Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Three-Component Reaction of *C,N*-Cyclic *N'*-Acyl Azomethine Imines, Isocyanides, and Azide Compounds: Effective Synthesis of 1,5-Disubstituted Tetrazoles with Tetrahydroisoquinoline Skeletons

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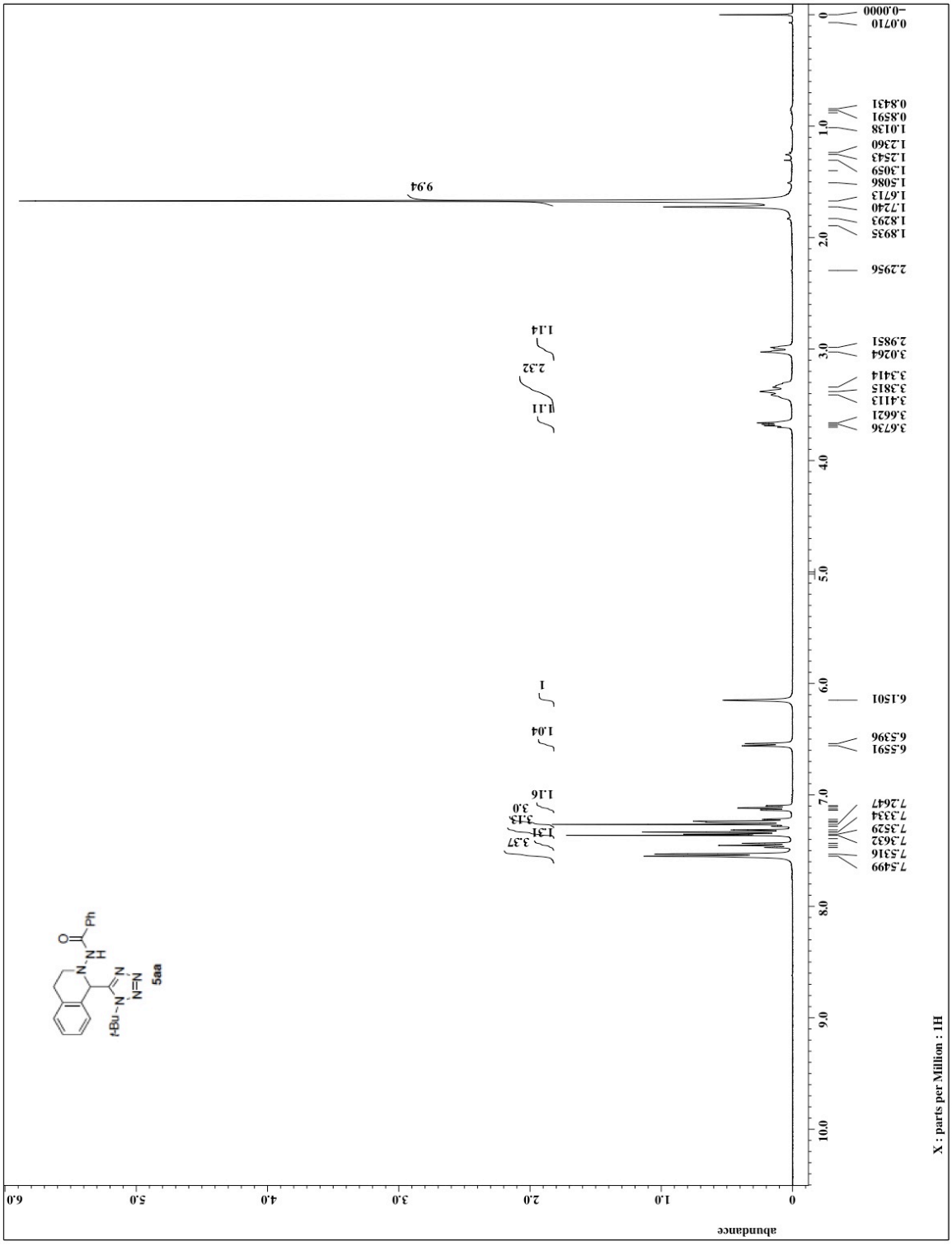
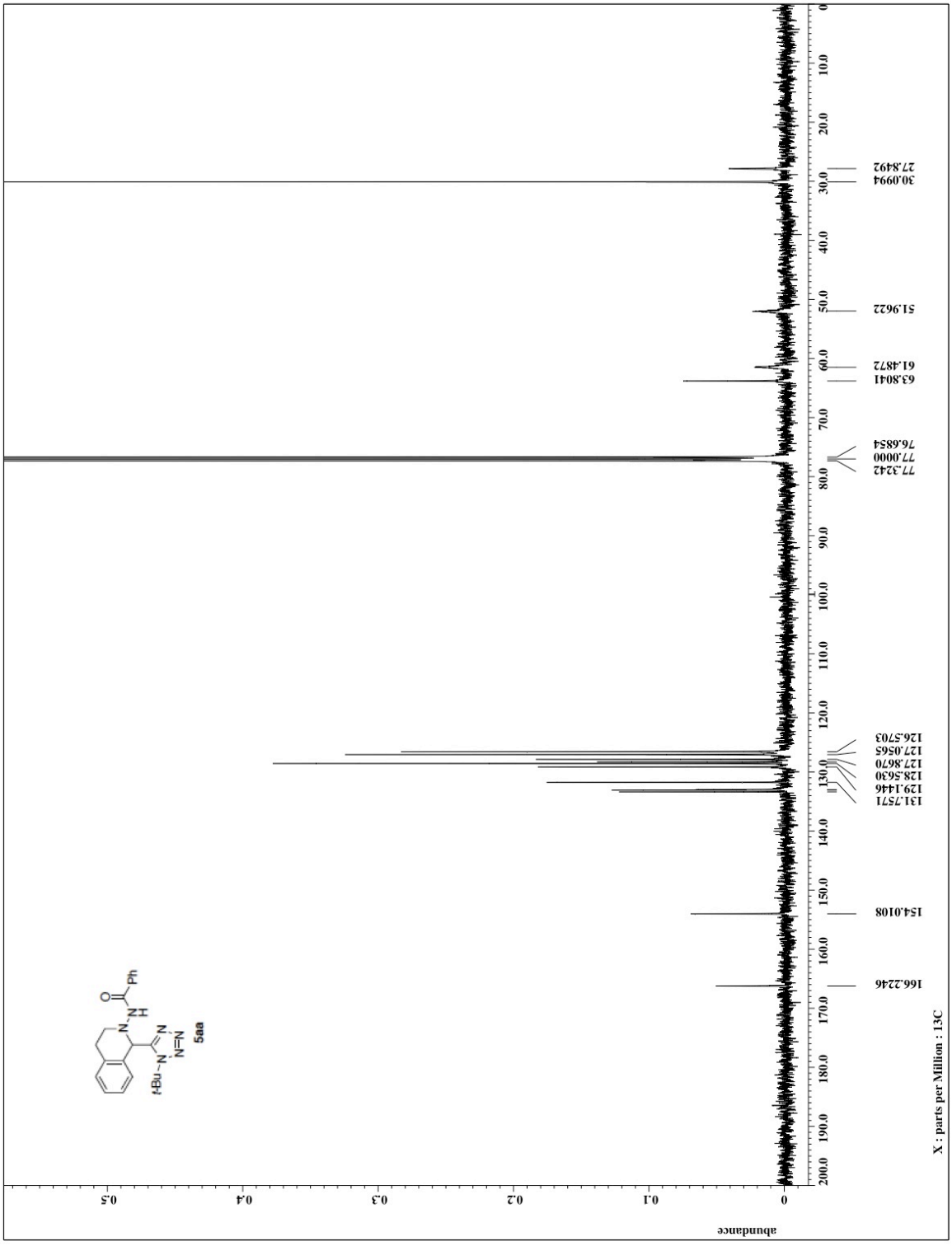
General: ^{1}H NMR was recorded on a JEOL ECS 400 (400 MHz) NMR spectrometer. Chemical shifts δ are reported in ppm using TMS as an internal standard. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (J) and integration. ^{13}C NMR spectra were recorded on JEOL ECS 400 (100 MHz) NMR spectrometer. The chemical shifts were determined in the δ-scale relative to $CDCl_3$ (δ = 77.0 ppm). The IR spectra were measured on JASCO FT/IR-230 spectrometers. The MS spectra was recorded with JEOL SX-102A mass spectrometers. All of the melting points were measured with YANAGIMOTO micro melting point apparatus. Toluene was dried and distilled over sodium. THF, Et_2O and 1,4-dioxane were freshly distilled from sodium

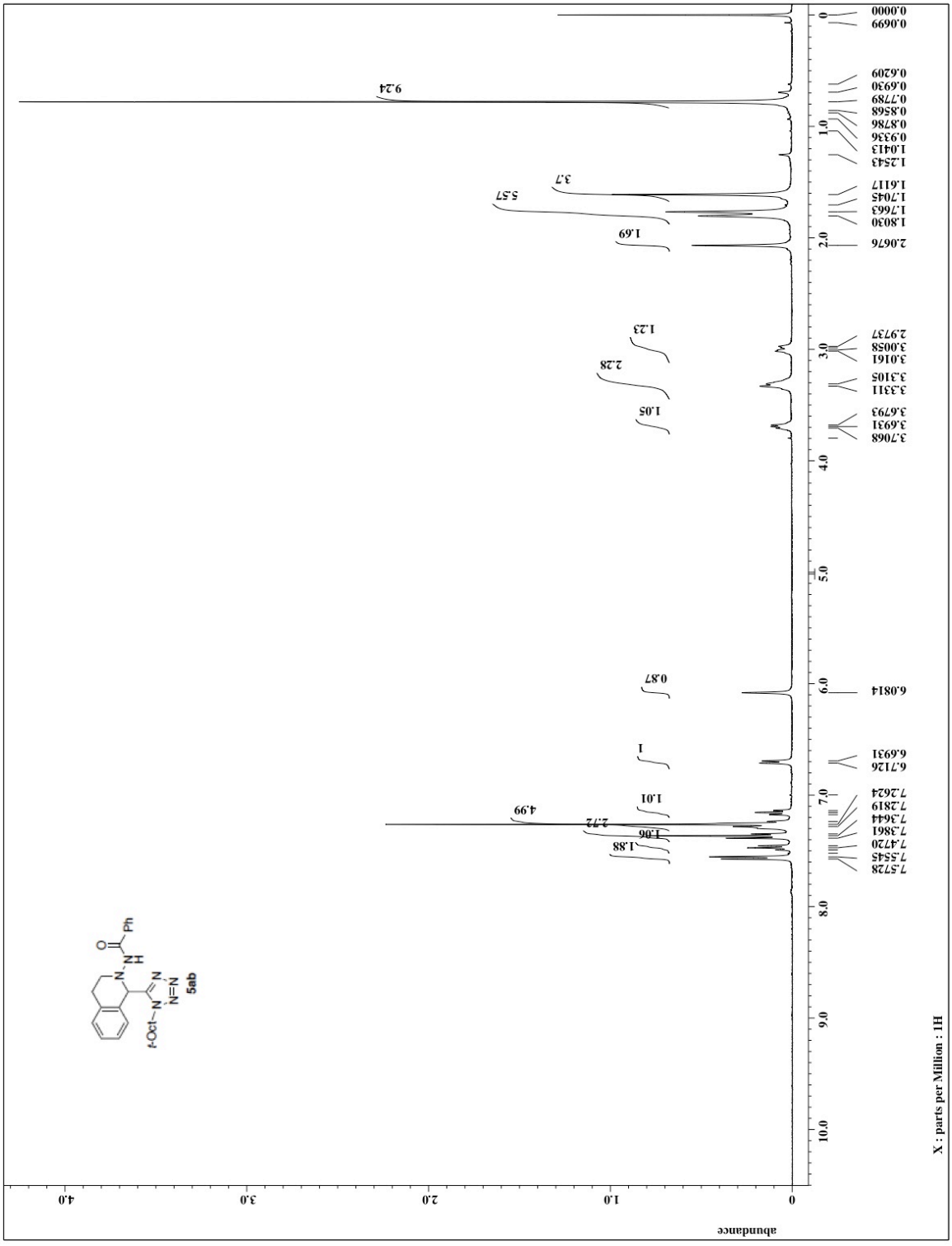
diphenylketyl. All other solvents were distilled and stored over drying agents. Flash column chromatography was performed by using Cica silica gel 60N, spherical neutral (37563-84).

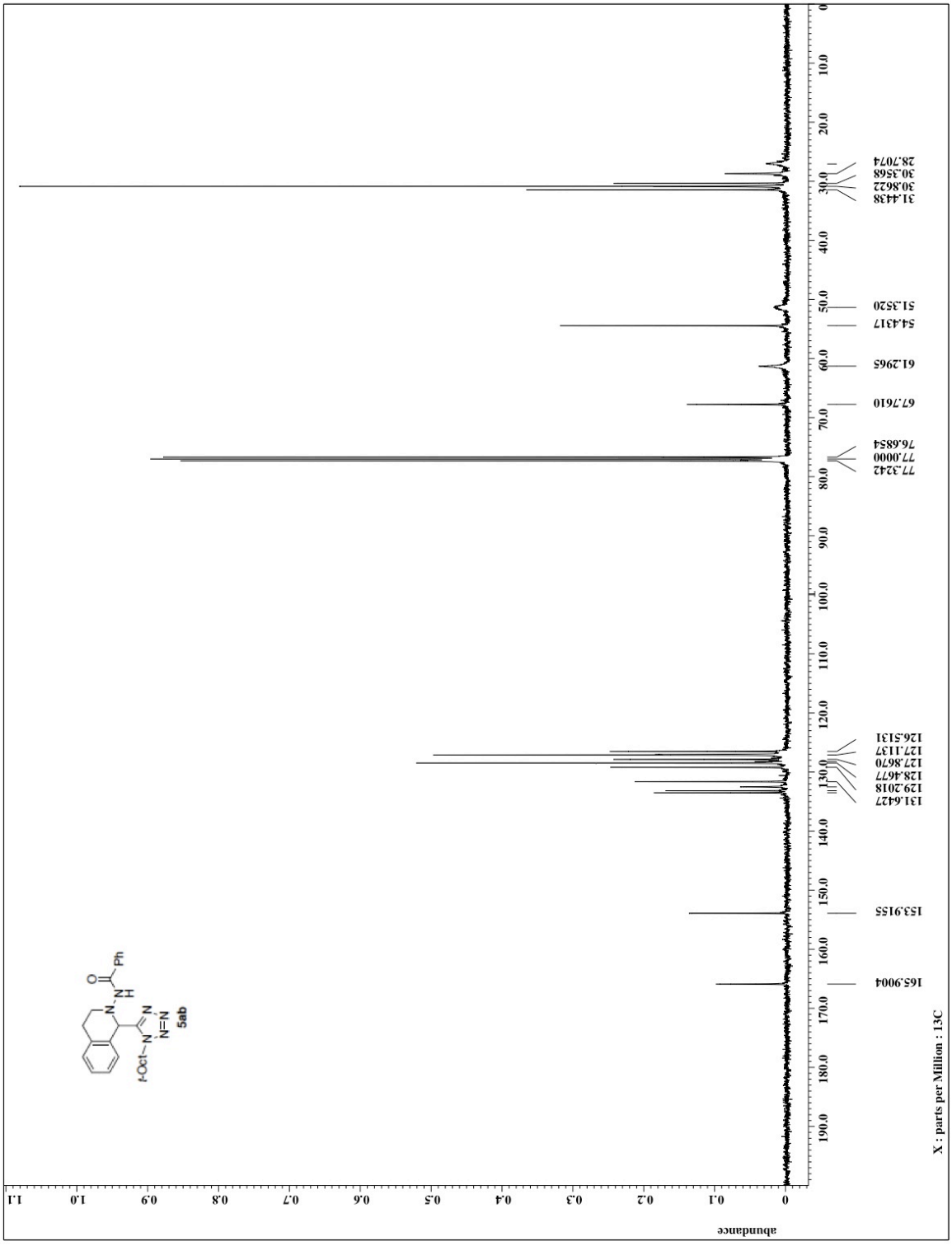
C,N-Cyclic *N'*-acyl azomethine imines **1** were prepared according to the reported procedure.[1]

1 (a) Hashimoto, T.; Omote, M.; Maruoka, K. *Angew. Chem. Int. Ed.* **2011**, *50*, 3489–3492. (b) Hashimoto, T.; Maeda, Y.; Omote, M.; Nakatsu, H.; Maruoka, K. *J. Am. Chem. Soc.* **2010**, *132*, 4076–4077.





