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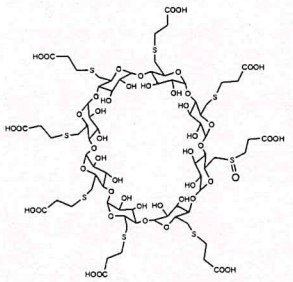
CERTIFICATE OF ANALYSIS

NAME OF THE PRODUCT/ IMPURITY (IUPAC Name)		**SUGAMMADEX SULFOXIDE DIASTEREOMER-1 IMPURITY** IUPAC NAME: 6-Deoxy-6-((2-carboxyethyl)sulfinyl)-heptakis(6-deoxy-6-(2-carboxyethyl)thio)-gamma-cyclodextrin Epimer A
CAS. No.	: 2412953-11-6	**STRUCTURE**
BATCH No.	: SRD054/SUG-IMP4-II/025/0622	
MFG. DATE	: JUN-2022	
AR No.	: SRD054/SUG-IMP4-II/025/0622/AR23-3849	
DATE OF ANALYSIS	: 20 JUN 2023	
RETEST DATE	: 19 JUN 2024	
GRADE	: IN-HOUSE	
BATCH QUANTITY	: 10 mg	

S. No.	TESTS	SPECIFICATIONS	RESULTS
1	Description	Report the result	White solid
2	Identification by MS	The exact mass of Sugammadex sulfoxide diastereomer-1 should be confirmed as 2016Da	Mass spectral data confirms the exact mass of Sugammadex sulfoxide diastereomer-1 as 2016Da
3	Identification by ^{1}H & ^{13}C NMR	The molecular structure of Sugammadex sulfoxide diastereomer-1 should be confirmed by ^{1}H & ^{13}C NMR spectra	^{1}H & ^{13}C NMR spectra confirms the molecular structure of Sugammadex sulfoxide diastereomer-1
4	Purity by HPLC	For Informative	96.32% Area
5	Volatile loss by TGA	For Informative	0.15% w/w
6	Potency	For Informative	96.17% 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)		Reviewed By (Sign& Date)		Approved By (Sign& Date)	
	21 Jun 23		21/6/2023		21 Jun 23

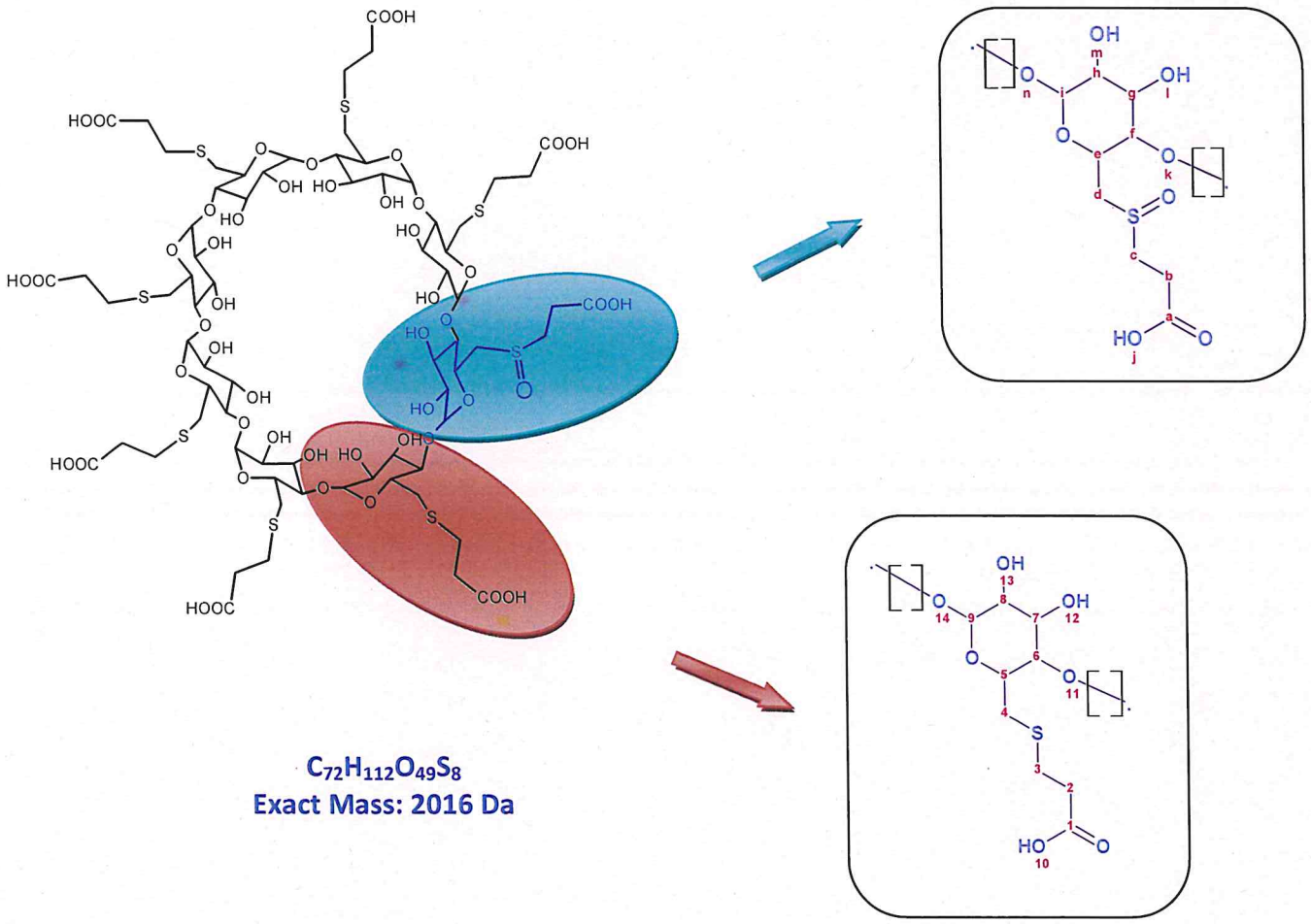
^{1}H & ^{13}C NMR Spectral data

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

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

The structure of Sugammadex sulfoxide diastereomer-1 impurity (SRD054/SUG-IMP4-II/025/0622) is confirmed with the signals of the ^{1}H & ^{13}C NMR spectra and their respective interpretation

