

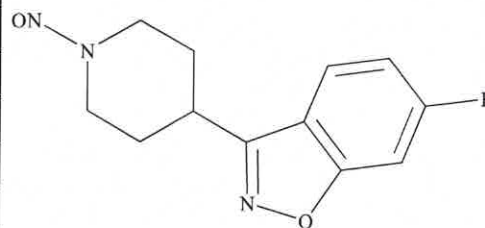
# Certificate of Analysis

Certificate No.: 20230315013

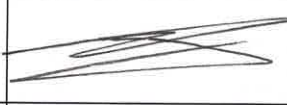

Date: March 15, 2023

Retest date: March 13, 2026

<b>Compound Name:</b> <i>N</i> -Nitroso Risperidone EP Impurity M	
<b>Synonyms:</b>	6-fluoro-3-(1-nitrosopiperidin-4-yl)benzo[d]isoxazole
<b>TLC Catalogue Number:</b>	R-0648
<b>CAS Number:</b>	2416230-38-9
<b>Molecular Weight:</b>	249.25
<b>Molecular Formula:</b>	C <sub>12</sub> H <sub>12</sub> FN <sub>3</sub> O <sub>2</sub>
<b>Source:</b>	TLC Pharmaceutical Standards
<b>Source Lot No.:</b>	5456-095A3
<b>Storage Conditions:</b>	Store at 2-8 °C
<b>Solubility:</b>	Methanol, DMSO



Test Description	Specifications	Results
<b>Visual Description</b>	Pale yellow solid	<b>Conforms</b>
<b>Identification</b>		
MS	Conforms to structure	<b>Conforms</b>
<sup>1</sup> H NMR	Conforms to structure	<b>Conforms</b>
<b>Purity (HPLC)</b>	Not less than 95.0%	<b>99.7%</b>
<b>Impurity (HPLC)</b>	RT 5.05, 0.29%	
<b>Water Content (KF)</b>	N/A	<b>0.0%</b>
<b>Residual Solvents (NMR)</b>	0.1% methanol	
<b>Assay (%)</b>	Not less than 90.0%	<b>99.6%</b>
<b>Recommendation:</b>	<b>Release.</b> The compound is an <i>N</i> -Nitroso product. Avoid using metal spatula and applying pressure. Wear proper personal protective equipment when handling.	

Name	Department	Signature	Date
Reviewed and approved by:	Quality Control		03/17/2023
Approved by:	Quality Assurance		03/17/2023

**Attachments:** HPLC, MS and NMR spectra.

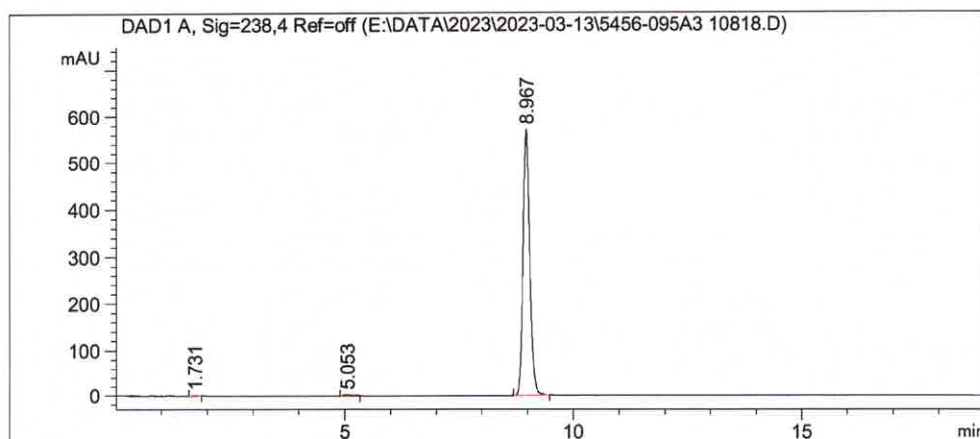
Sample Name: 5456-095A3

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Acq. Operator   : Xiaocui Zhu
Sample Operator : Xiaocui Zhu
Acq. Instrument : LC-1260_3                      Location : Vial 16
Injection Date  : 3/13/2023 2:09:28 PM          Inj Volume : 1.000 µl

Acq. Method     : C:\CHEM32\1\METHODS\2.M
Last changed    : 3/13/2023 2:05:05 PM by Xiaocui Zhu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\1.M
Last changed    : 3/13/2023 2:40:05 PM by Xiaocui Zhu
                  (modified after loading)
Sample Info     : Poroshell HPH-C18 (4.6*100mm,2.7µm); F=1.0mL/min; T=30 degree;
                  CH3OH/10mmol/L NH4HCO3(adjusted to pH 9.0 with NH3·H2O)=42/58(0-6min),42/58
                  -65/35(6-18min),65/35(after 18min)
                  ~1.6mg in 0.5mL CH3OH
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                        Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