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abundance
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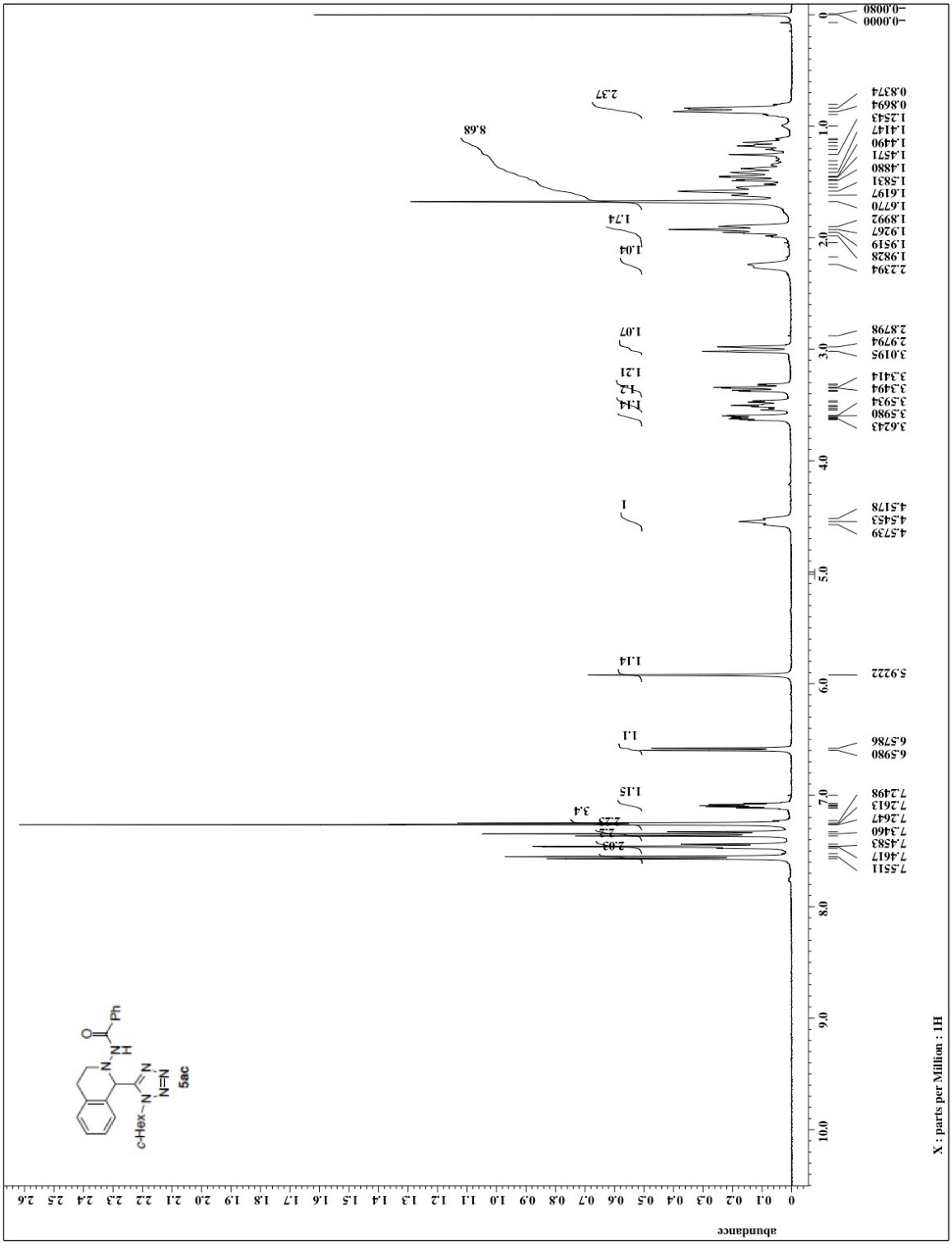