Sugammadex sulfoxide diastereomer -1 impurity



$C_{72}H_{112}O_{49}S_8$
Exact Mass: 2016 Da

The assignment of ^{1}H and ^{13}C NMR signals of Sugammadex sulfoxide diastereomer-1 impurity

Position [a]	^{1}H	^{1}H δ(ppm)	Multiplicity [b]	^{13}C δ(ppm)	Position [a]	^{1}H	^{1}H δ(ppm)	Multiplicity [b]	^{13}C δ(ppm)	^{13}C
1	--	--	--	173.286	a	--	--	--	172.918	COOH
2[c]	14H	2.462-2.545	m	33.222	b	2H	2.606	m	26.968	CH_2
3	14H	2.648-2.745	t	28.019	c	2H	2.818	m	46.942	CH_2
4	14H	2.845-2.934 3.023-3.085	m	34.701	d	2H	3.140	m	51.981	CH_2
5	7H	3.340-3.380	m	72.589	e	1H	3.484	m	72.589	CH
6	7H	3.762	m	71.582	f	1H	4.060-4.122	t	71.582	CH
7	7H	3.537-3.626	t	72.457	g	1H	3.537-3.626	t	72.457	CH
8	7H	3.429	m	84.251	h	1H	3.484	m	84.024	CH
9	7H	4.917 4.982-5.002	d	102.234	i	1H	5.076	d	101.738	CH
10[d]	7H	--	--	--	J	1H	--	--	--	CO-OH
11	--	--	--	--	k	--	--	--	--	--
12	7H	5.926	sb	--	l	1H	5.926	sb	--	OH
13	7H	5.926	sb	--	m	1H	5.926	sb	--	OH
14	--	--	--	--	n	--	--	--	--	--

[a] **Carbon** and Hydrogen numbering - only for NMR spectral data assignments,

[b] **Resonance multiplicities, coupling constants, and coupled partners in parentheses:** *(s)* singlet, *(t)* triplet, *(m)* multiplet, *(sb)* singlet broad, *(d)* Doublet.

[c] At 2nd position of CH_2 protons overlapped with NMR solvent DMSO-d_6,

[d] carboxylic acid proton got exchanged DMSO-d6 water in the proton NMR spectrum.

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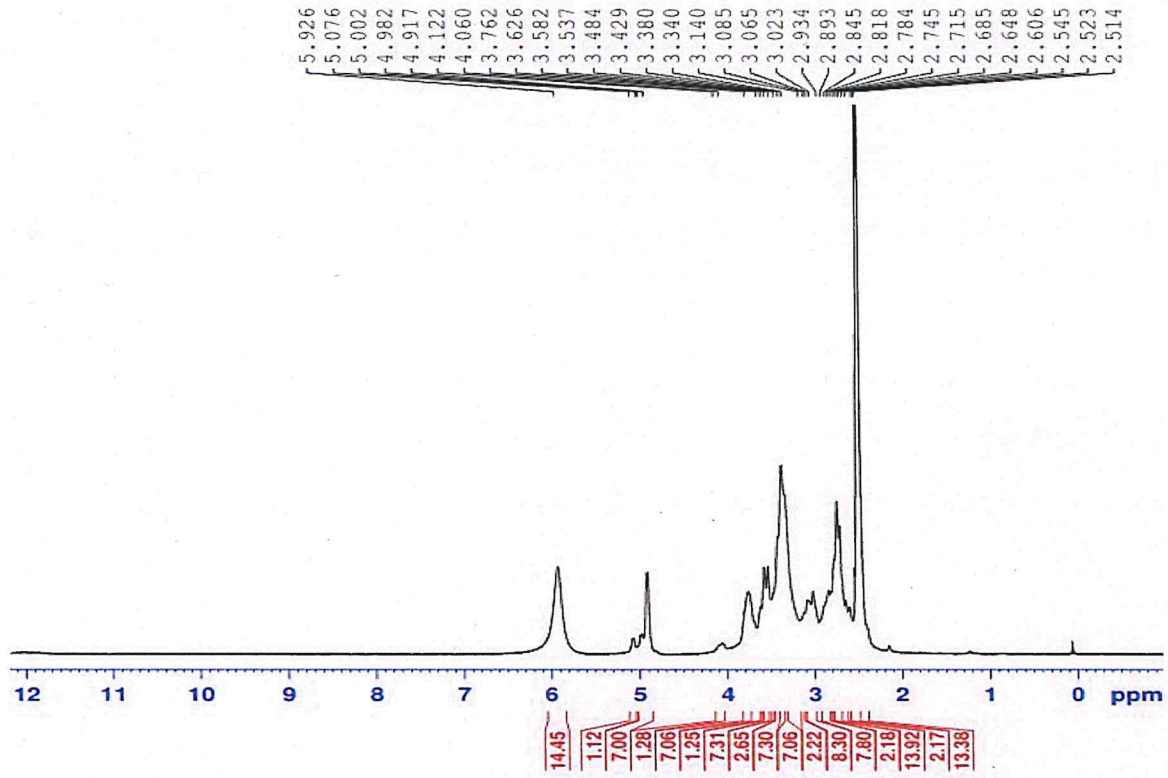
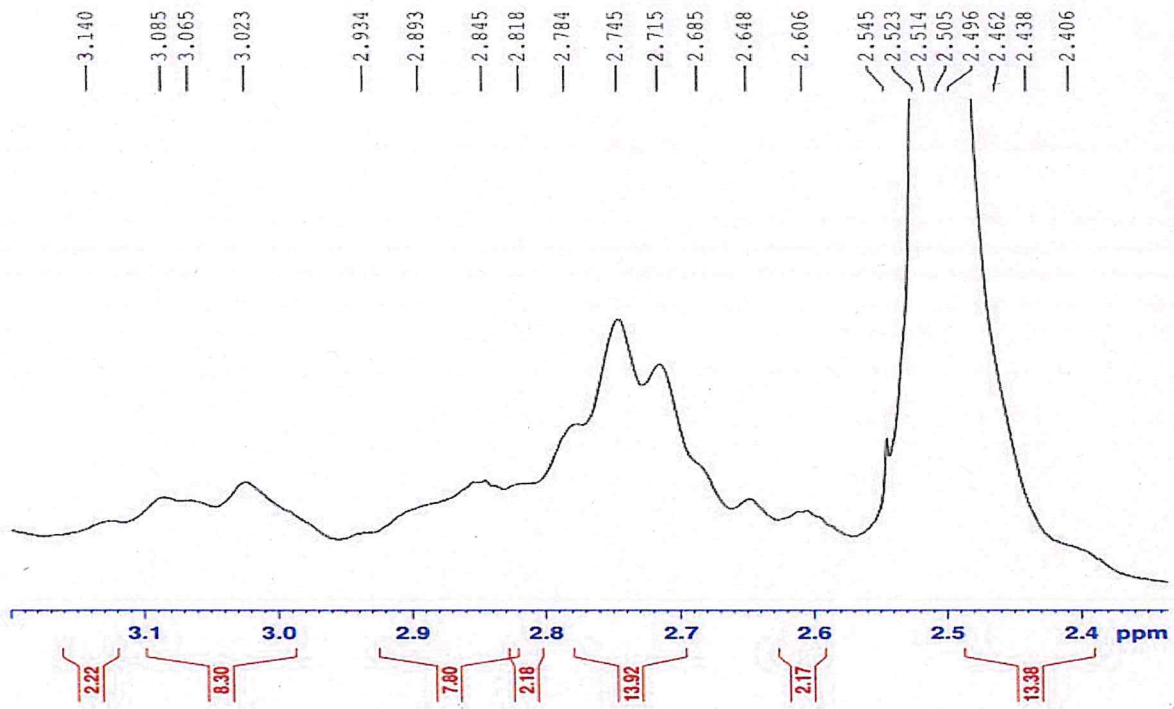
^{1}H NMR spectrum of Sugammadex sulfoxide diastereomer -1 impurity

