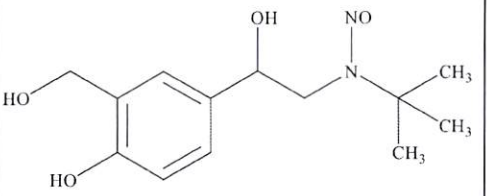
TLC PHARMACEUTICAL STANDARDS

Certificate of Analysis

Certificate No.: **20220216015**

Date: February 16, 2022 Retest date: February 14, 2025

Compound Name:	***N*-Nitroso-Salbutamol (*N*-Nitroso-Albuterol)**
Synonyms:	*N*-(*tert*-butyl)-*N*-(2-hydroxy-2-(4-hydroxy-3-(hydroxymethyl)phenyl)ethyl)nitrous amide
TLC Catalogue Number:	S-1781
CAS Number:	N/A
Molecular Weight:	268.31
Molecular Formula:	$C_{13}H_{20}N_2O_4$
Source:	TLC Pharmaceutical Standards
Source Lot No.:	4888-047A3
Storage Conditions:	Store at 2-8 °C
Solubility:	Methanol, DMSO



Test Description	**Specifications**	**Results**
Visual Description	Off-white solid	**Conforms**
Identification **MS** **^{1}H NMR**	 Conforms to structure Conforms to structure	 **Conforms** **Conforms**
Purity (HPLC)	Not less than 95.0%	**99.9%**
Impurity (HPLC)	N/A	
Water Content (KF)	N/A	**0.1%**
Residual Solvents (NMR)	1.0% ethyl acetate	
Assay (%)	Not less than 90.0%	**98.8%**
Recommendation:	**Release.** The compound is an *N*-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		02 / 17 / 2022
Approved by:	Quality Assurance		02 / 17 / 2022

Attachments: HPLC, MS and NMR spectra.

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Newmarket, Ontario L3Y 7B6 Canada
Phone: (905)-898-3645
Fax: (905)-898-0595

Data File E:\DATA\2022\2022-02-14\DEF_LC 2022-02-14 17-15-04\021-0401.D
Sample Name: 4888-047A3

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==========================================================================
Acq. Operator    : Jingjing Tong                  Seq. Line :   4
Acq. Instrument  : LC-1260_2                       Location : Vial 21
Injection Date   : 2/14/2022 6:55:41 PM               Inj :   1
                                                 Inj Volume : 2.000 µl
Acq. Method      : E:\DATA\2022\2022-02-14\DEF_LC 2022-02-14 17-15-04\4.M
Last changed     : 2/14/2022 5:15:04 PM by Jingjing Tong
Analysis Method  : C:\CHEM32\1\METHODS\1.M
Last changed     : 2/15/2022 8:53:34 AM by Jingjing Tong
                   (modified after loading)
Sample Info      : ZORBAX Eclipse Plus-C18 (4.6*100mm,3.5um); F=1.0ml/min; T=30 degree;
                   CH3OH/10mmol/L CH3COONH4+0.03% TEA=30/70(0-6min),30/70-55/45(6-16min),55/45
                   (after 16min)
                   ~0.7mg in 0.6mL CH3OH
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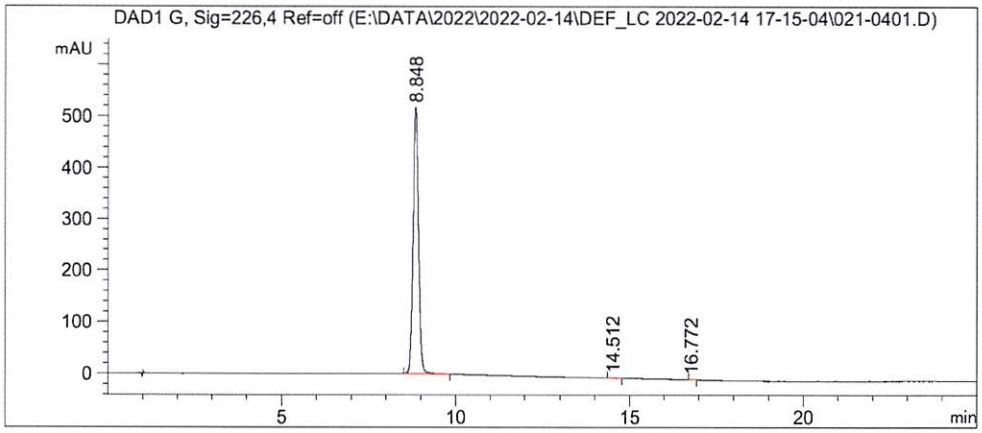