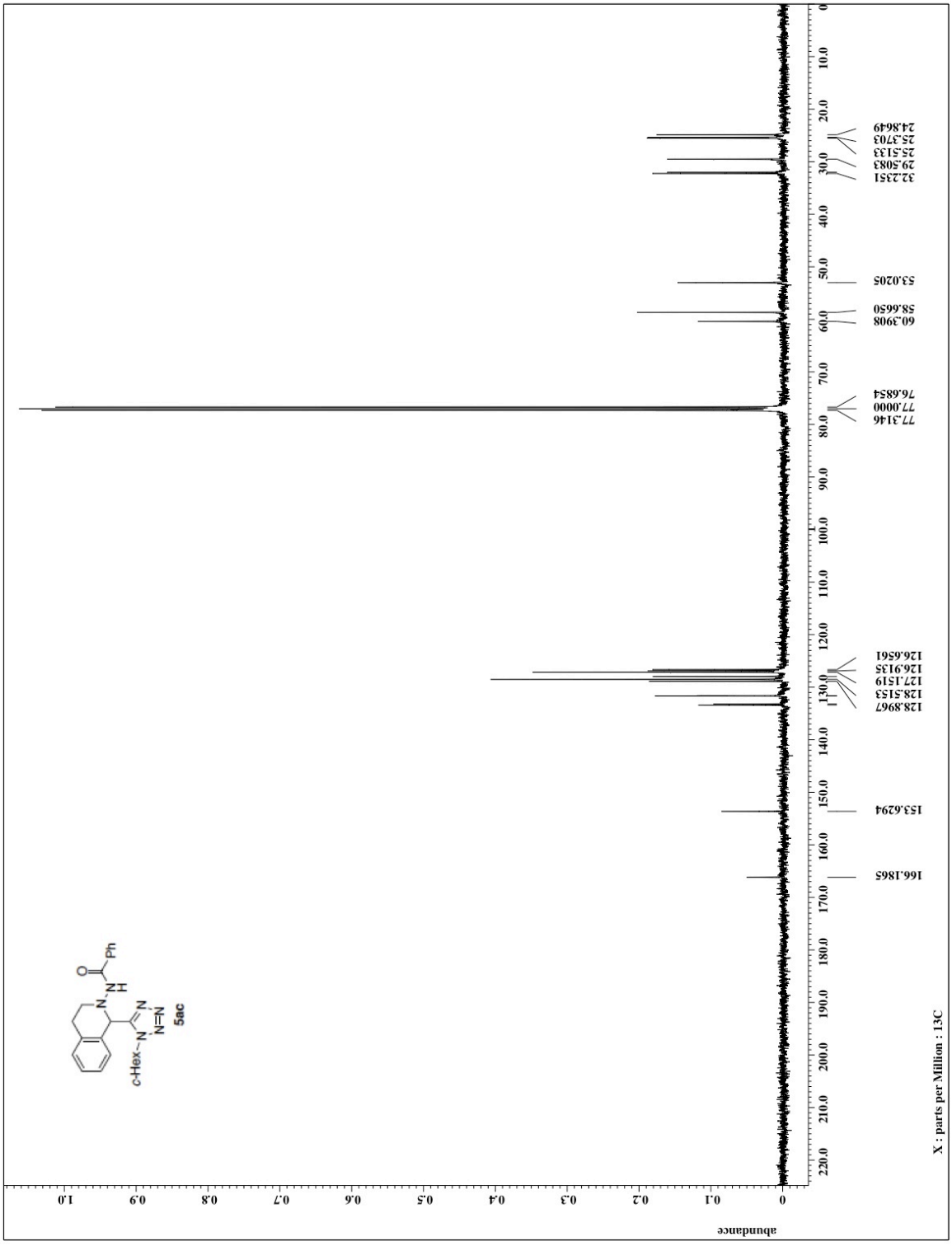

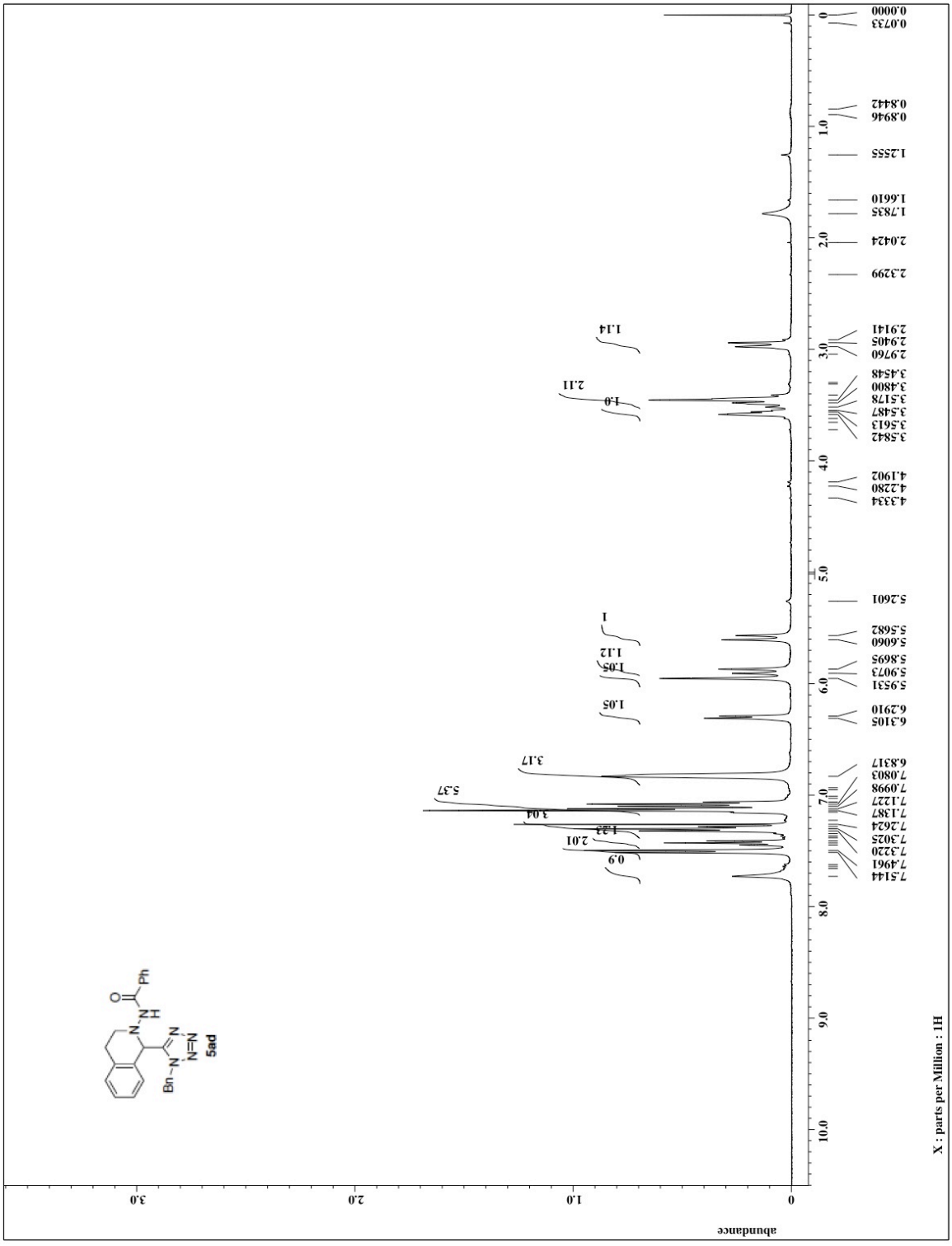

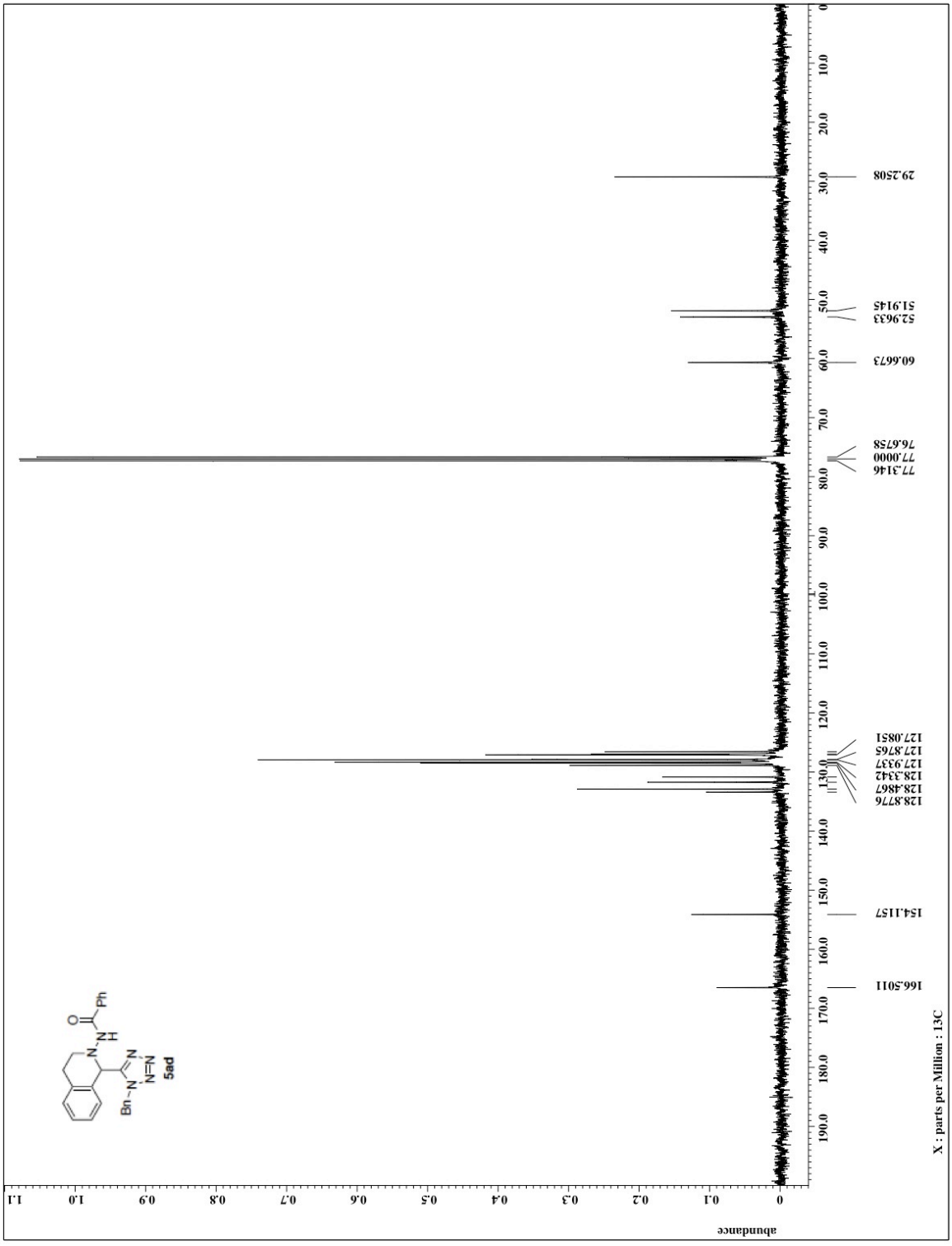

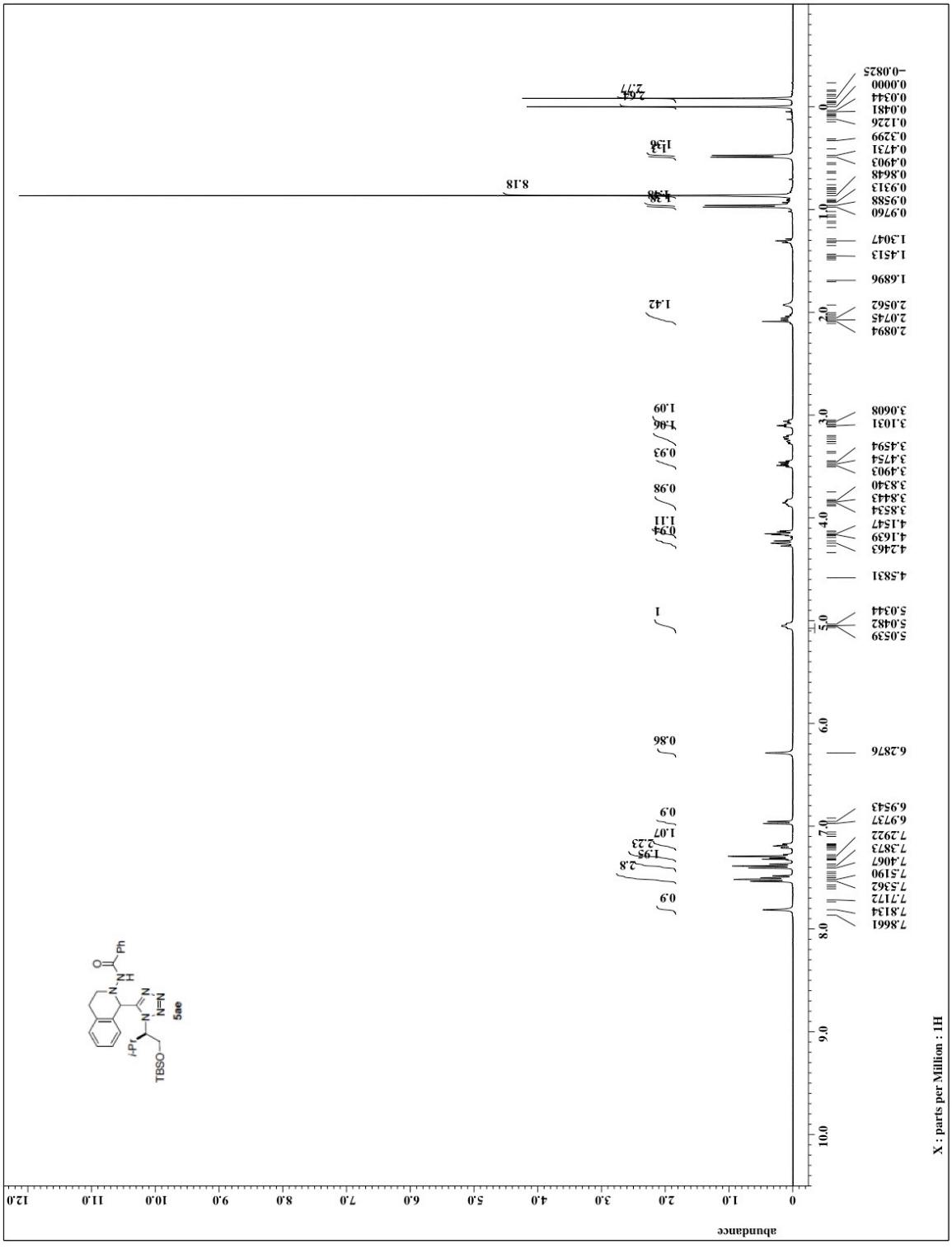

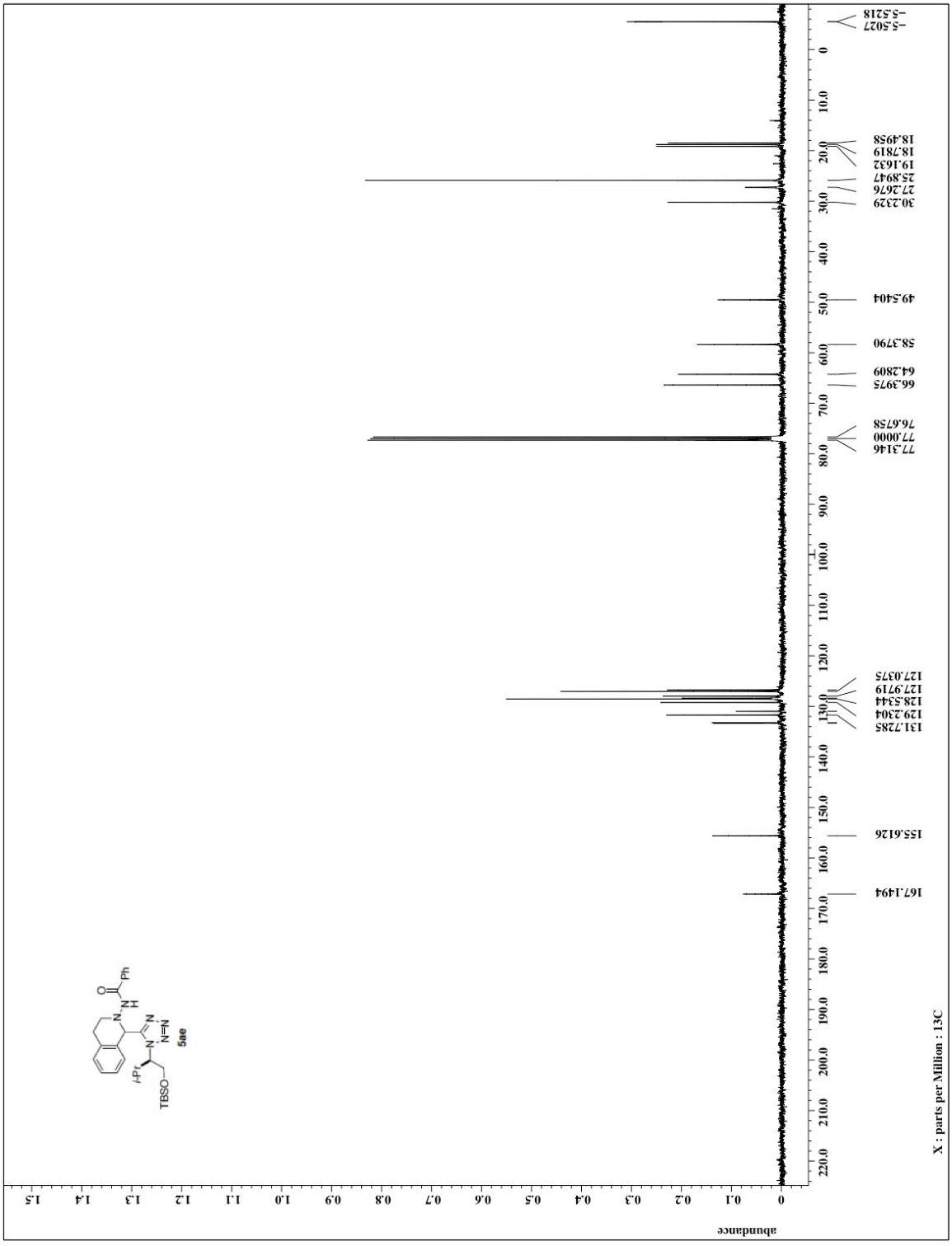

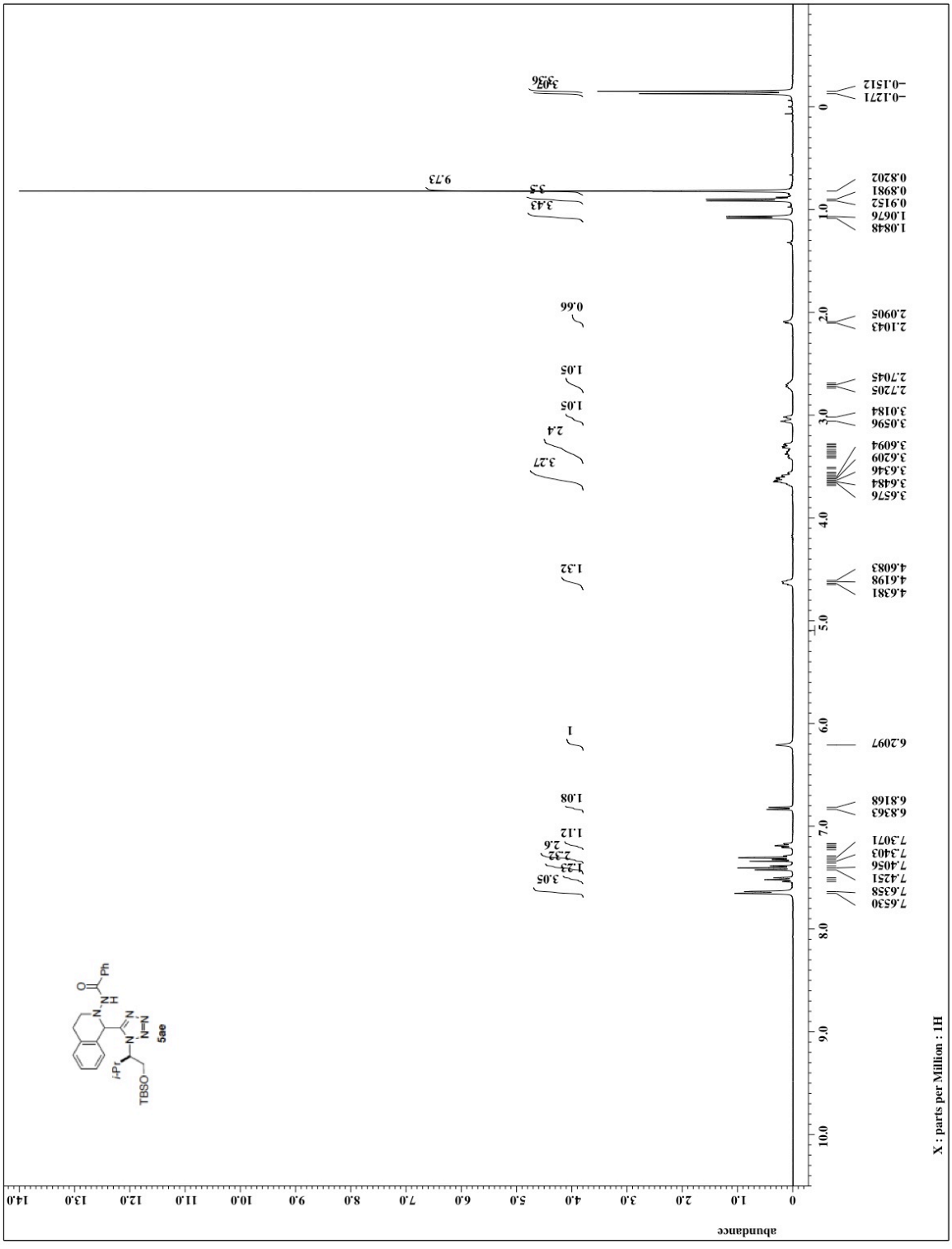

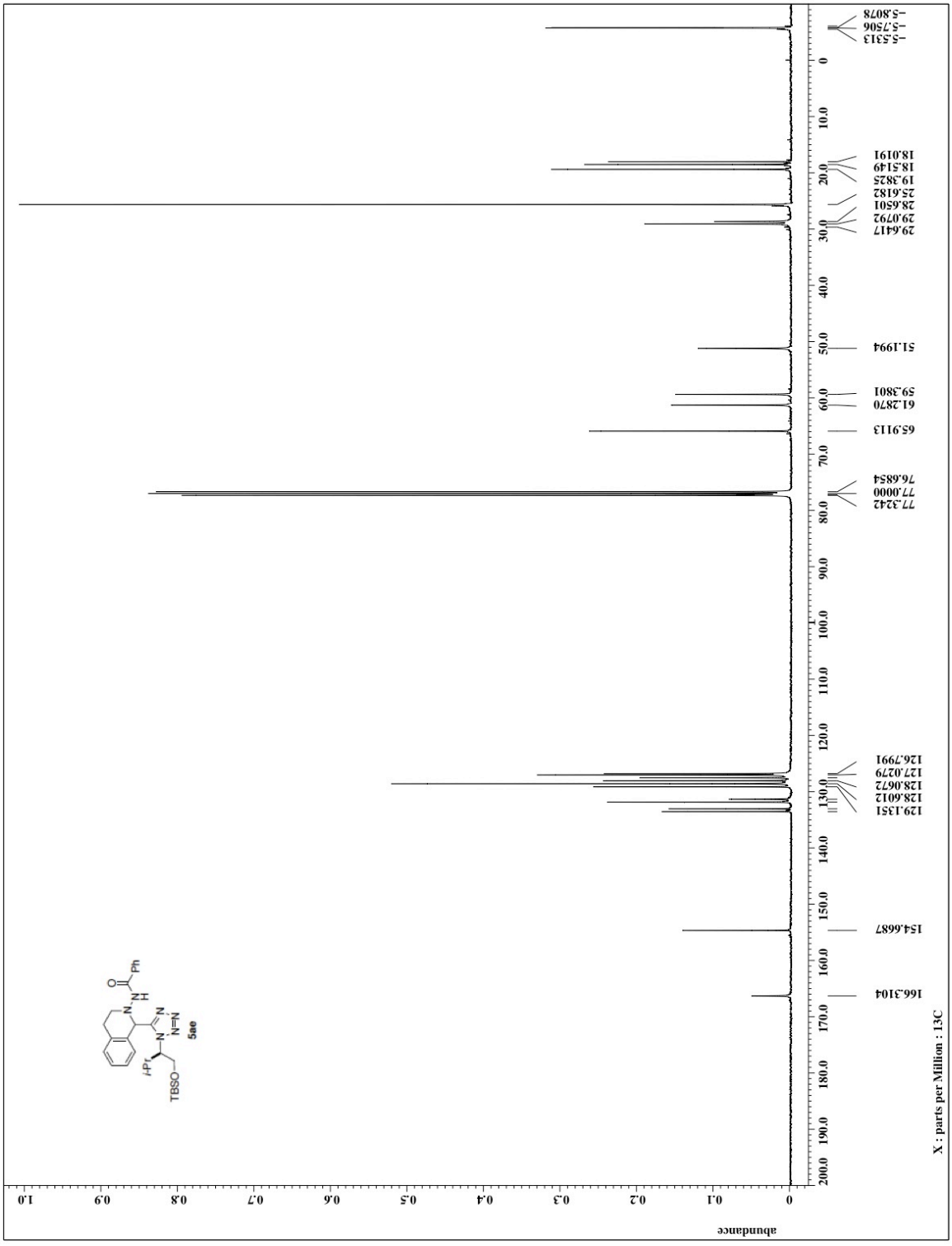

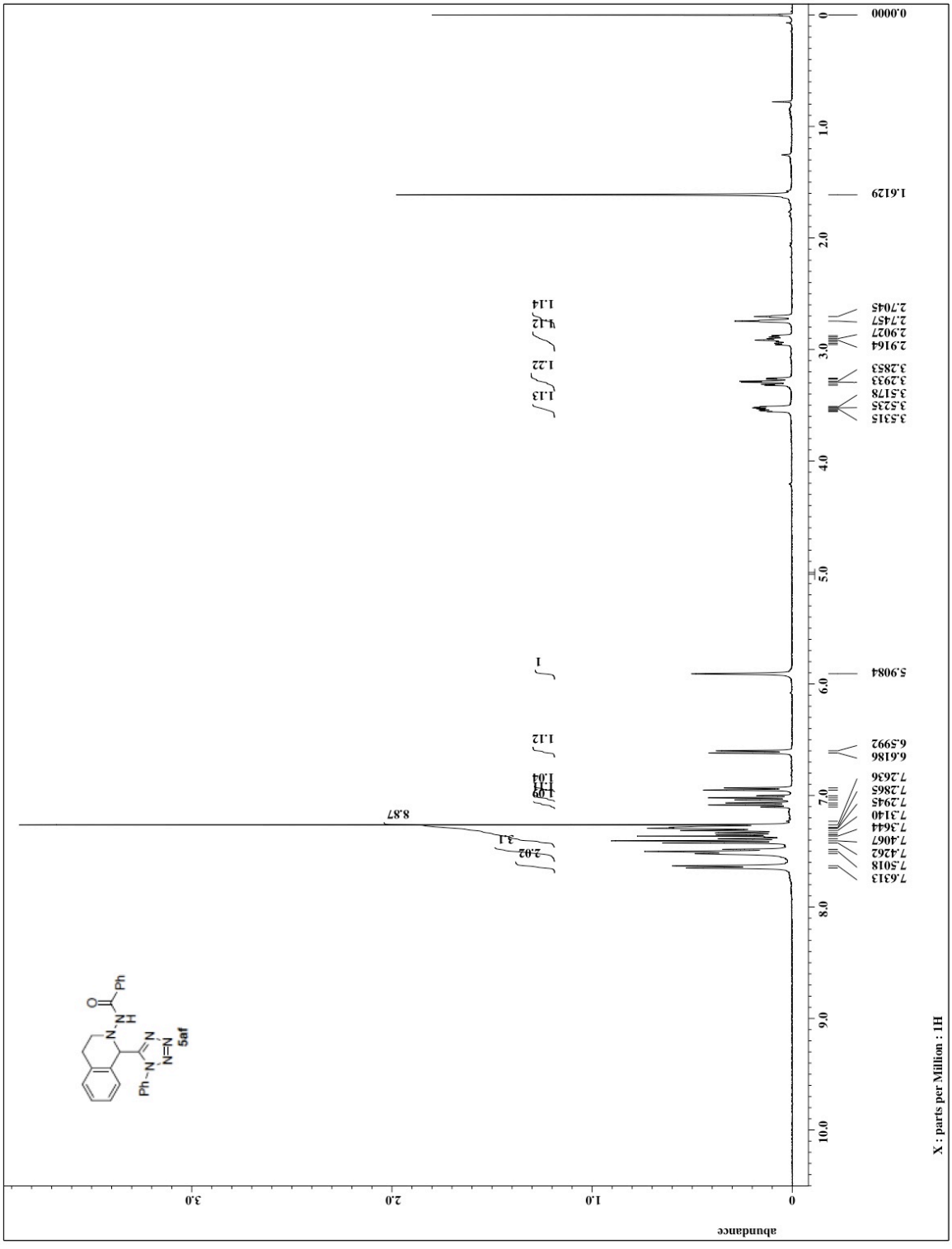

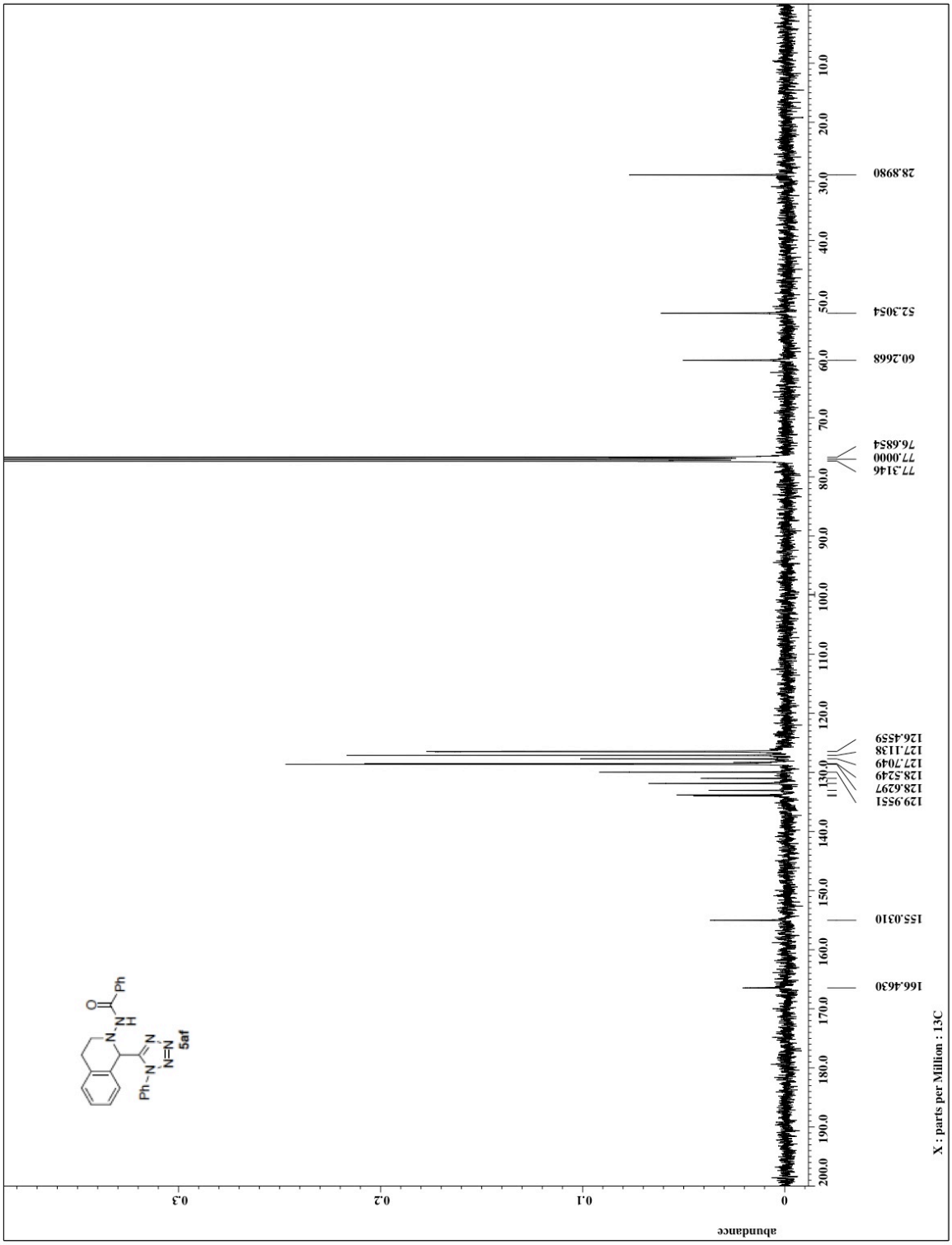

