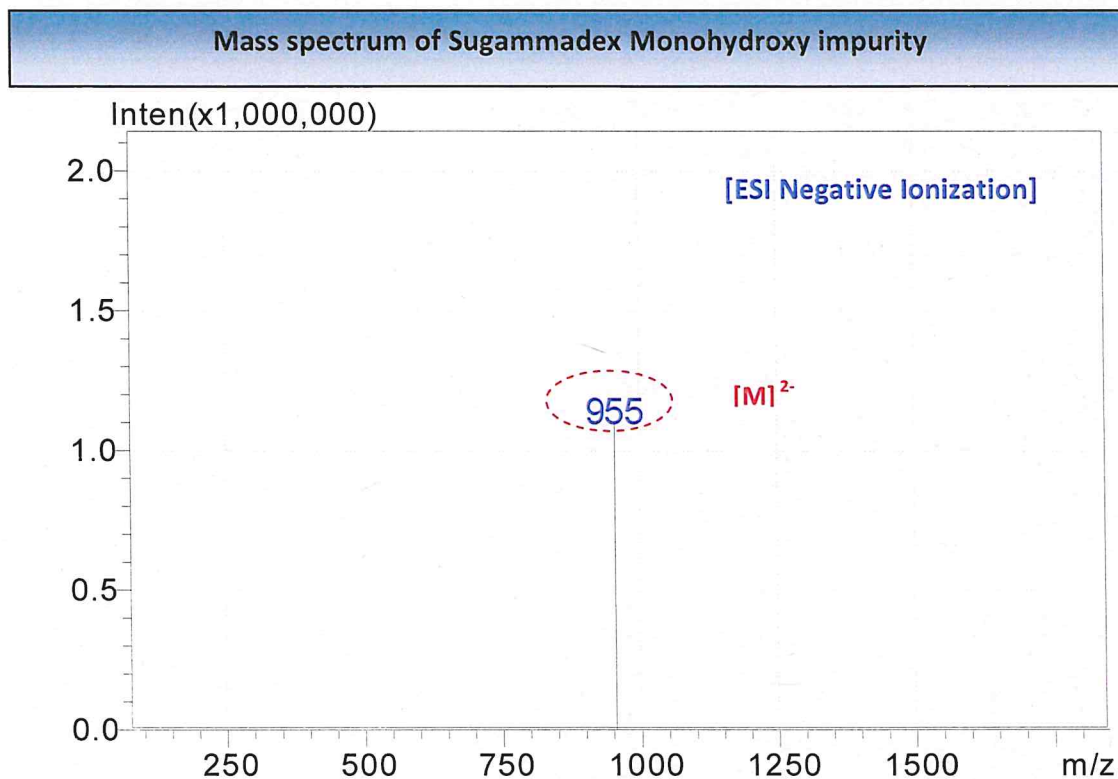
Spkars
21 Jun 23

L.H
21 Jun 2023

Mass Spectral data

Method conditions: ESI 3.5kV Negative DL temperature 230°C

The exact mass of Sugammadex Monohydroxy impurity (SRD054/SUG-IMP3-II/019/0622) is confirmed as 1912Da (Seen as m/z 955(-ve) $[M]^{2-}$ di-charged ion) by the signals of ESI mass spectral data.



Gokul
21 Jan 23

L. K.
21 Jan 2023

Purity by HPLC

The purity of Sugammadex Monohydroxy impurity is analyzed by HPLC using In-house method

Purity : 90.09% Area

Attached the HPLC chromatogram

Blank



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Sample Information

Acquired by : Stanis
Sample Name : Blank
Sample ID :
Tray# : 4
Vial# : 12
Injection Volume : 50
Data File : SPL-GIC-006_2006010.lcd
Method File : Sugammadex HPLC mthd.lcm
Batch File : Sugammadex SPL-GIC-006_200623.lcb
Date Acquired : 6/20/2023 11:40:43 PM

Mobile Phase-A : 0.05% formic acid in 1000mL of water

Mobile Phase-B : Acetonitrile:Methanol (90:10)

Column : Shim-pack GIST C18(150X4.6mm.3μ)

Flow rate :0.6ml/min

A.R.No:

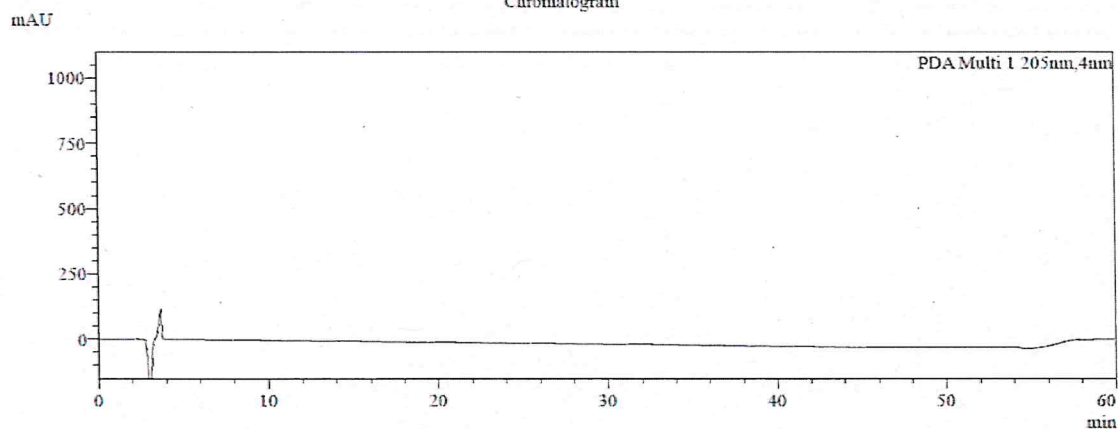
Analysed by:A.Stanislaus

<<LC Time Program>>

Time	Module	Command	Value	Comment
0.01	Pumps	B.Conc	20	
40.00	Pumps	B.Conc	25	
50.00	Pumps	B.Conc	25	
52.00	Pumps	B.Conc	20	
60.00	Controller	Stop		

Method

Chromatogram



Peak Table

Peak#	Ret. Time	Area	Area%	Name	RRT
Total					

Stanis
21 Jun 23

L. S. S.
21 Jun 2023

Sugammadex Monohydroxy impurity



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ALATHUR.

Sample Information

Acquired by : Stanis
Sample Name : Sugammadex Monohydroxy Impurity
Sample ID : SRD054/SUG-IMP3-II/019/0622
Tray# : 4
Vial# : 17
Injection Volume : 50
Data File : SPL-GIC-006_2006011.lcd
Method File : Sugammadex HPLC mthd.lcm
Batch File : Sugammadex SPL-GIC-006_200623.lcb
Date Acquired : 6/21/2023 12:41:08 AM

Mobile Phase-A : 0.05% formic acid in 1000mL of water
Mobile Phase-B : Acetonitrile:Methanol (90:10)
Column : Shim-pack GIST C18(150X4.6mm,3μ)
Flow rate : 0.6ml/min

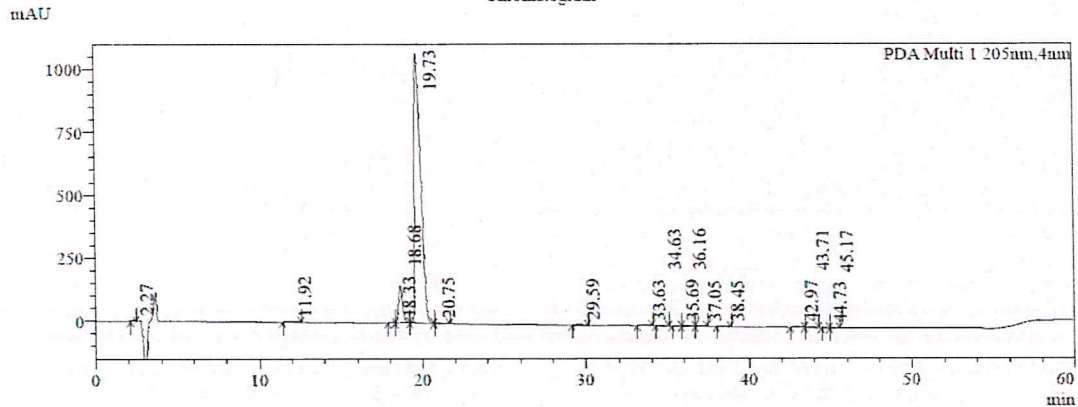
A.R.No:SRD054/SUG-IMP3-II/019/0622/AR23-3851
Analysed by:A.Stanislaus

Method

<<LC Time Program>>

Time	Module	Command	Value	Comment
0.01	Pumps	B.Conc	20	
40.00	Pumps	B.Conc	25	
50.00	Pumps	B.Conc	25	
52.00	Pumps	B.Conc	20	
60.00	Controller	Stop		