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Signal 1: DAD1 A, Sig=238,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.731	BBA	0.0899	1.16118	1.60522e-1	0.0199
2	5.053	BBA	0.1380	16.79736	1.77403	0.2886
3	8.967	BBA	0.1554	5802.72461	569.22906	99.6915

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Totals :                      5820.68315  571.16362
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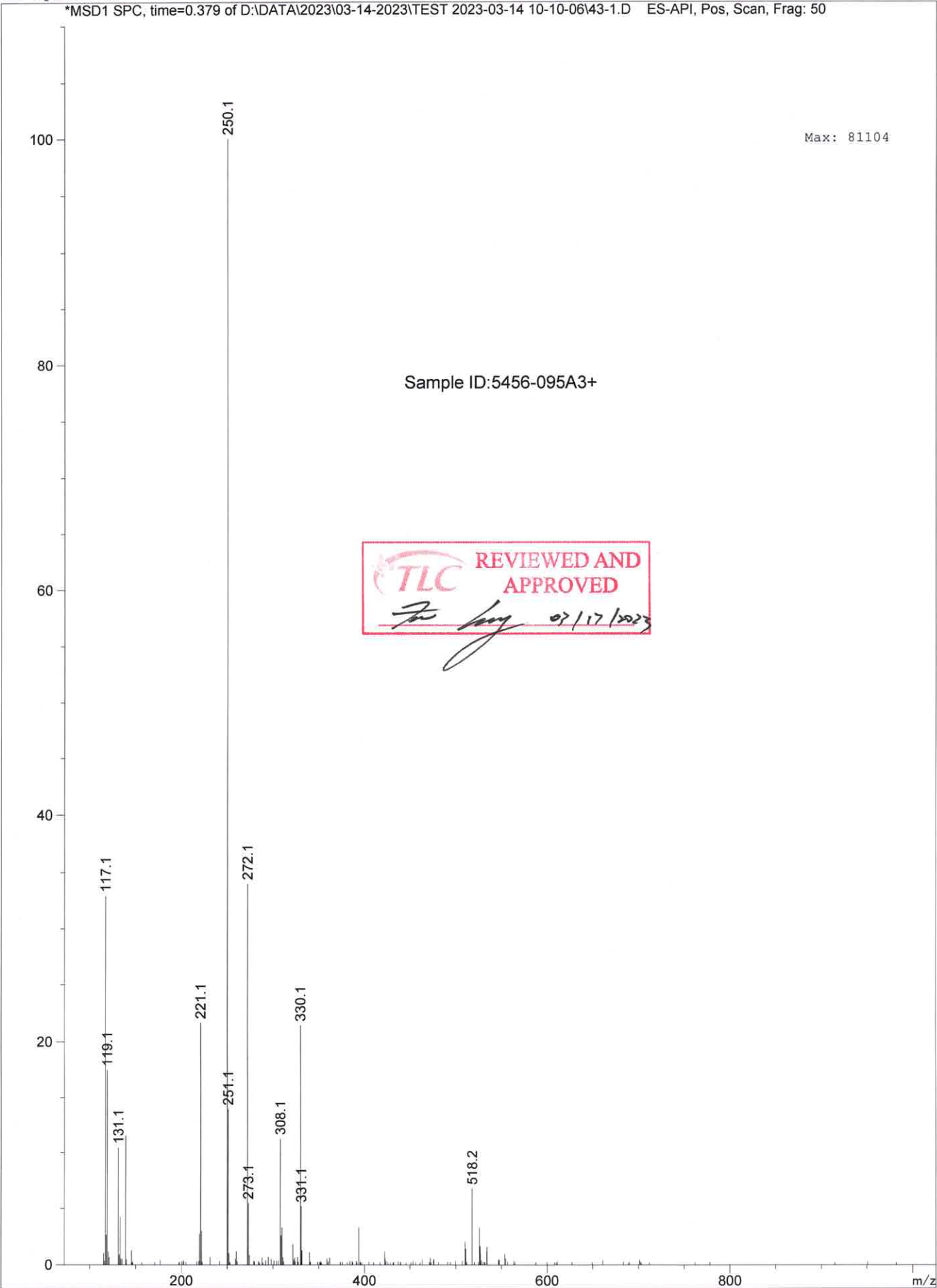
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*** End of Report ***
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MS Spectrum

\*MSD1 SPC, time=0.379 of D:\DATA\2023\03-14-2023\TEST 2023-03-14 10-10-06\43-1.D ES-API, Pos, Scan, Frag: 50