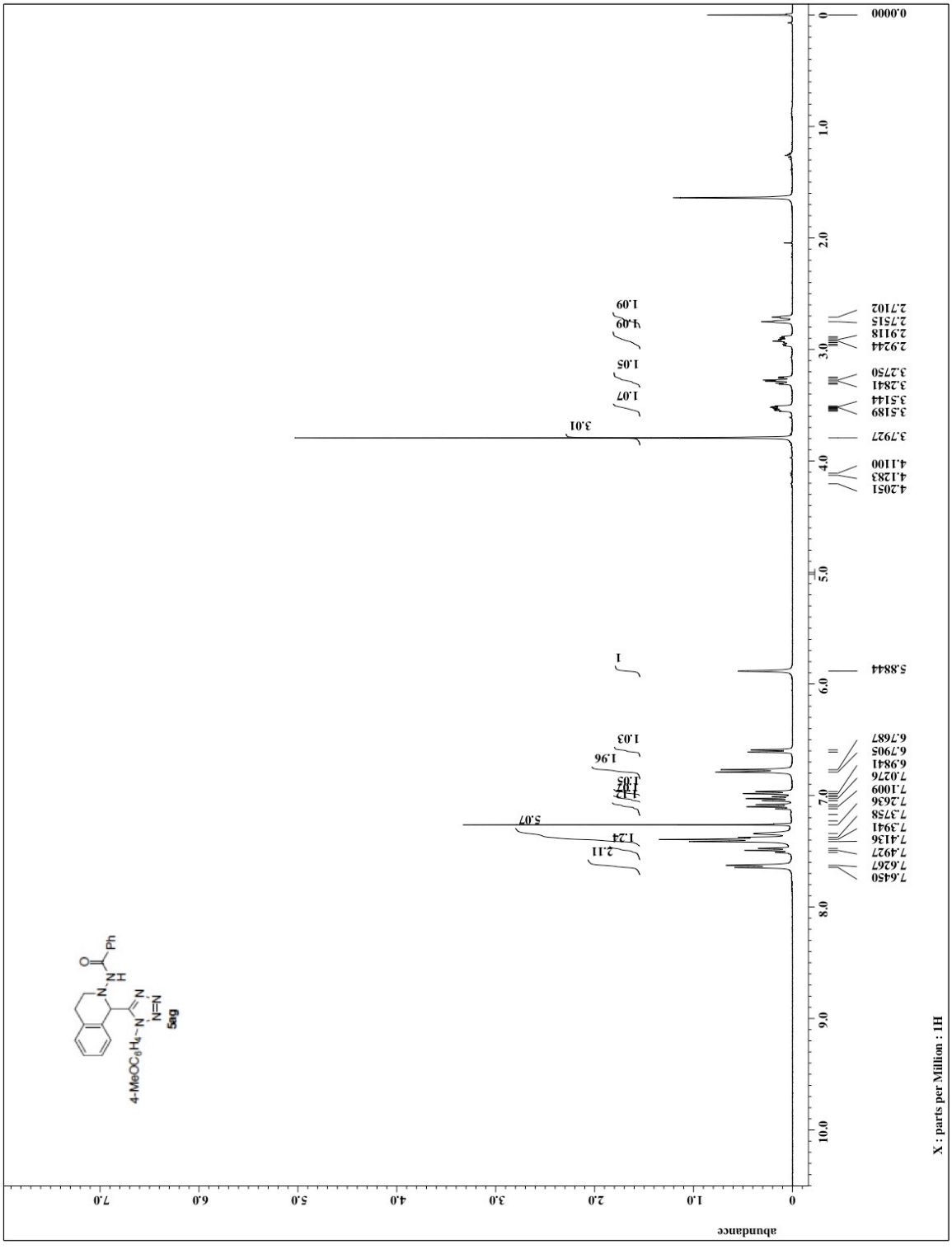
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Ph
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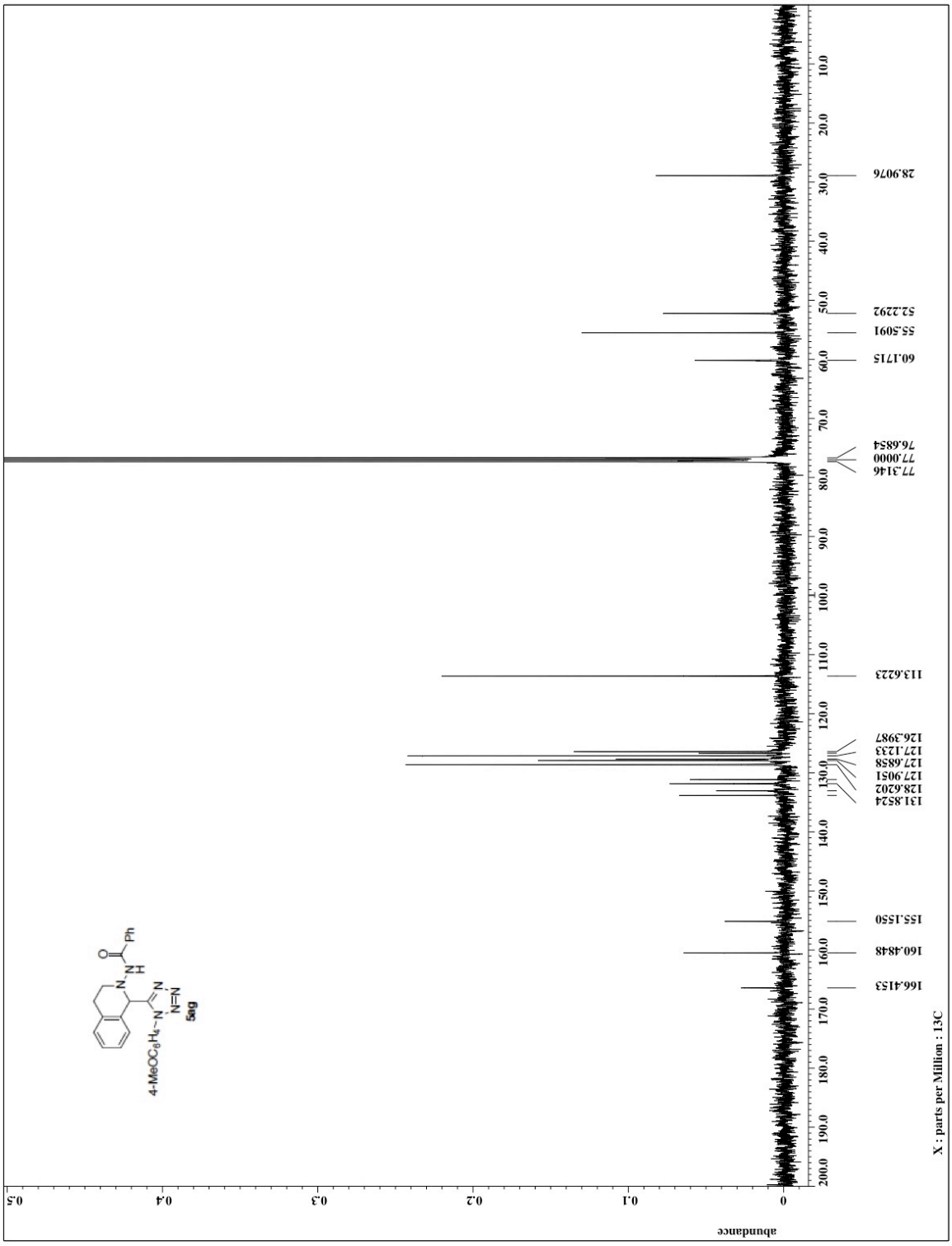
abundance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X : parts per Million : 13C
abundance
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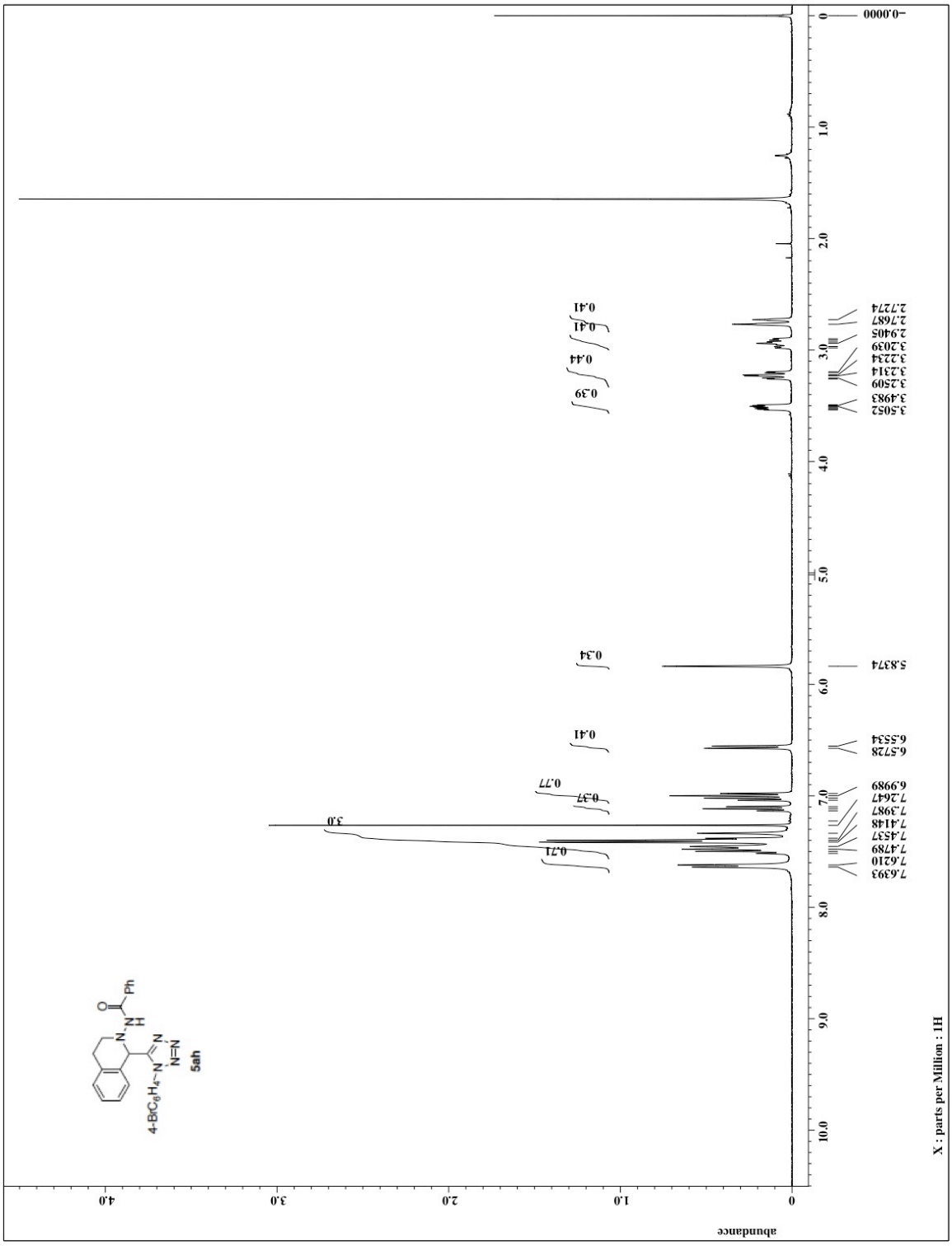