B.No : SRD054/SUG-IMP4-II/025/0622



^{1}H NMR spectrum of Sugammadex sulfoxide diastereomer -1 impurity (Zoomed 1)

B.No : SRD054/SUG-IMP4-II/025/0622



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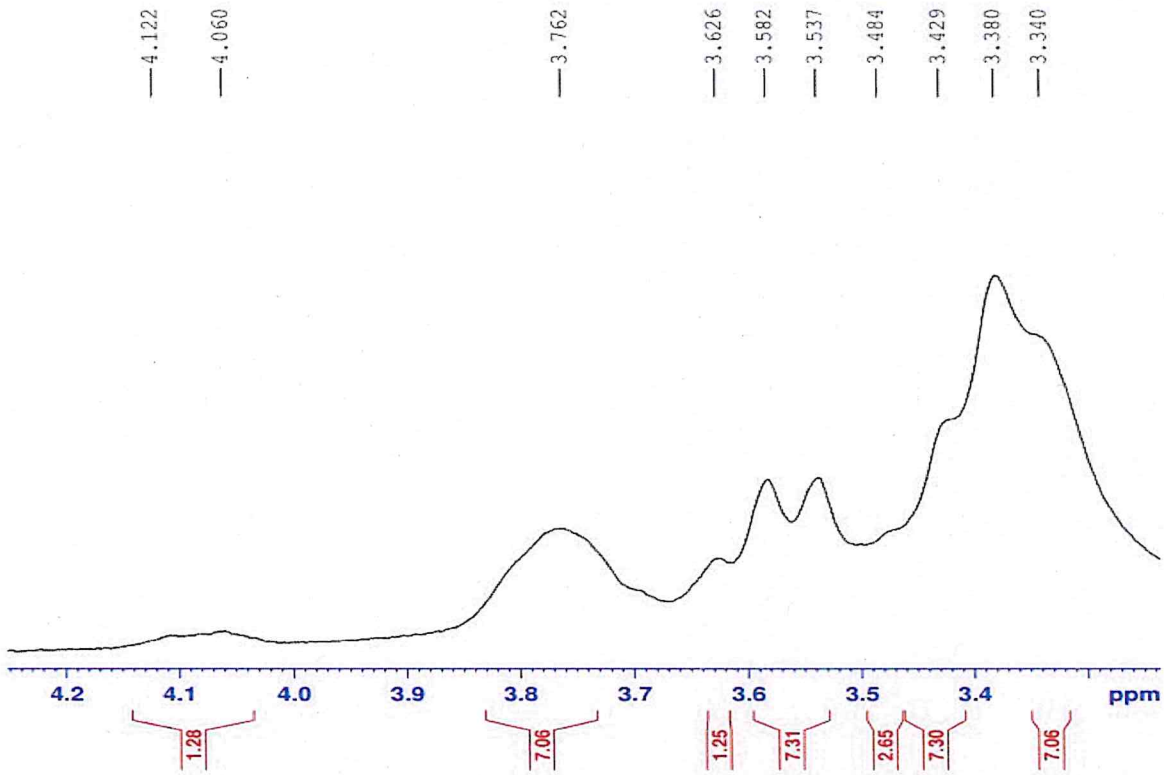
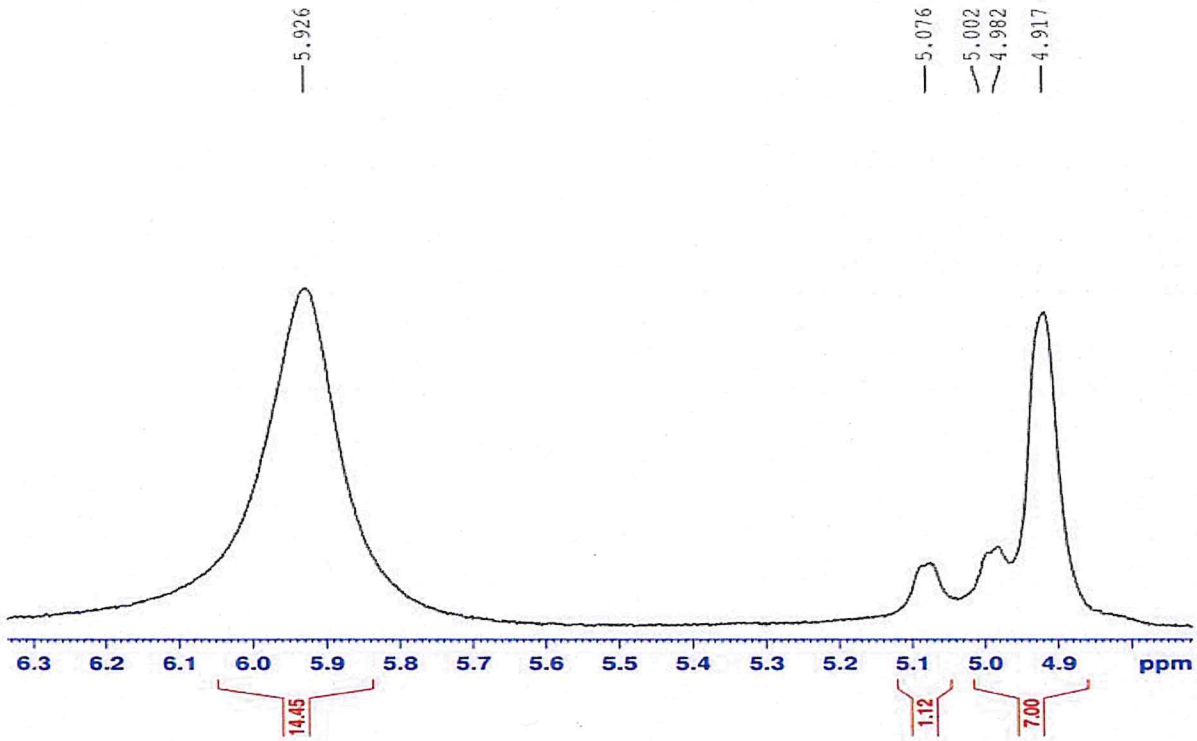
^{1}H NMR spectrum of Sugammadex sulfoxide diastereomer-1 impurity (Zoomed 2)