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==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 G, Sig=226,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.848 BBA   0.1693 5640.15869  517.04510  99.8574
   2  14.512 BBA   0.1050    5.24870 7.75370e-1   0.0929
   3  16.772 BBA   0.0810    2.80423 4.74088e-1   0.0496

Totals :                  5648.21162  518.29456

==========================================================================
                          *** End of Report ***
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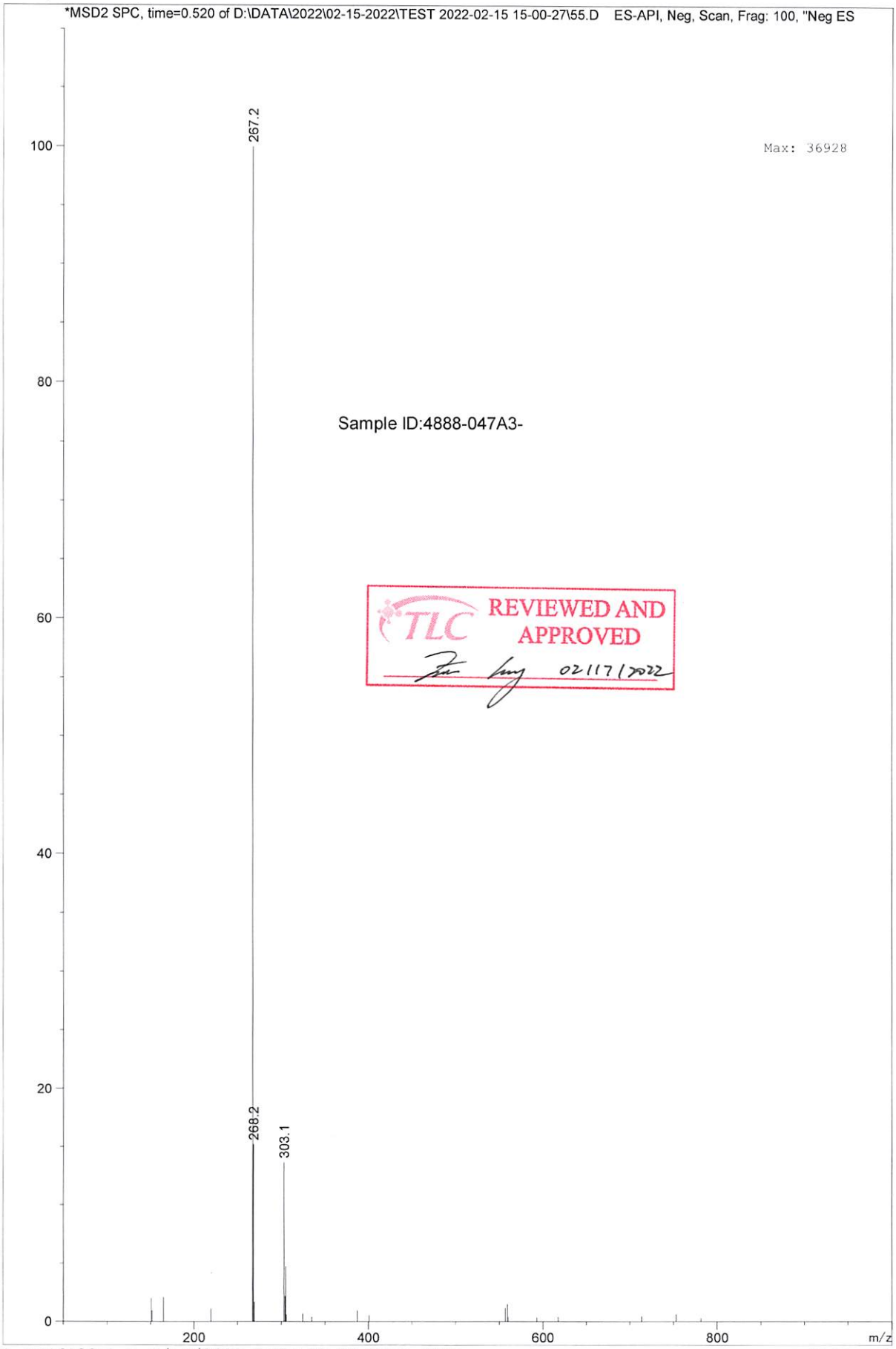
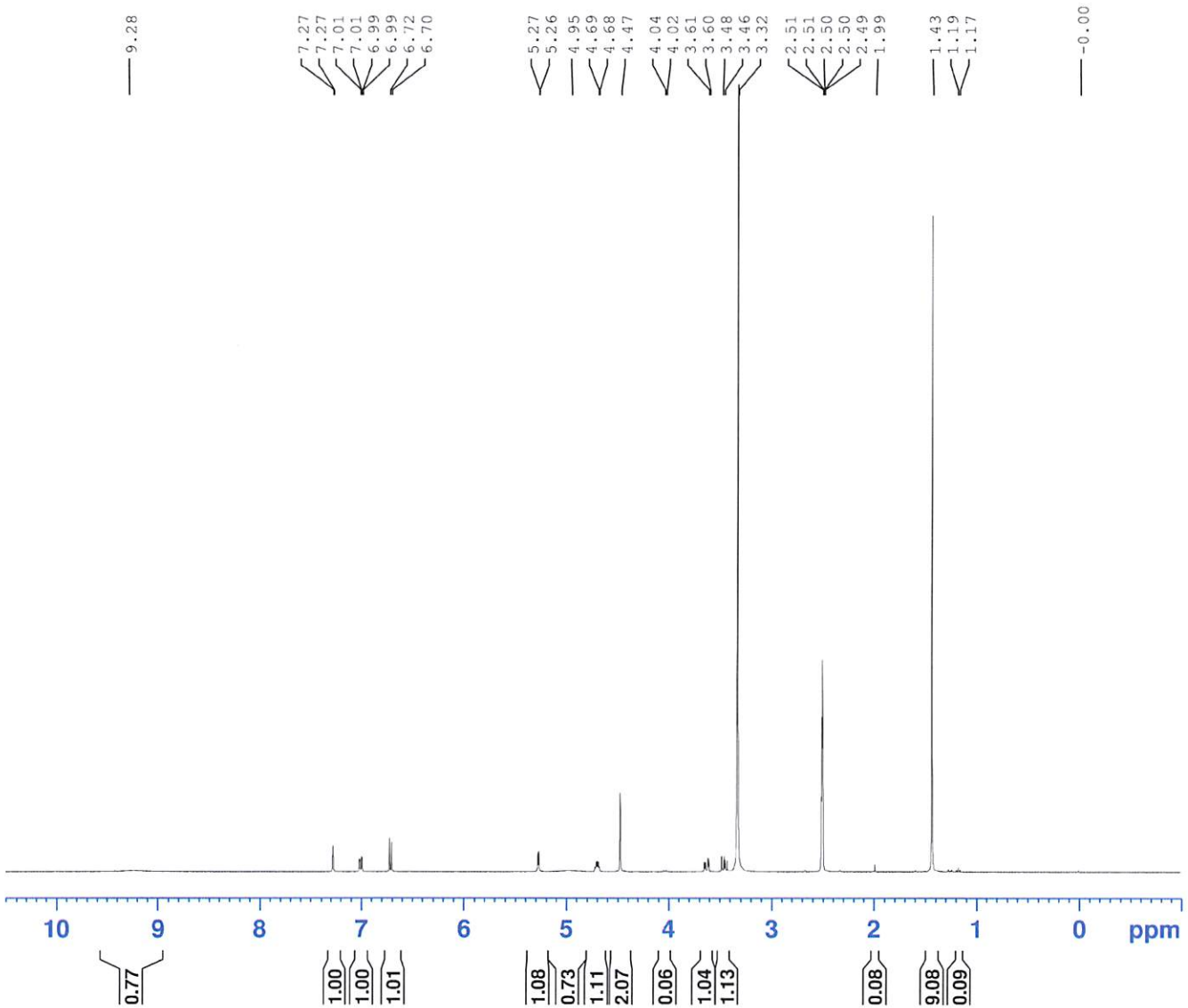
TLC REVIEWED AND APPROVED 02/17/2022

MS Spectrum


*MSD2 SPC, time=0.520 of D:\DATA\2022\02-15-2022\TEST 2022-02-15 15-00-27\55.D ES-API, Neg, Scan, Frag: 100, "Neg ES
Max: 36928
267.2
268.2
303.1
Sample ID:4888-047A3-
TLC
REVIEWED AND APPROVED
02/17/2022
100
80
60
40
20
0
200
400
600
800
m/z

4888-047A3 1H NMR in DMSO-d6



BRUKER

Current Data Parameters
NAME 2022-02-14 (NMR-400_2)
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220214
Time 11.17 h
INSTRUM spect
PROBHD Z116098_0656 (
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 9.40 usec
PLW1 19.54100037 W

F2 - Processing parameters
SI 65536
SF 400.1800027 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50

TLC REVIEWED AND APPROVED
02/17/2022