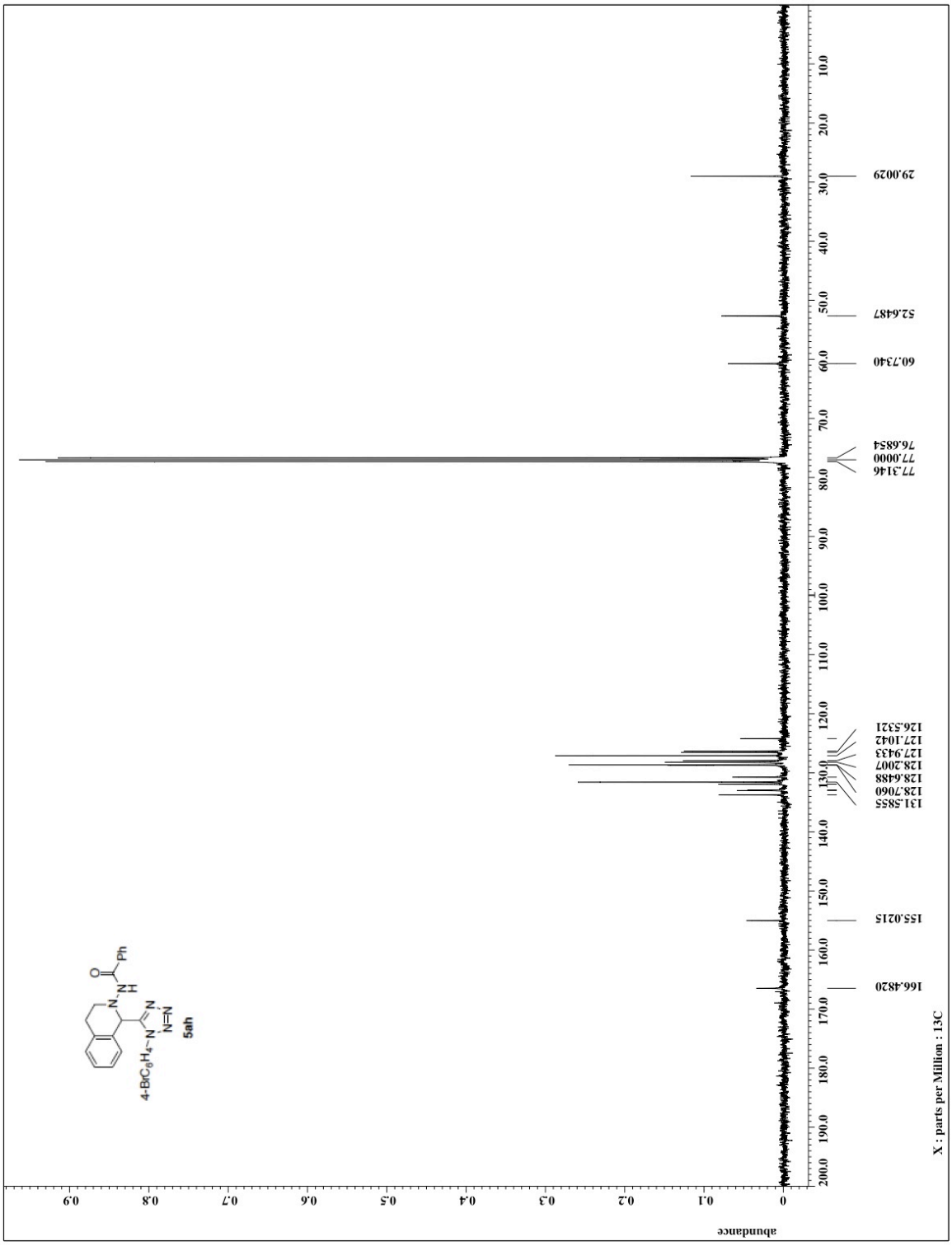

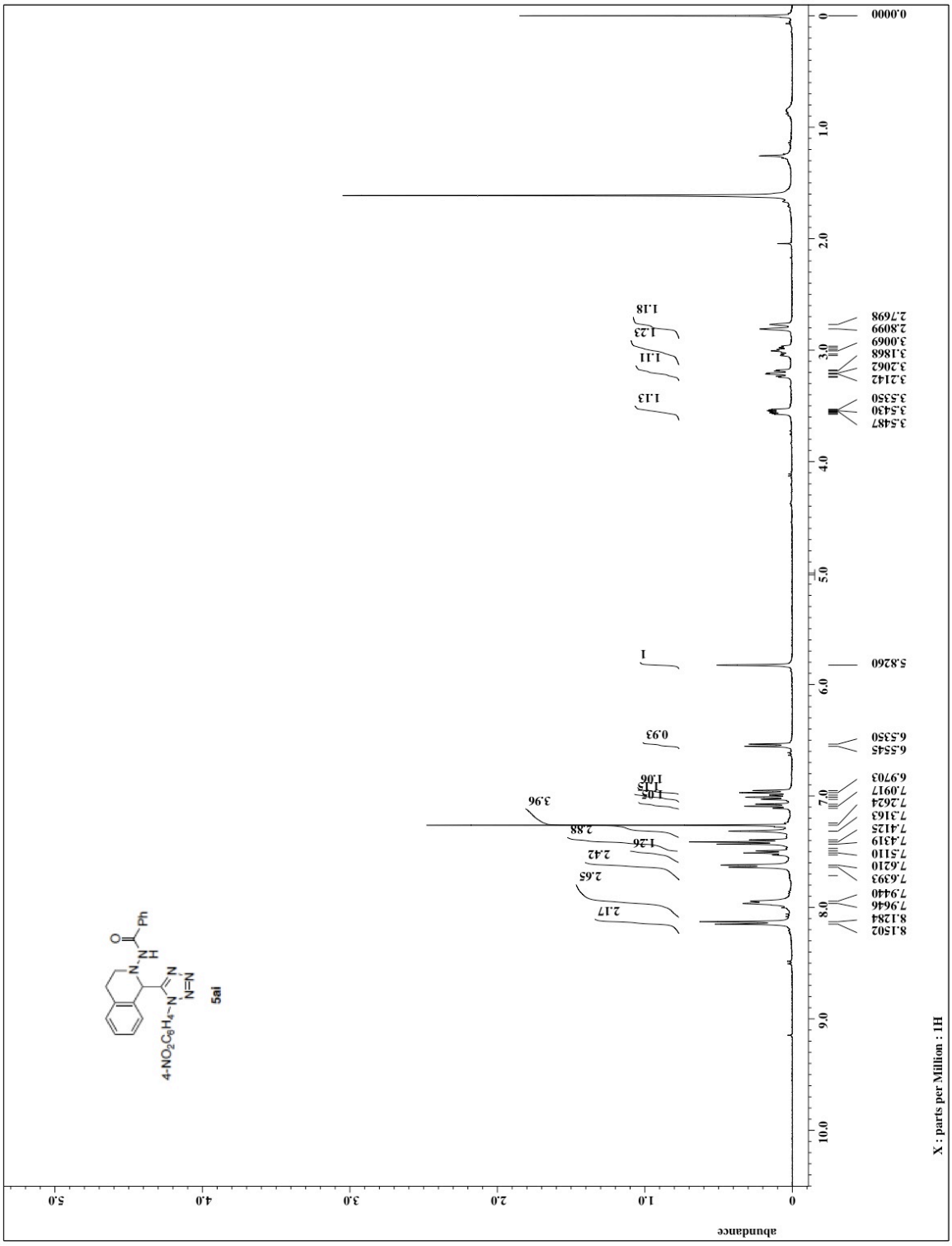

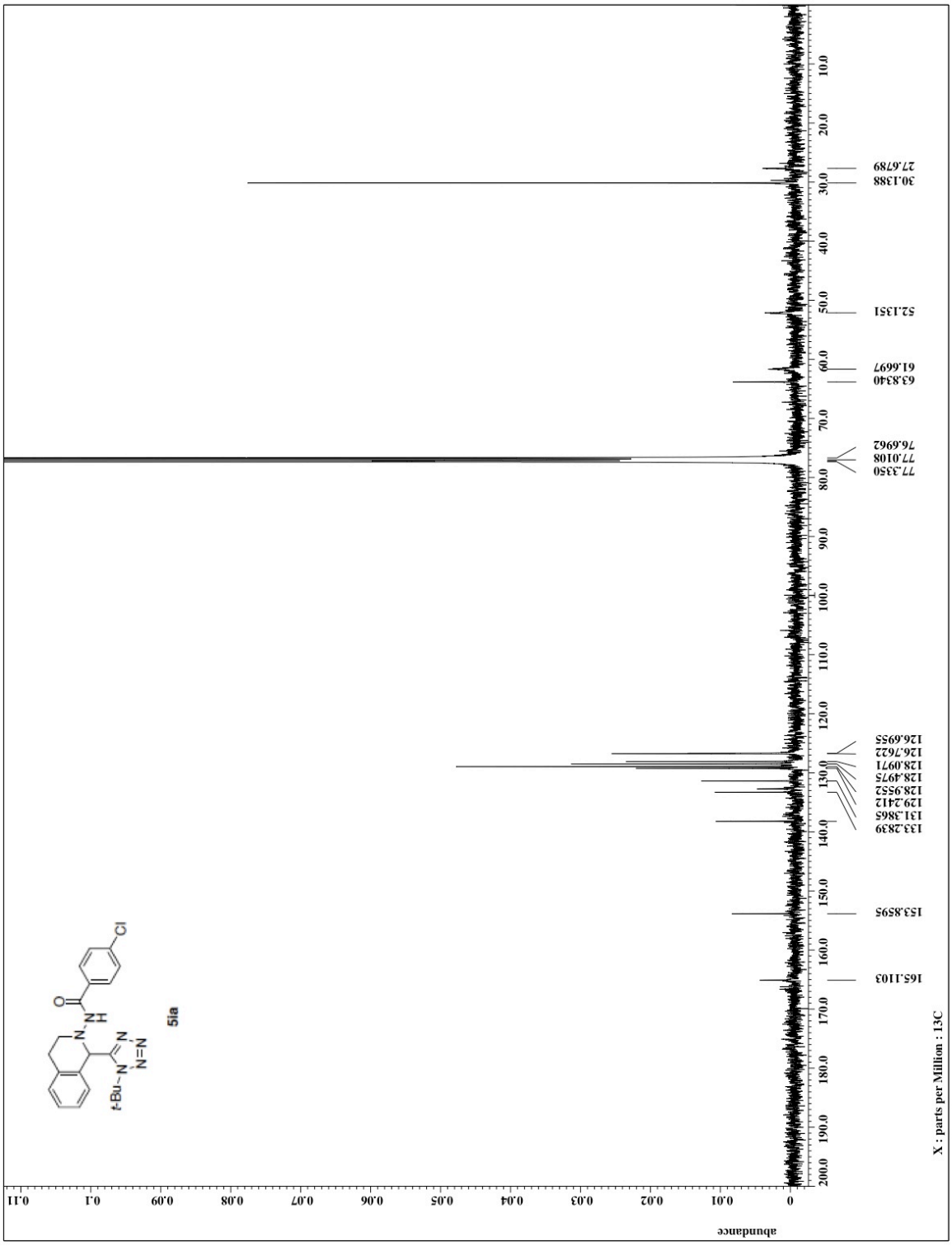

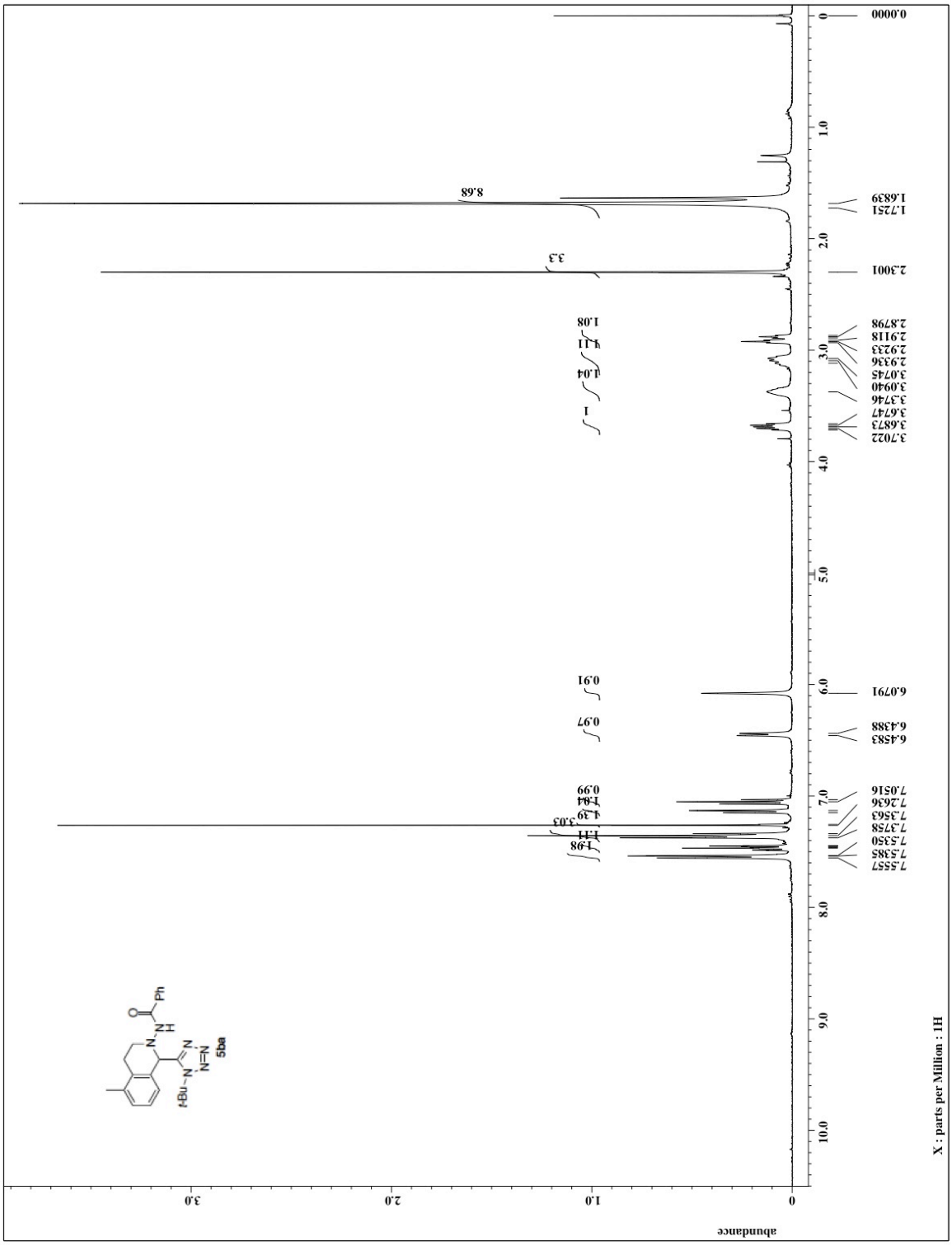

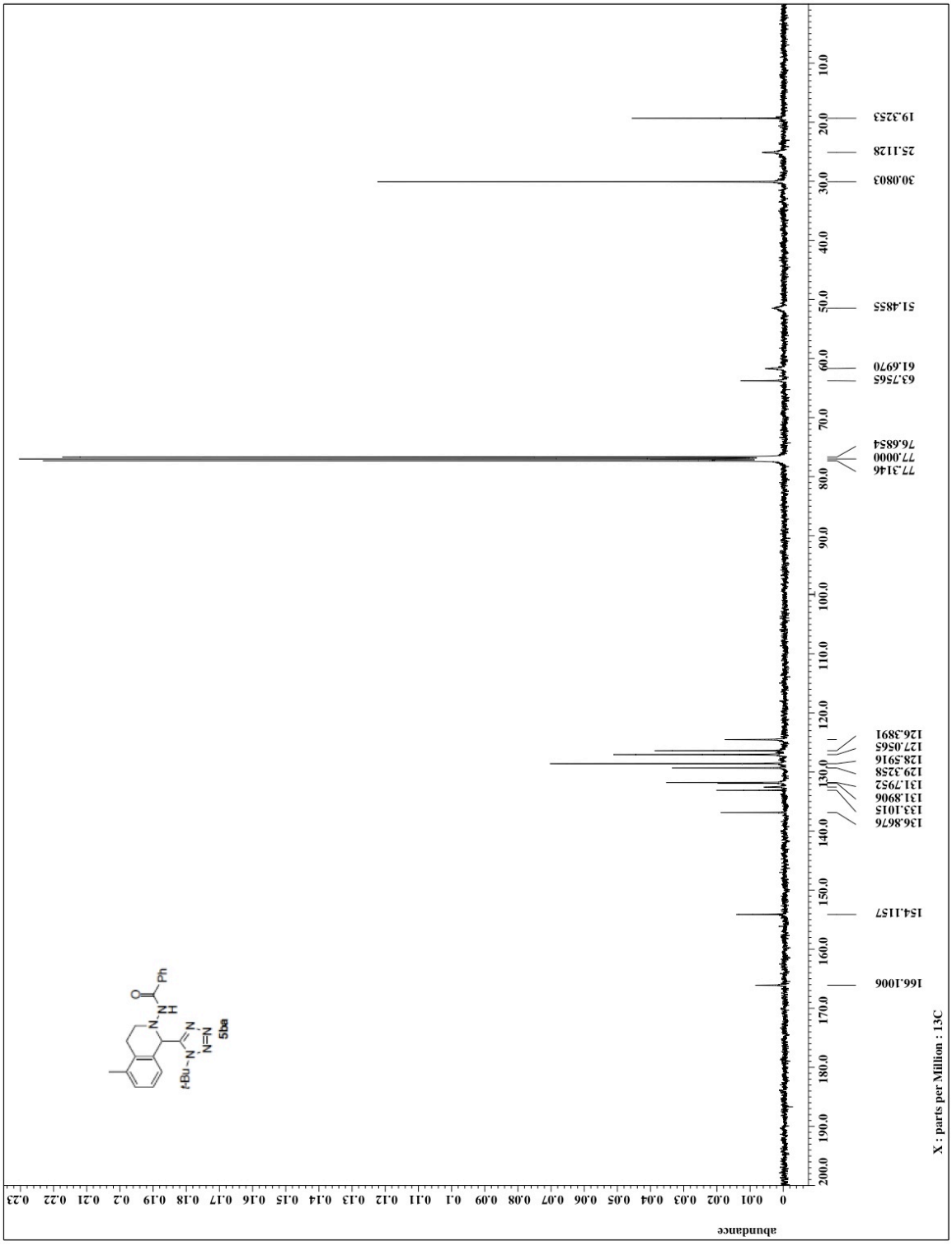

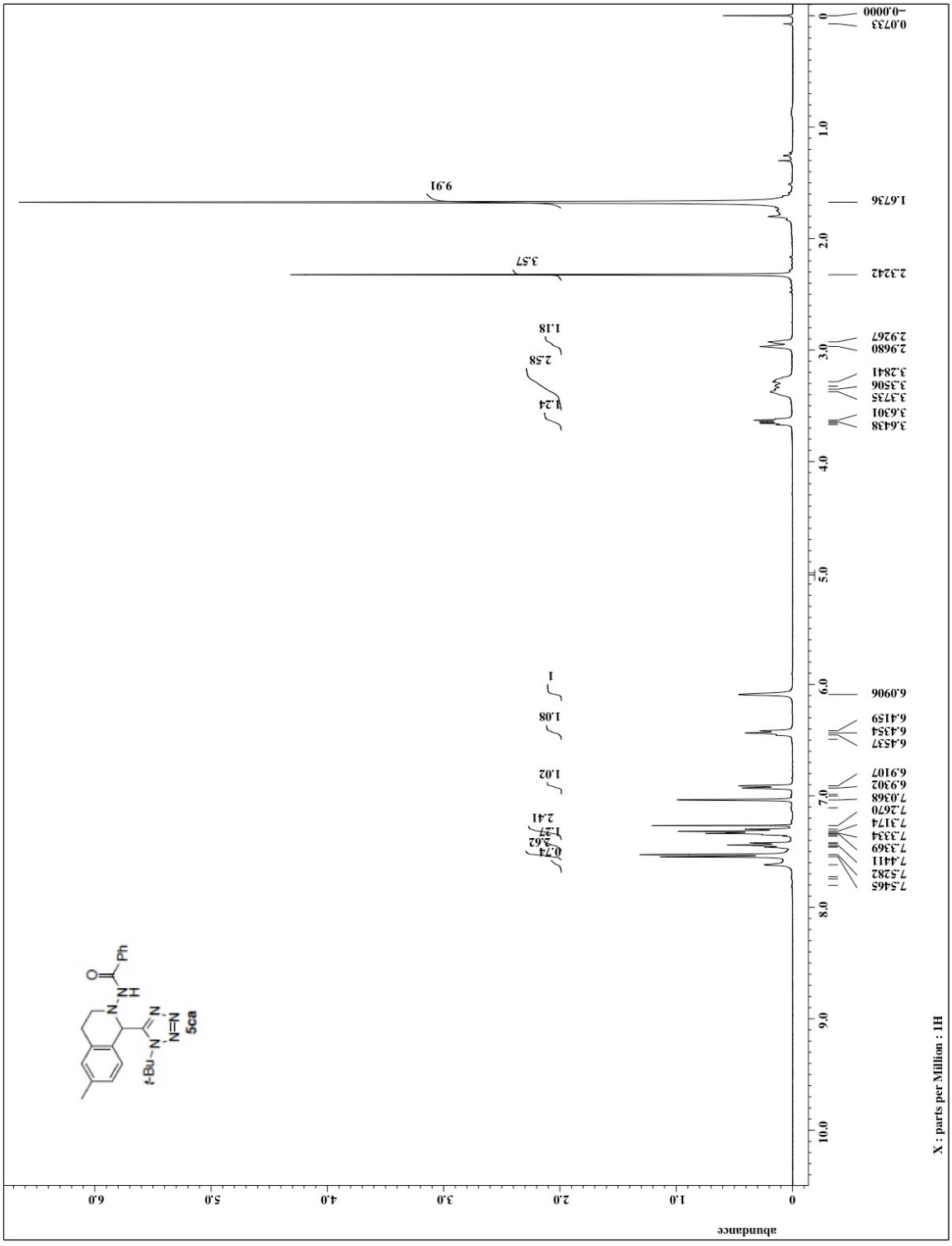