B.No : SRD054/SUG-IMP4-II/025/0622



^{1}H NMR spectrum of Sugammadex sulfoxide diastereomer-1 impurity (Zoomed 3)

B.No : SRD054/SUG-IMP4-II/025/0622



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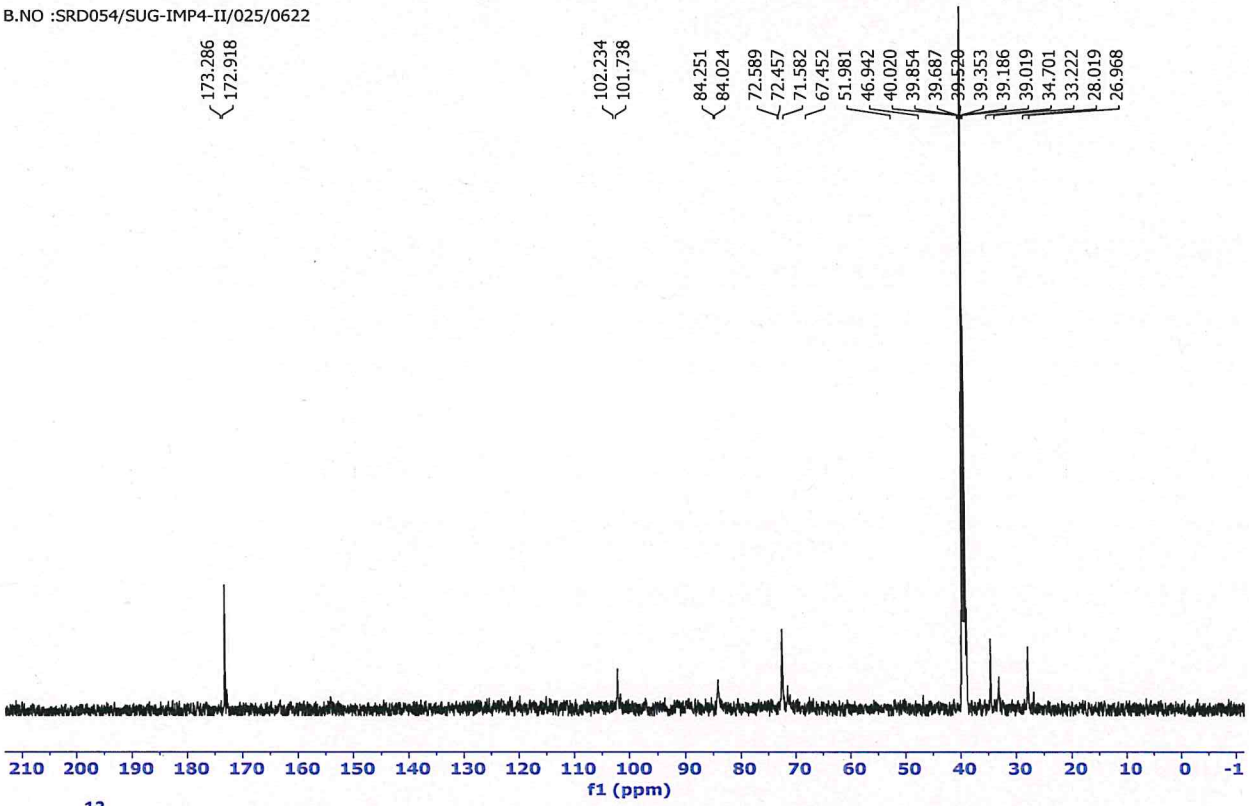
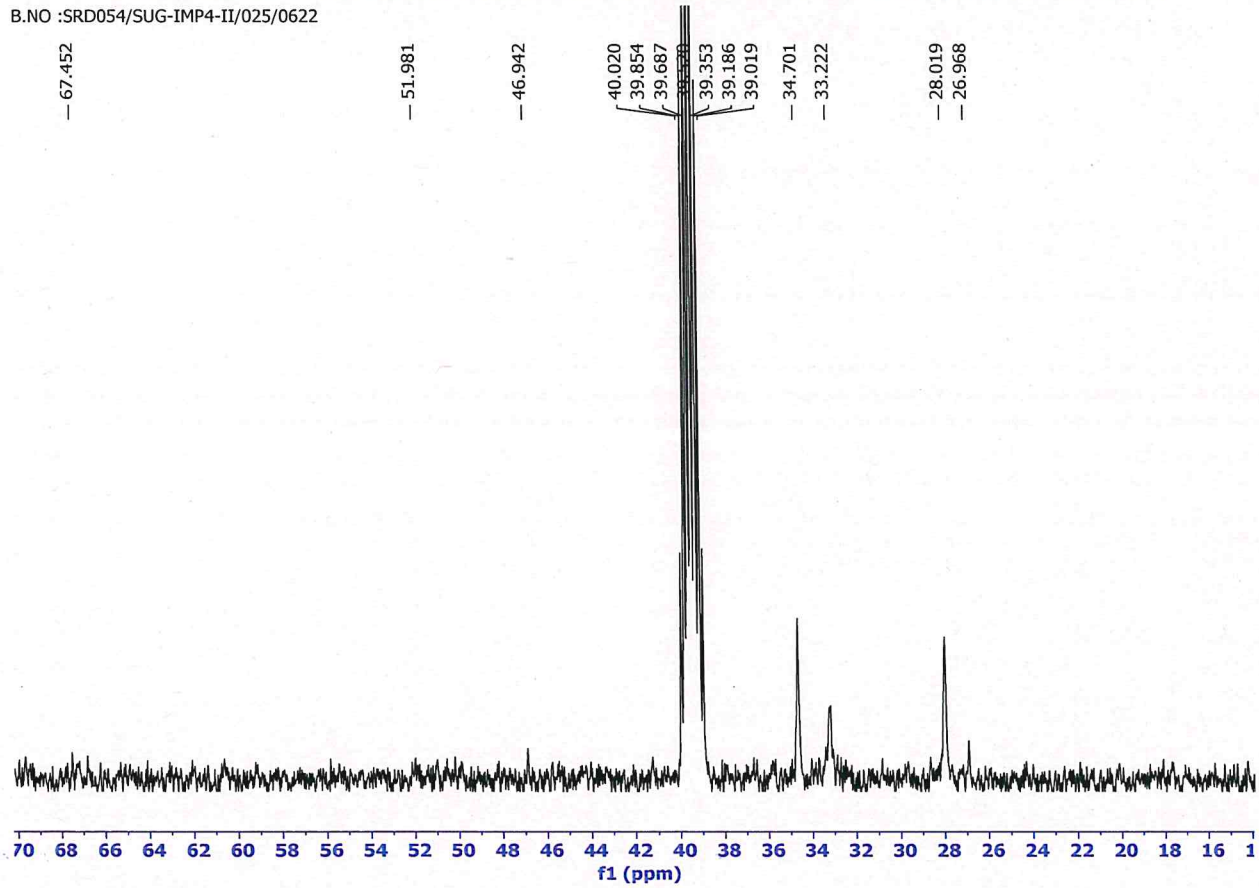
^{13}C NMR spectrum of Sugammadex sulfoxide diastereomer-1 impurity