Chromatogram



Peak Table

Peak#	Ret. Time	Area	Area%	Name	RRT
1	2.27	49144	0.15		0.12
2	11.92	54091	0.17		0.60
3	18.33	28328	0.09		0.93
4	18.68	2526523	7.90		0.95
5	19.73	28798425	90.09	Sugammadex Monohydroxy Impurity	1.00
6	20.75	63036	0.20		1.05
7	29.59	68276	0.21		1.50
8	33.63	49470	0.15		1.70
9	34.63	23540	0.07		1.76
10	35.69	22903	0.07		1.81
11	36.16	51646	0.16		1.83
12	37.05	22586	0.07		1.88
13	38.45	56904	0.18		1.95
14	42.97	75357	0.24		2.18
15	43.71	39929	0.12		2.22
16	44.73	19606	0.06		2.27
17	45.17	16325	0.05		2.29
Total		31966089	100.00		

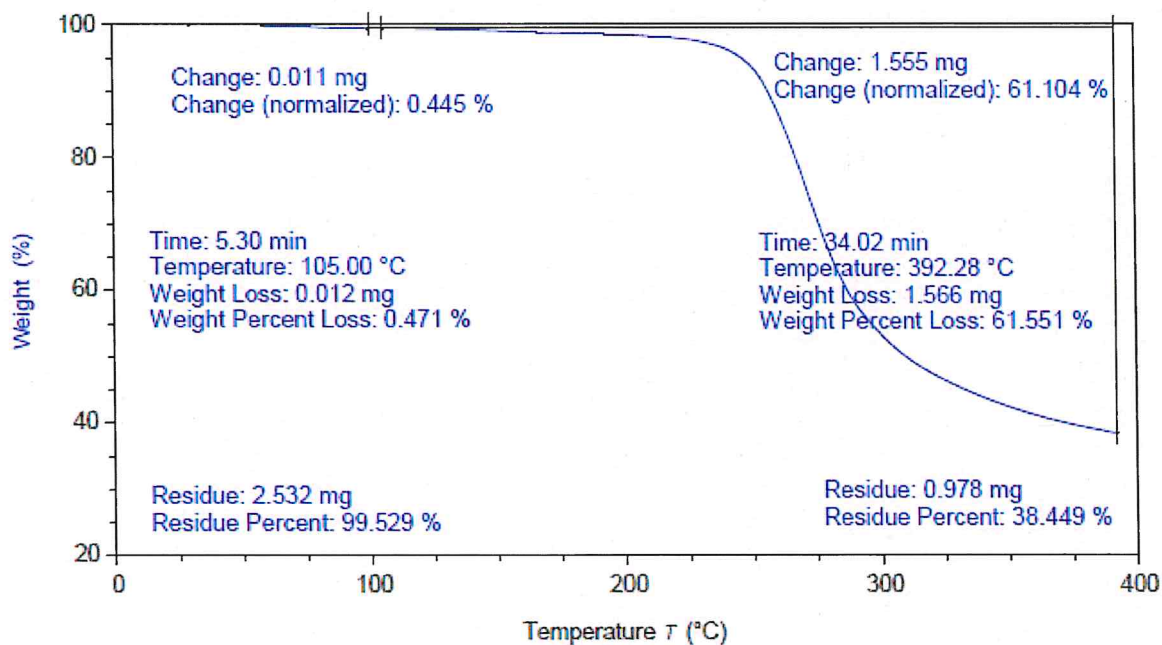
Qo/ky2-6
21-Jun-23

Lik
21/6/2023

Volatile Loss Analysis by Thermogravimetry (TGA)

Sample Name: Sugammadex Monohydroxy Impurity

Batch No. : SRD054/SUG-IMP3-II/019/0622



Volatile loss Calculation:

Volatile loss: Initial percentage – percentage after volatile loss

$$= 100 - 99.529\%$$

$$= 0.47\% \text{ w/w}$$

Percentage of Volatile loss = 0.47 % w/w

Gokul
21 Jun 23

L. L. L.
21 Jun 2023

Impurity Potency Calculation

Sample Name: Sugammadex Monohydroxy Impurity

Batch No. : SRD054/SUG-IMP3-II/019/0622

Potency Calculation:

Potency : Purity – Percentage of volatile loss

$$= 90.09\% - 0.47\% \text{ w/w}$$

$$= 89.62\% \text{ w/w}$$

Potency = 89.62% w/w

Getupis
21/5/2017

Lily
21/5/2017