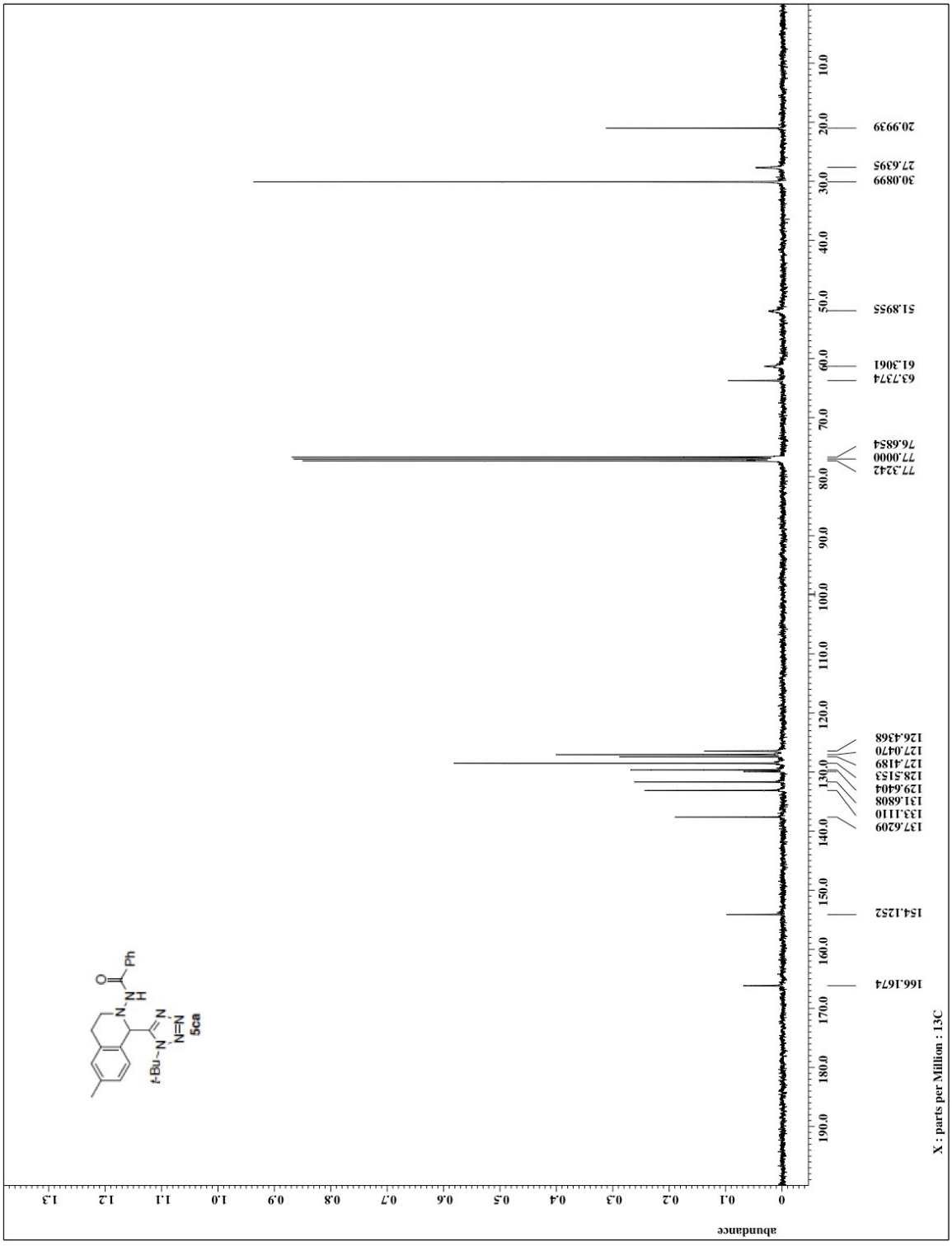

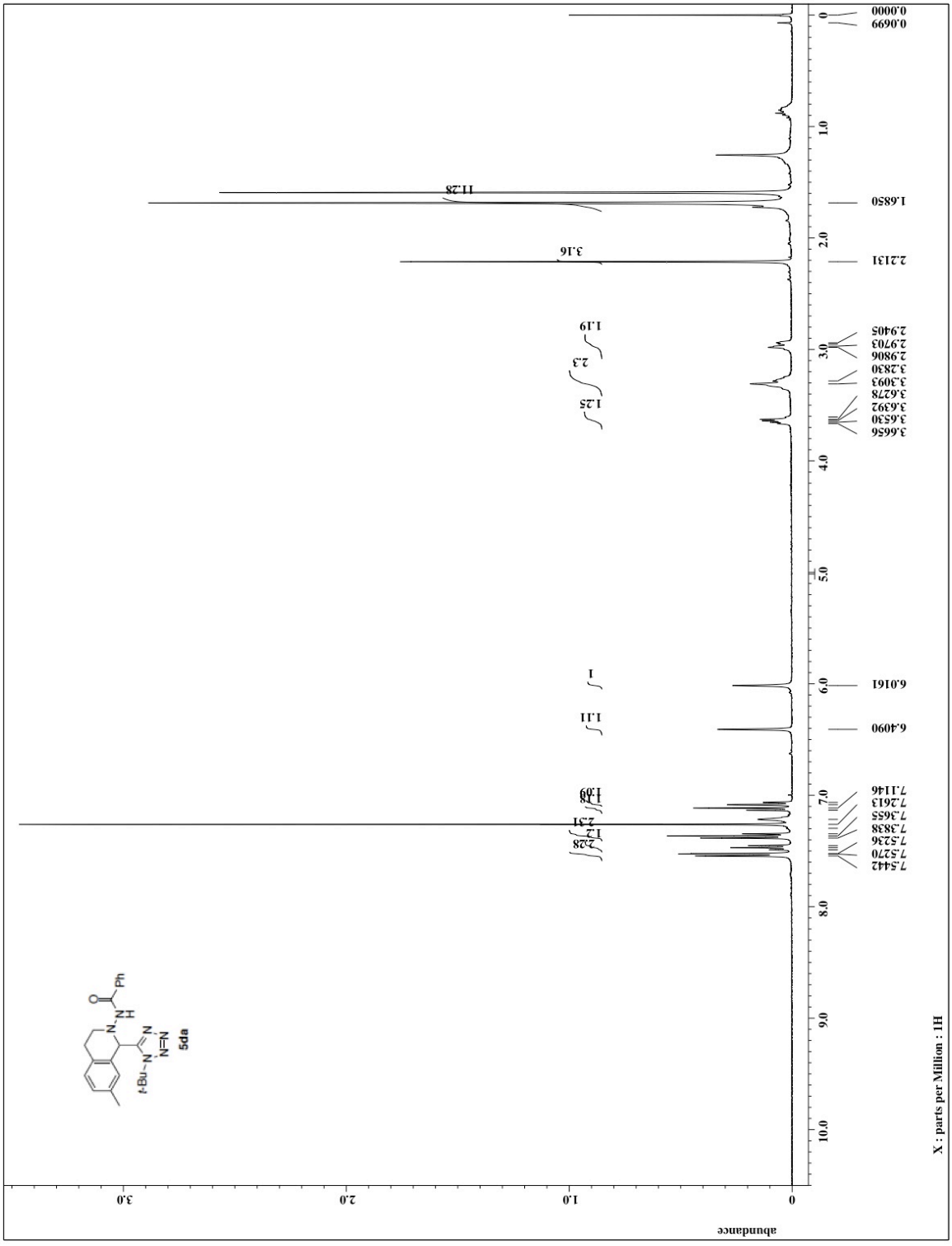

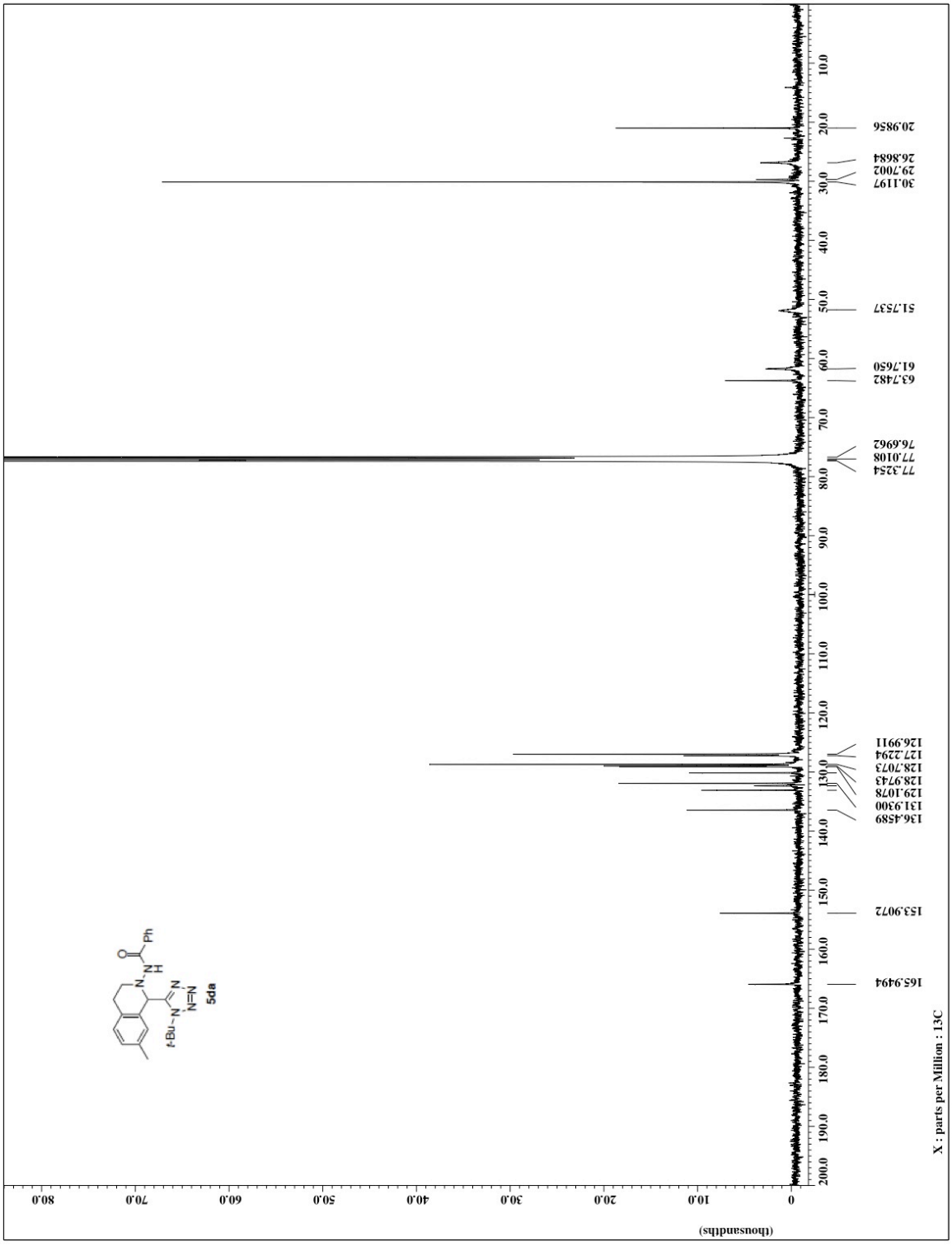
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(thousandths)
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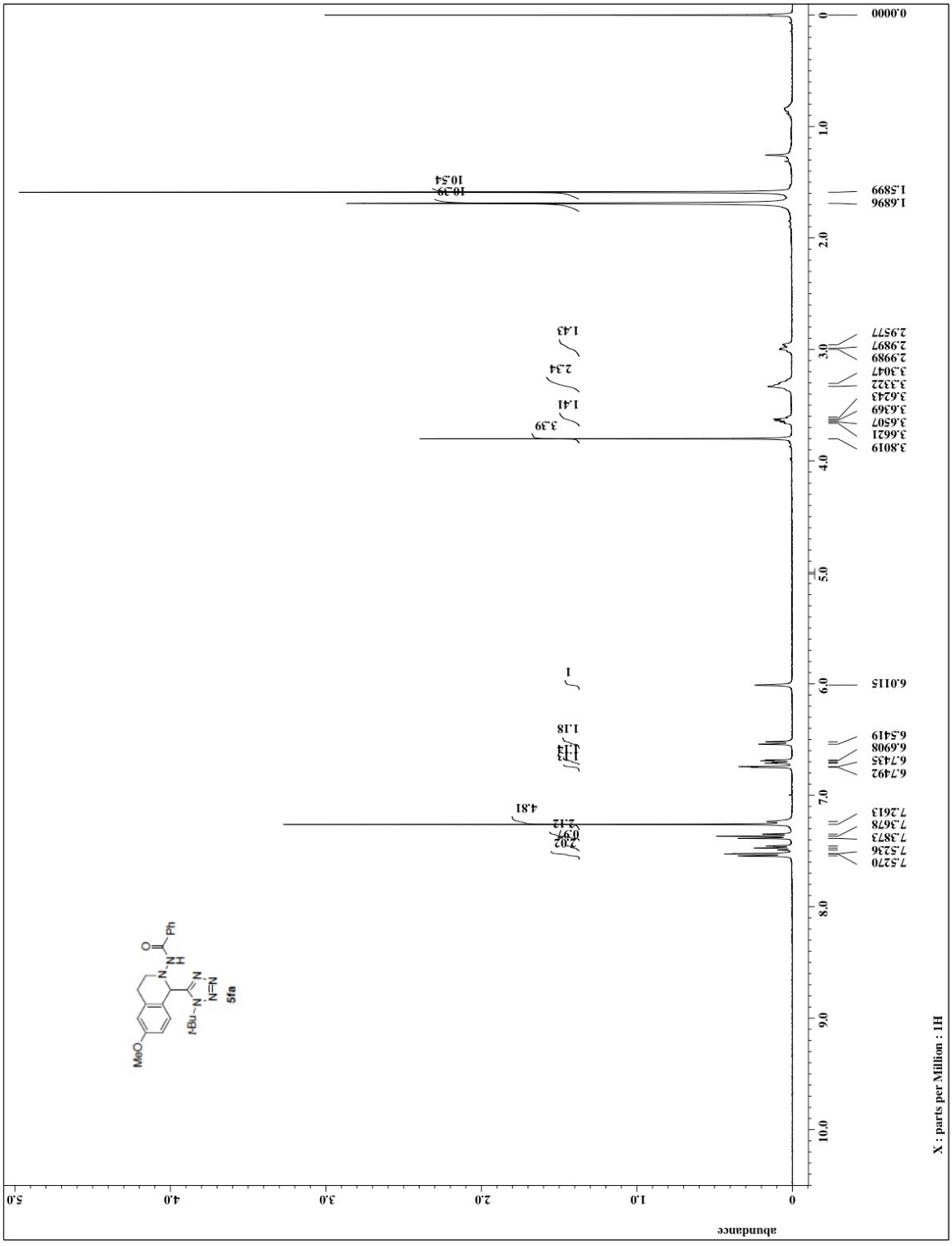