B.NO :SRD054/SUG-IMP4-II/025/0622



^{13}C NMR spectrum of Sugammadex sulfoxide diastereomer-1 impurity (Zoomed 1)

B.NO :SRD054/SUG-IMP4-II/025/0622



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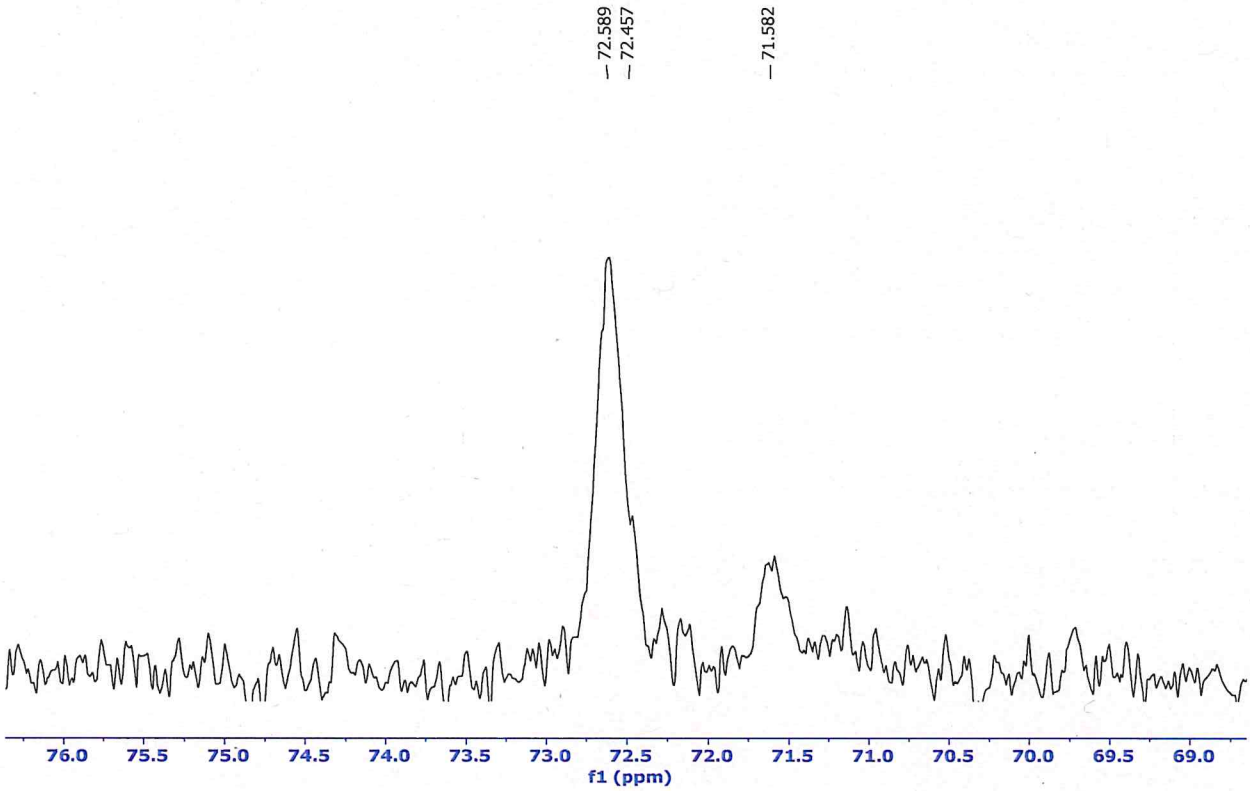
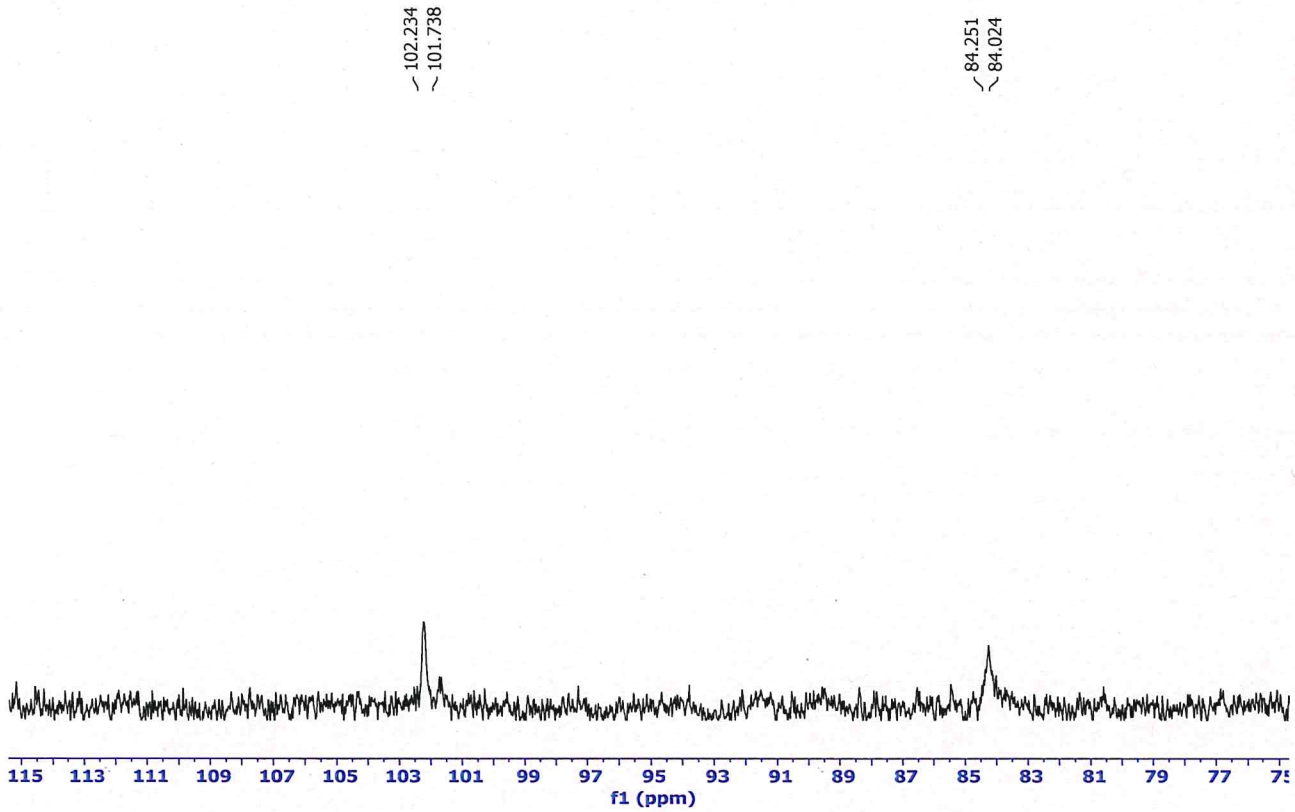
^{13}C NMR spectrum of Sugammadex sulfoxide diastereomer-1 impurity (Zoomed 2)