5456-095A3 1H NMR in DMSO-d6

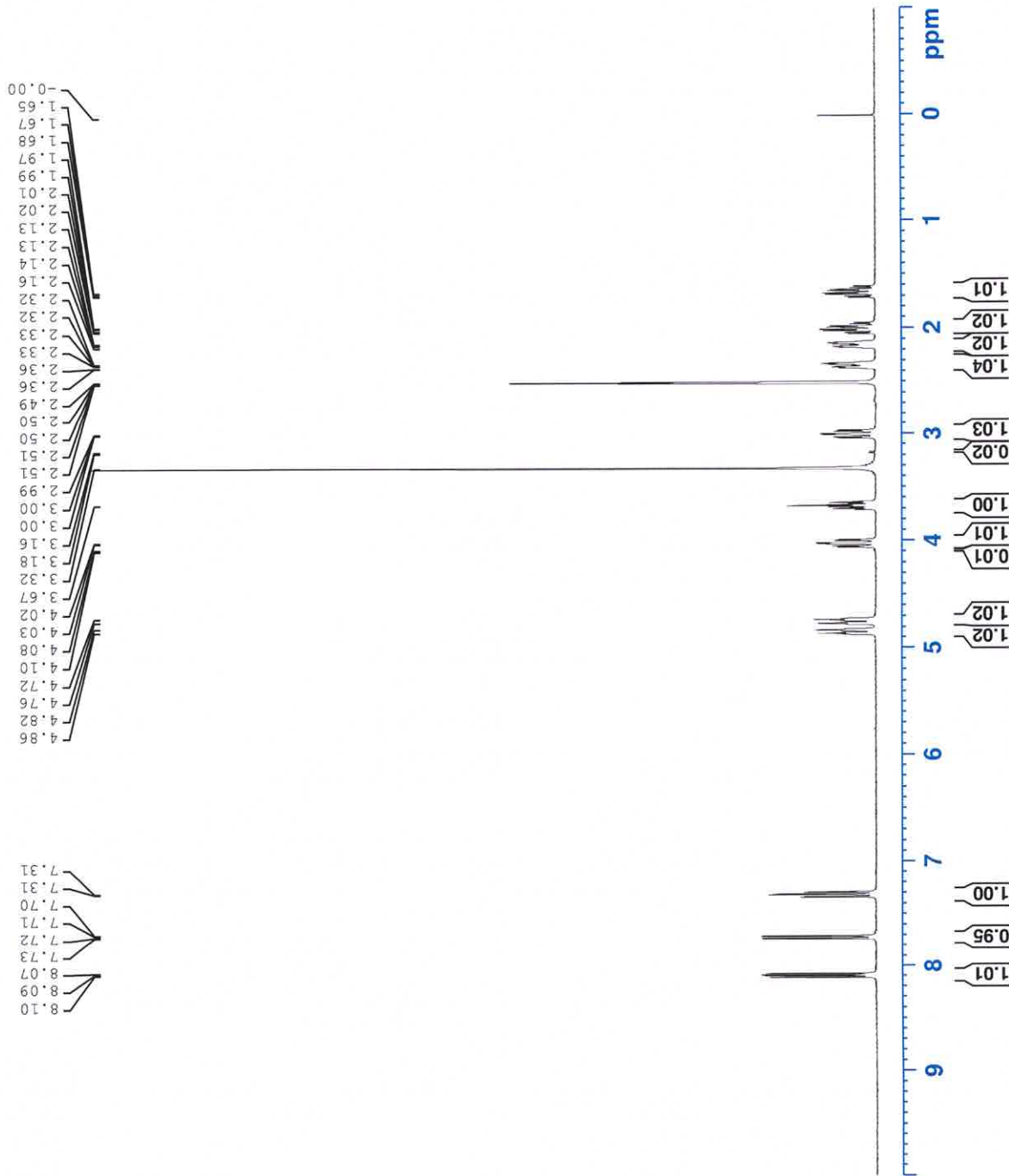


Current Data Parameters  
 NAME 2023-03-10 (NMR-400\_2)  
 EXPNO 155  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20230310  
 Time 16.13 h  
 INSTRUM spect  
 PROBHD 2116098\_0656 (zg30)  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO-d6  
 NS 4  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.293438 Hz  
 AQ 3.4078720 sec  
 RG 125.08  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 298.0 K  
 D1 1.00000000 sec  
 TD0 1  
 SFO1 400.1836016 MHz  
 NUC1 1H  
 P1 9.70 usec  
 PLW1 19.54100037 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1800019 MHz  
 EM  
 WDW 0  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 0.50



**TLC**  
 REVIEWED AND  
 APPROVED  
*For long* 03/17/2023