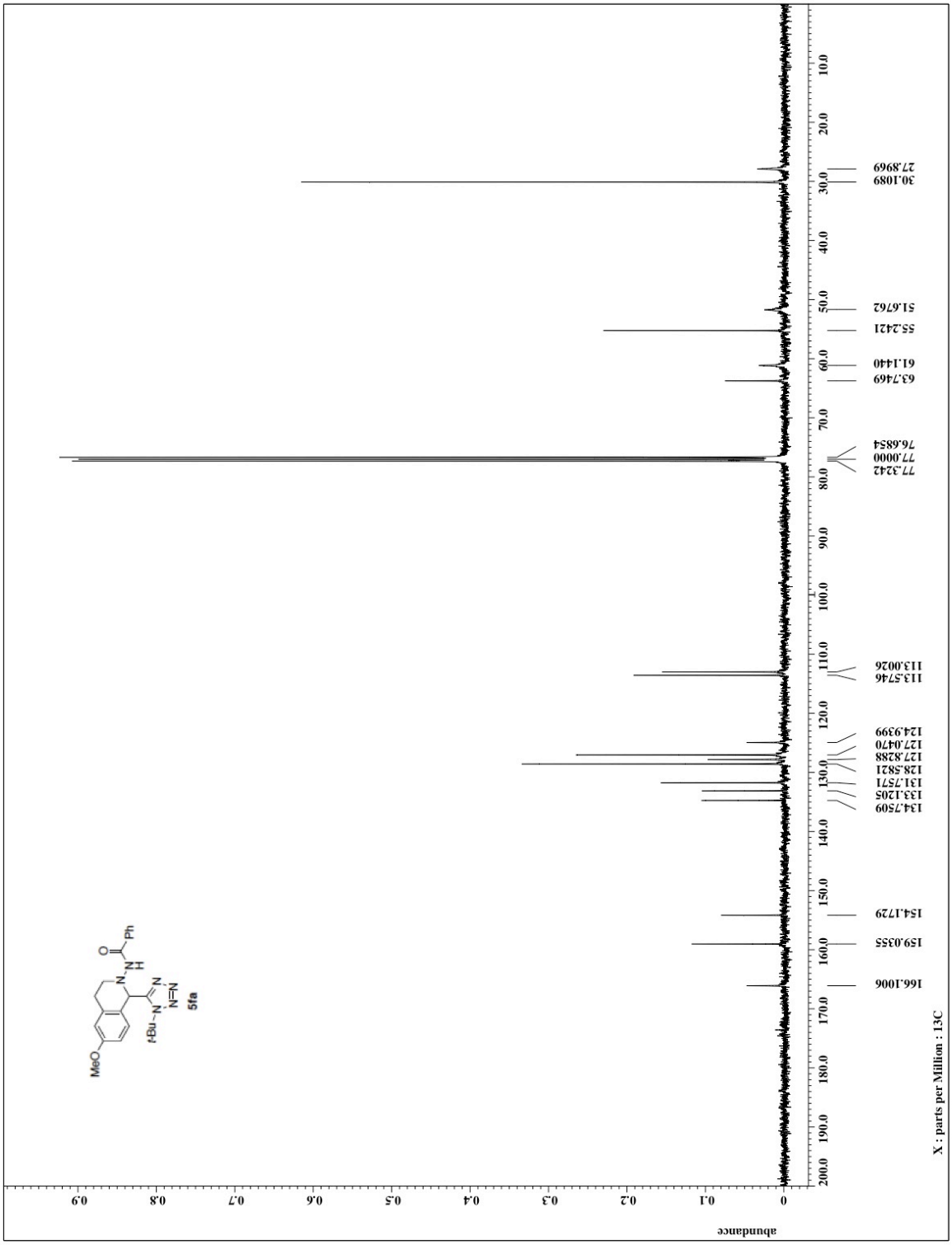
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X : parts per Million : 13C
abundance
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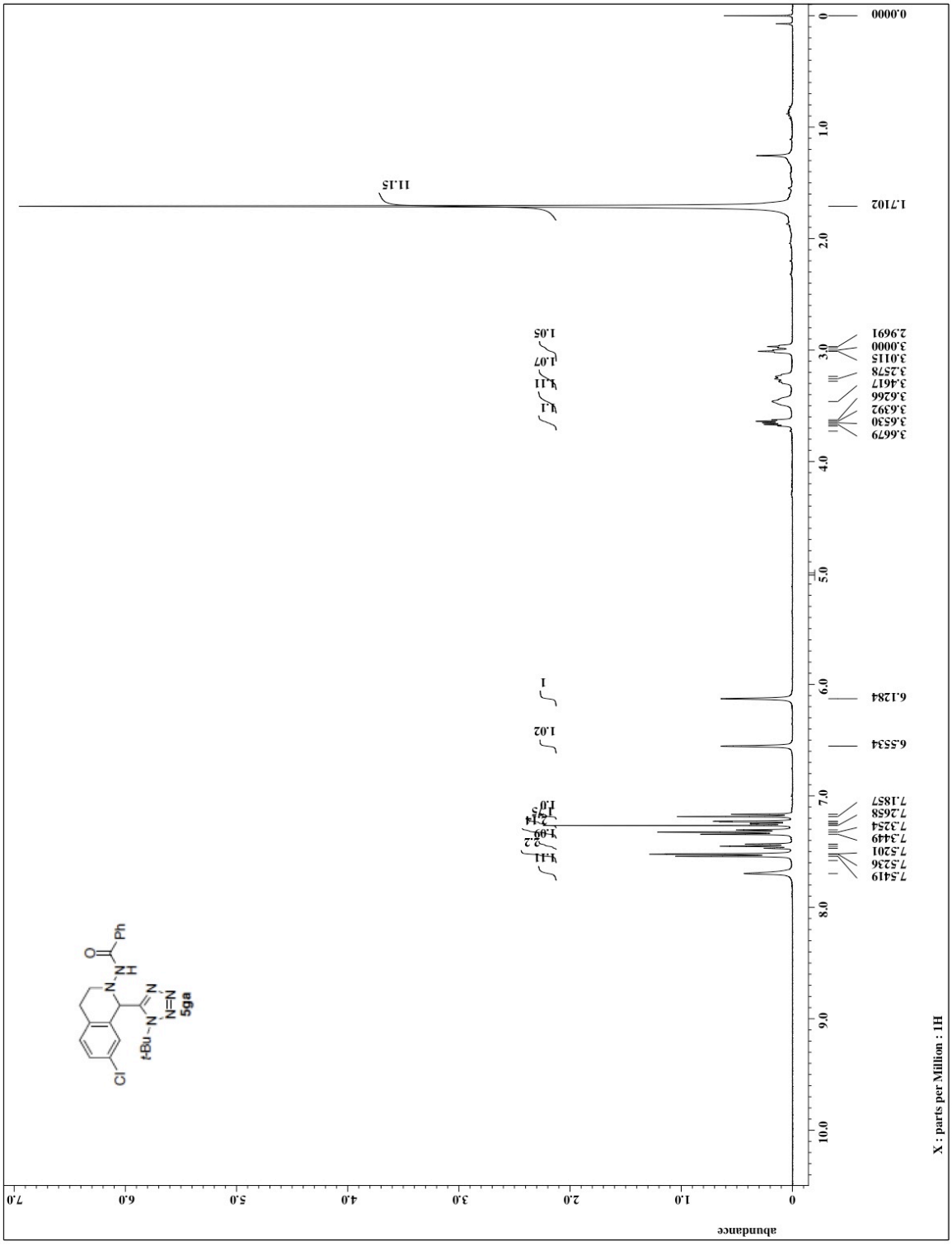
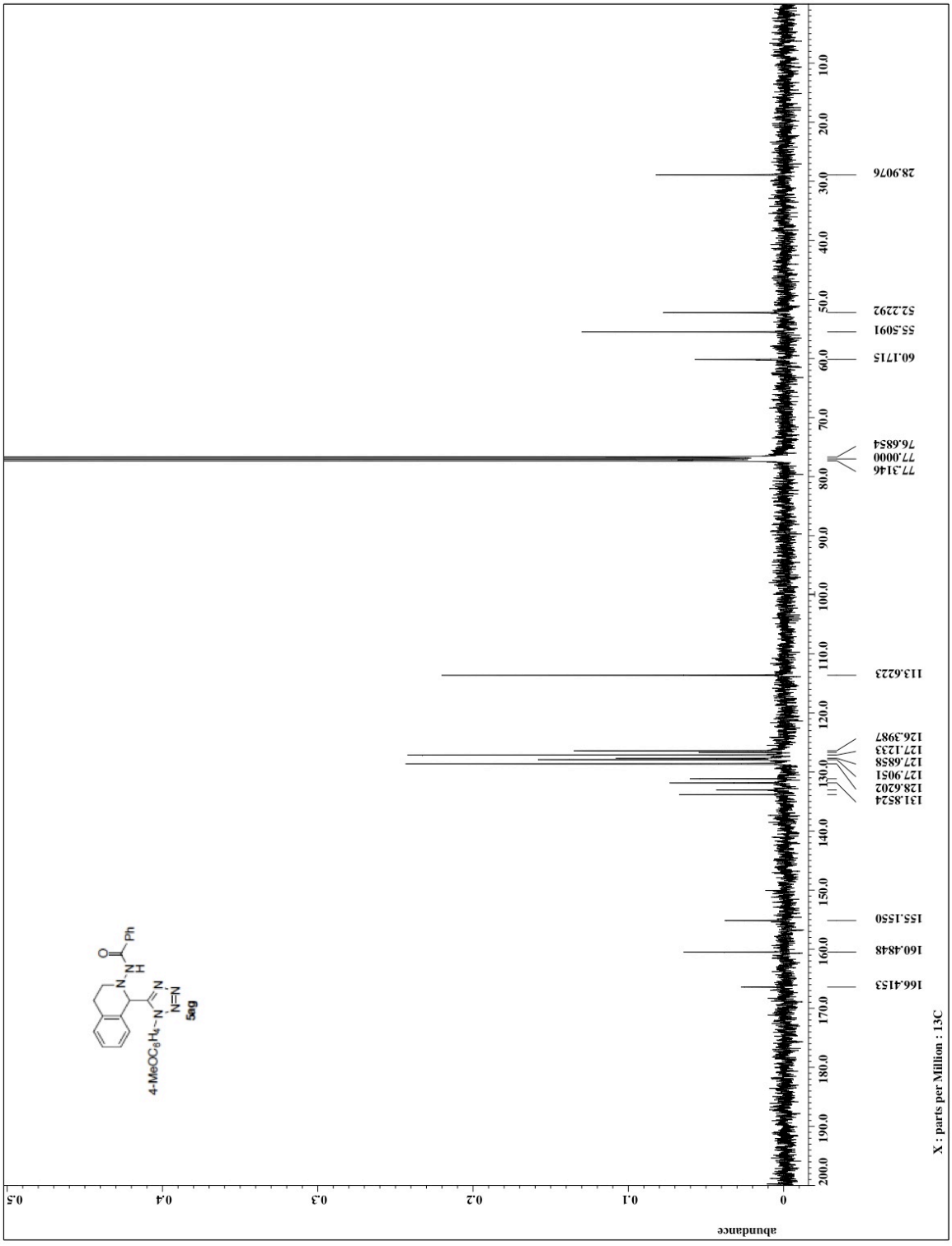
t-Bu
N=N
5fa

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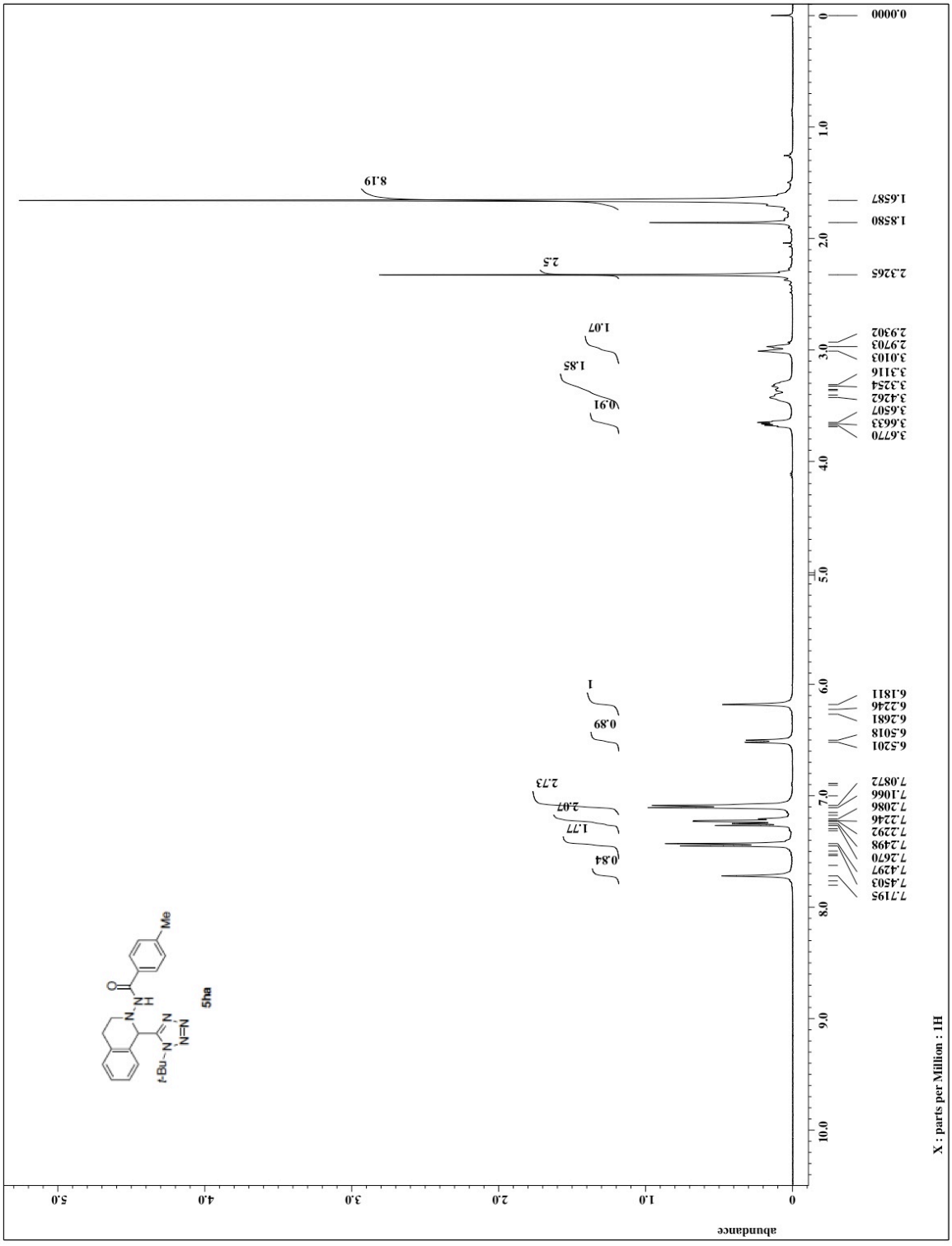