B.NO :SRD054/SUG-IMP4-II/025/0622



^{13}C NMR spectrum of Sugammadex sulfoxide Diastereomer -1 impurity (Zoomed 3)

B.NO :SRD054/SUG-IMP4-II/025/0622



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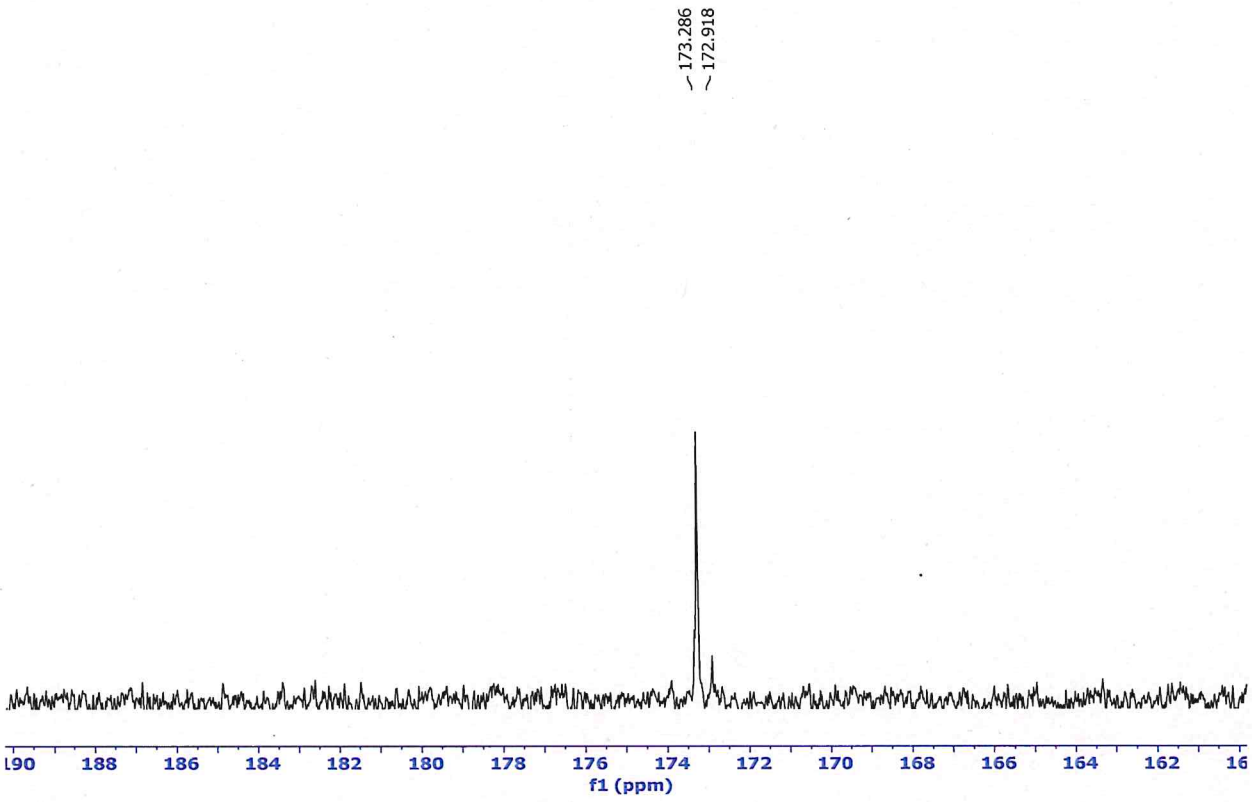
^{13}C NMR spectrum of Sugammadex sulfoxide Diastereomer -1 impurity (Zoomed 4)

B.NO :SRD054/SUG-IMP4-II/025/0622



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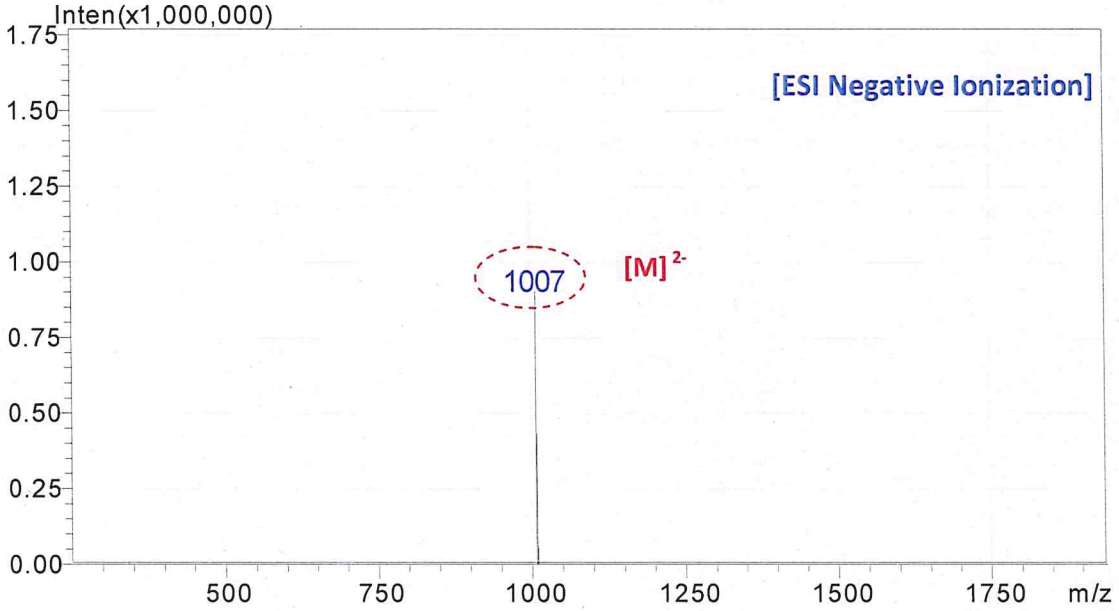
Mass Spectral data

Method conditions: ESI 3.5kV Negative DL temperature 250°C

The exact mass of Sugammadex sulfoxide diastereomer-1 impurity (SRD054/SUG-IMP4-II/025/0622) is confirmed as 2016Da (Seen as m/z 1007 (-ve) as di-charge by the signals of ESI mass spectral data.

Mass spectrum of Sugammadex sulfoxide diastereomer-1 impurity



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Purity by HPLC

The purity of Sugammadex sulfoxide diastereomer-1 impurity is analyzed by HPLC using

In-house method

Purity: 96.32% 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_2006005.lcd
Method File	: Sugammadex HPLC mthd..lcm
Batch File	: Sugammadex_SPL-GIC-006_200623.lcb
Date Acquired	: 6/20/2023 6:38:39 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

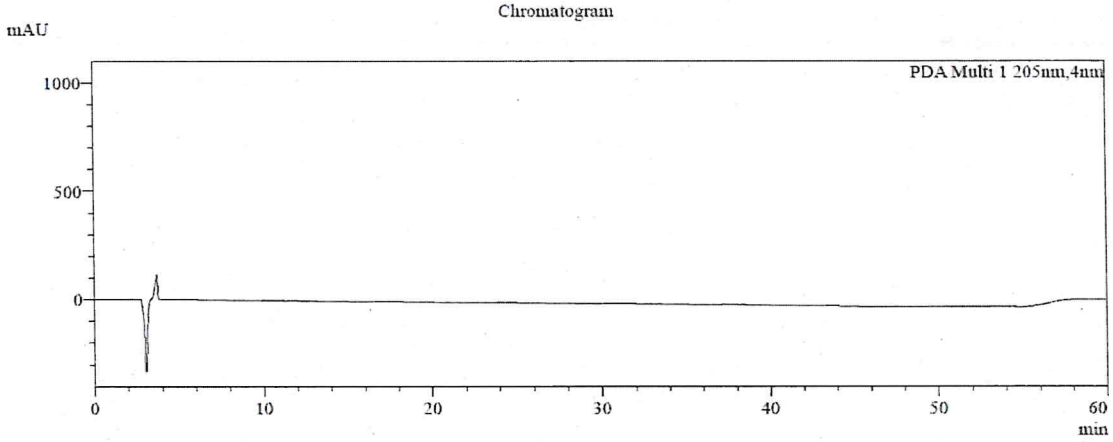
A.R.No:
Analysed by:A.Stanislus

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

PDA Ch1 205nm

Peak#	Ret. Time	Area	Area%	Name	RRT
Total					

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Sample Information

Acquired by	: Stanis
Sample Name	: Sugammadex sulfoxide Diastereomer-1 Impurity
Sample ID	: SRD054/SUG-IMP4-II/025/0622
Tray#	: 4
Vial#	: 13
Injection Volume	: 50
Data File	: SPL-GIC-006_2006006.lcd
Method File	: Sugammadex HPLC mthd..lcm
Batch File	: Sugammadex_SPL-GIC-006_200623.lcb
Date Acquired	: 6/20/2023 7:39:04 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

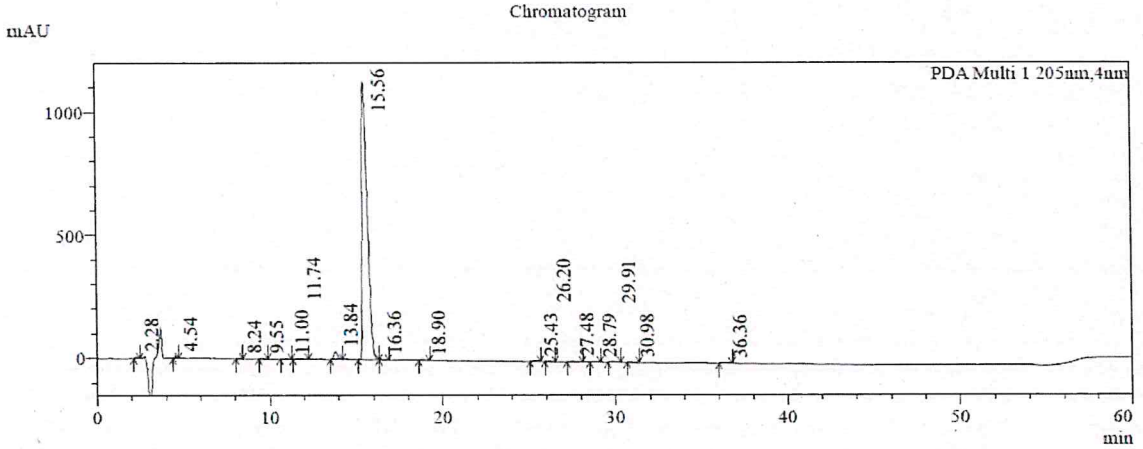
A.R.No:SRD054/SUG-IMP4-II/025/0622/AR23-3849
Analysed by:A.Stanislus

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

PDA Ch1 205nm

Peak#	Ret. Time	Area	Area%	Name	RRT
1	2.28	61806	0.24		0.15
2	4.54	63630	0.25		0.29
3	8.24	12898	0.05		0.53
4	9.55	22618	0.09		0.61
5	11.00	24273	0.10		0.71
6	11.74	25195	0.10		0.75
7	13.84	452640	1.78		0.89
8	15.56	24520320	96.32	Sugammadex Sulfoxide Diastereomer-1 Impurity	1.00
9	16.36	34939	0.14		1.05
10	18.90	58549	0.23		1.21
11	25.43	20182	0.08		1.63
12	26.20	16859	0.07		1.68
13	27.48	35972	0.14		1.77
14	28.79	17421	0.07		1.85
15	29.91	37001	0.15		1.92
16	30.98	19390	0.08		1.99
17	36.36	33181	0.13		2.34
Total		25456875	100.00		

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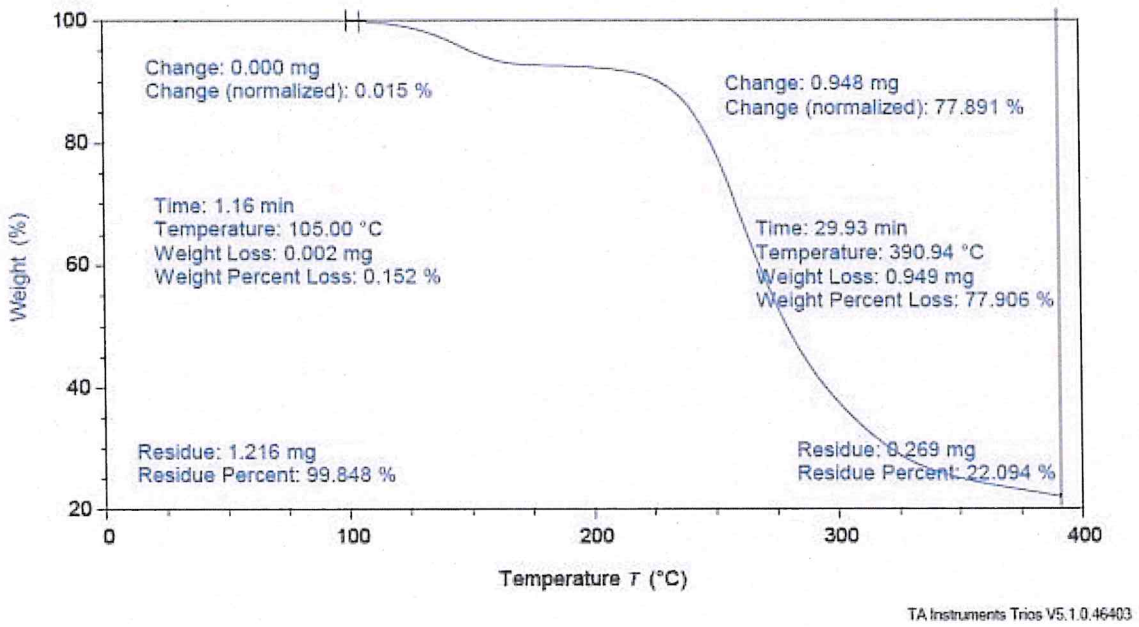
Volatile Loss Analysis by Thermogravimetry (TGA)

Sample Name: Sugammadex sulfoxide diastereomer-1 impurity

Batch No. : SRD054/SUG-IMP4-II/025/0622



Volatile loss Calculation:

Volatile loss: Initial percentage – percentage after volatile loss

= 100-99.848%

= 0.15% w/w

Percentage of Volatile loss = 0.15% w/w

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Impurity Potency Calculation

Sample Name: Sugammadex sulfoxide diastereomer-1 impurity
Batch No. : SRD054/SUG-IMP4-II/025/0622

Potency Calculation:

Potency : Purity – Percentage of volatile loss

= 96.32% - 0.15% w/w

= 96.17% w/w

Potency **= 96.17% w/w**

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