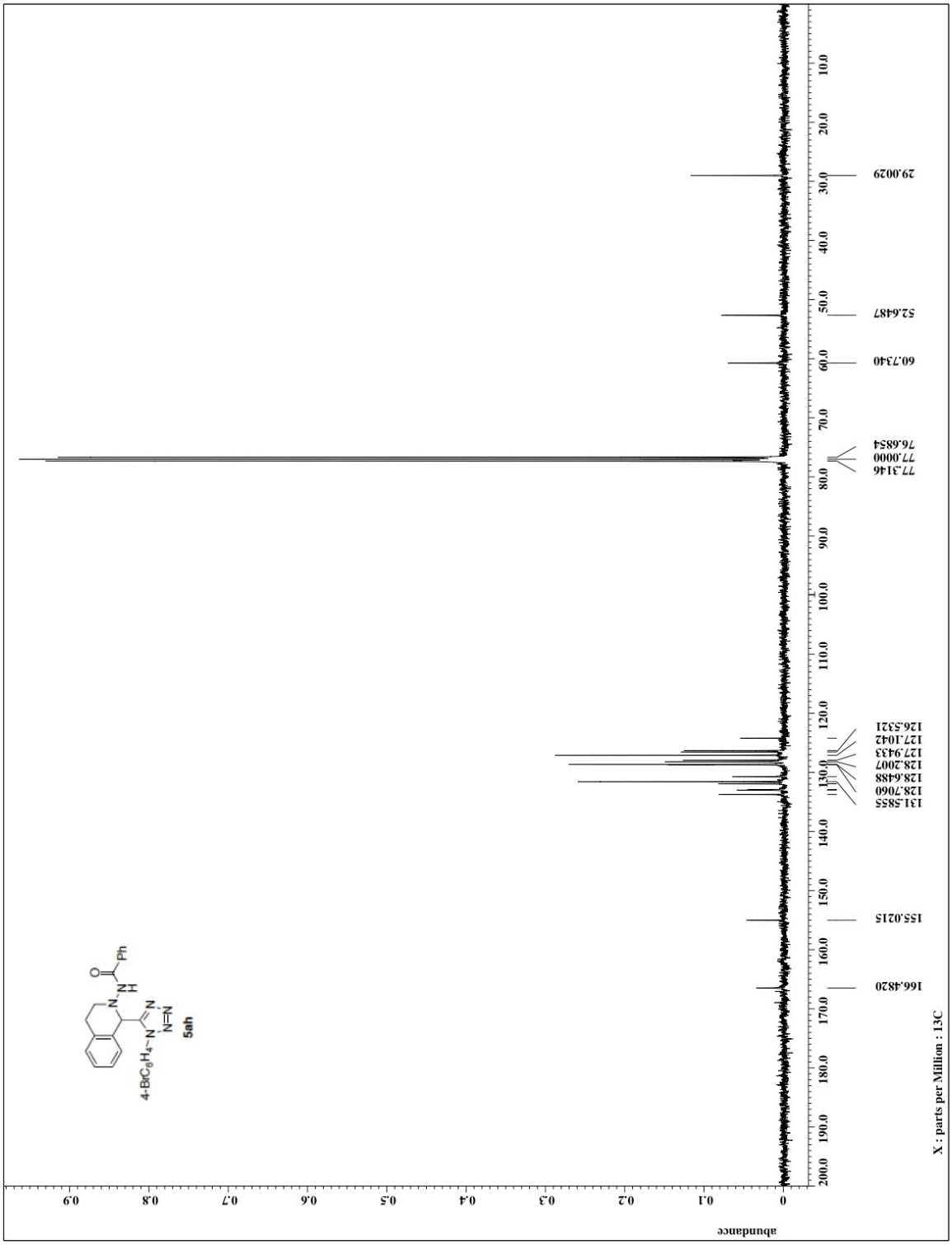
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abundance
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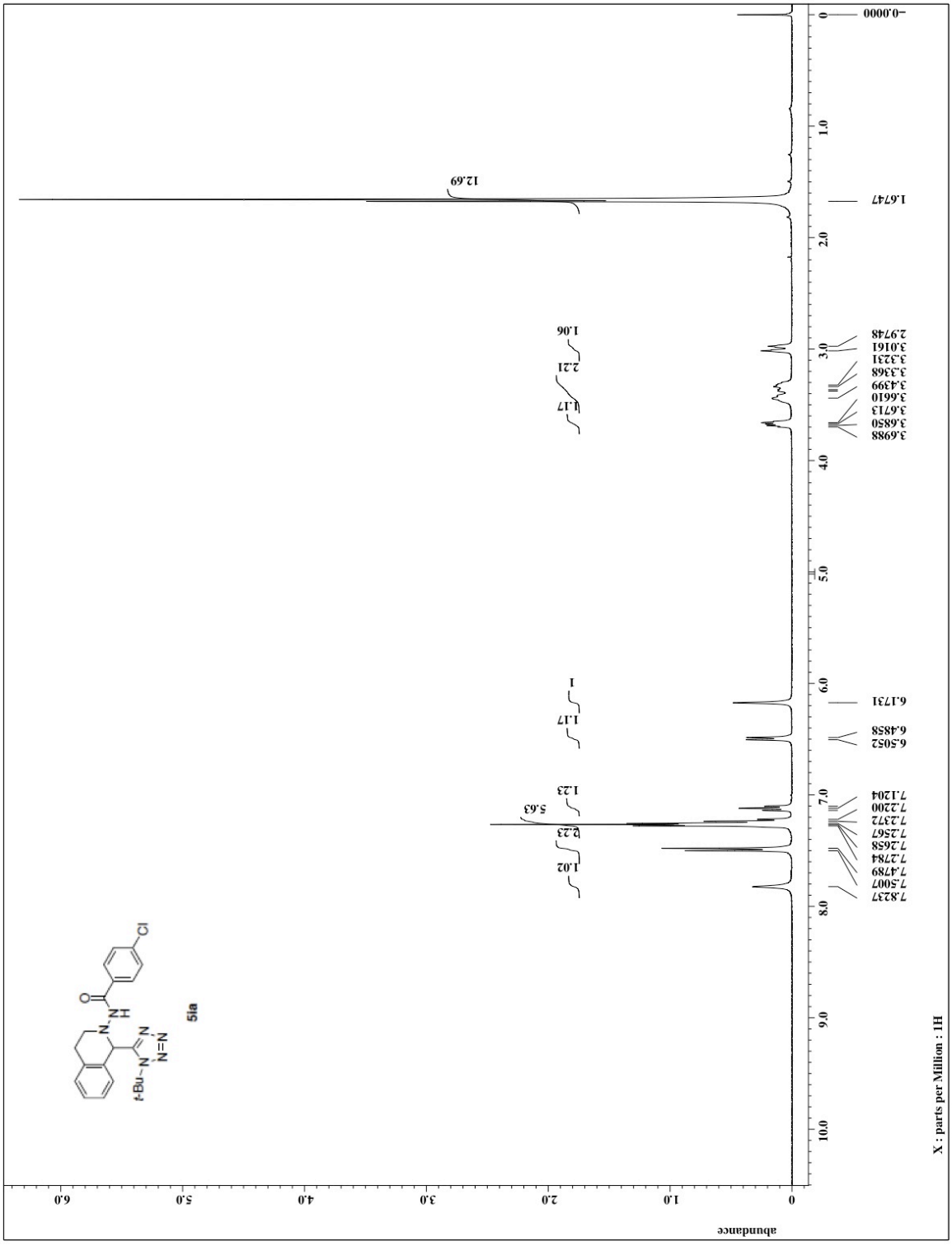
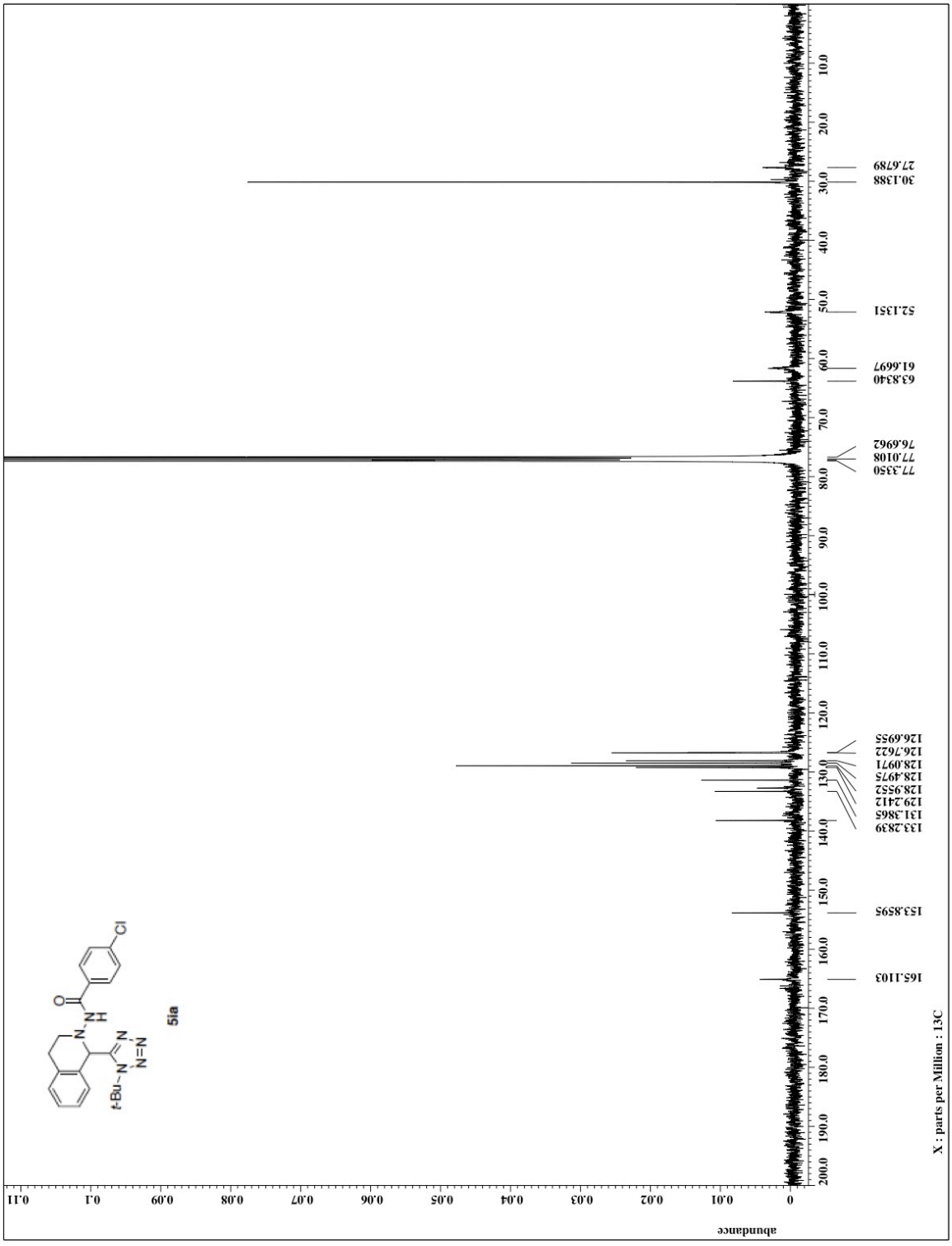
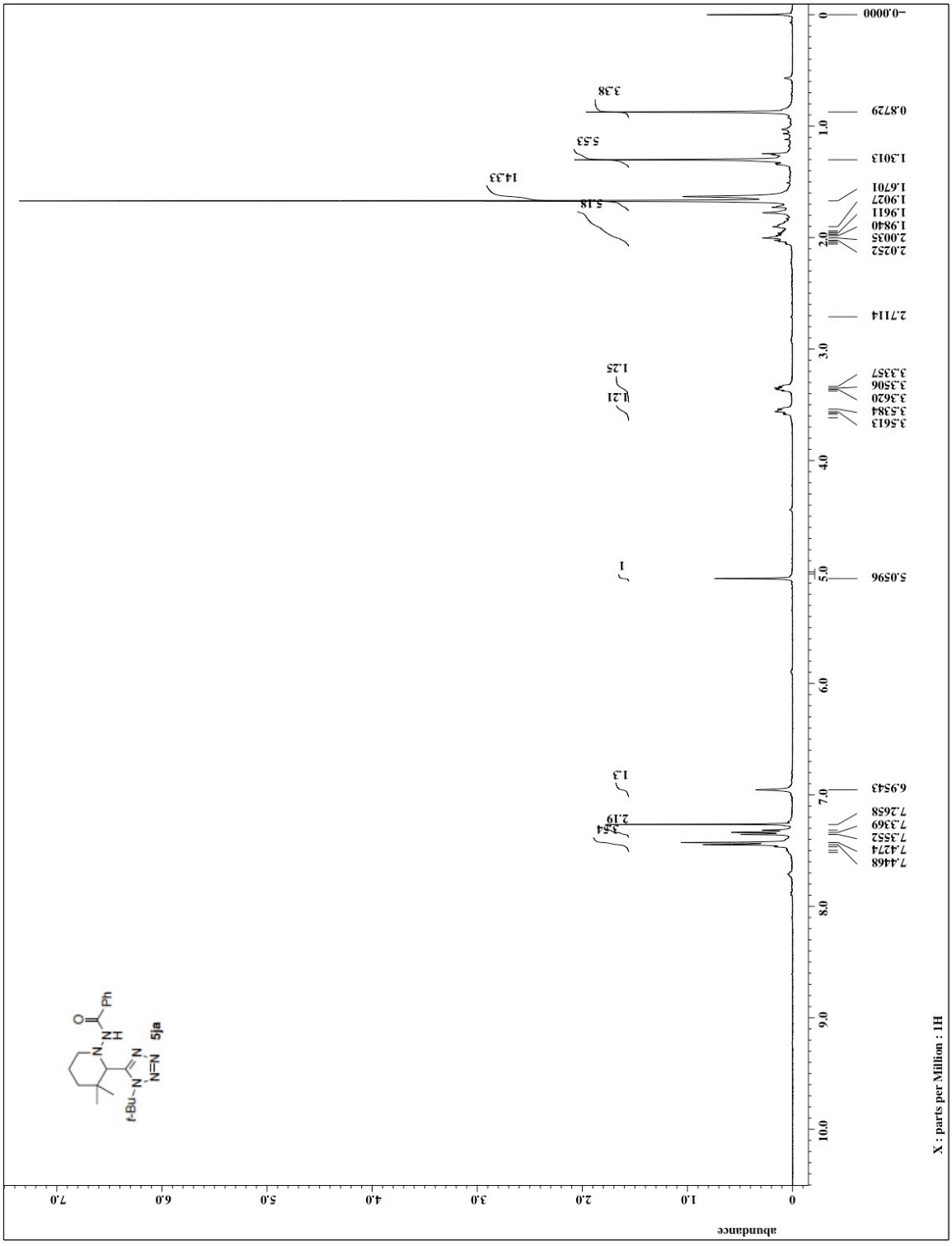
5ah



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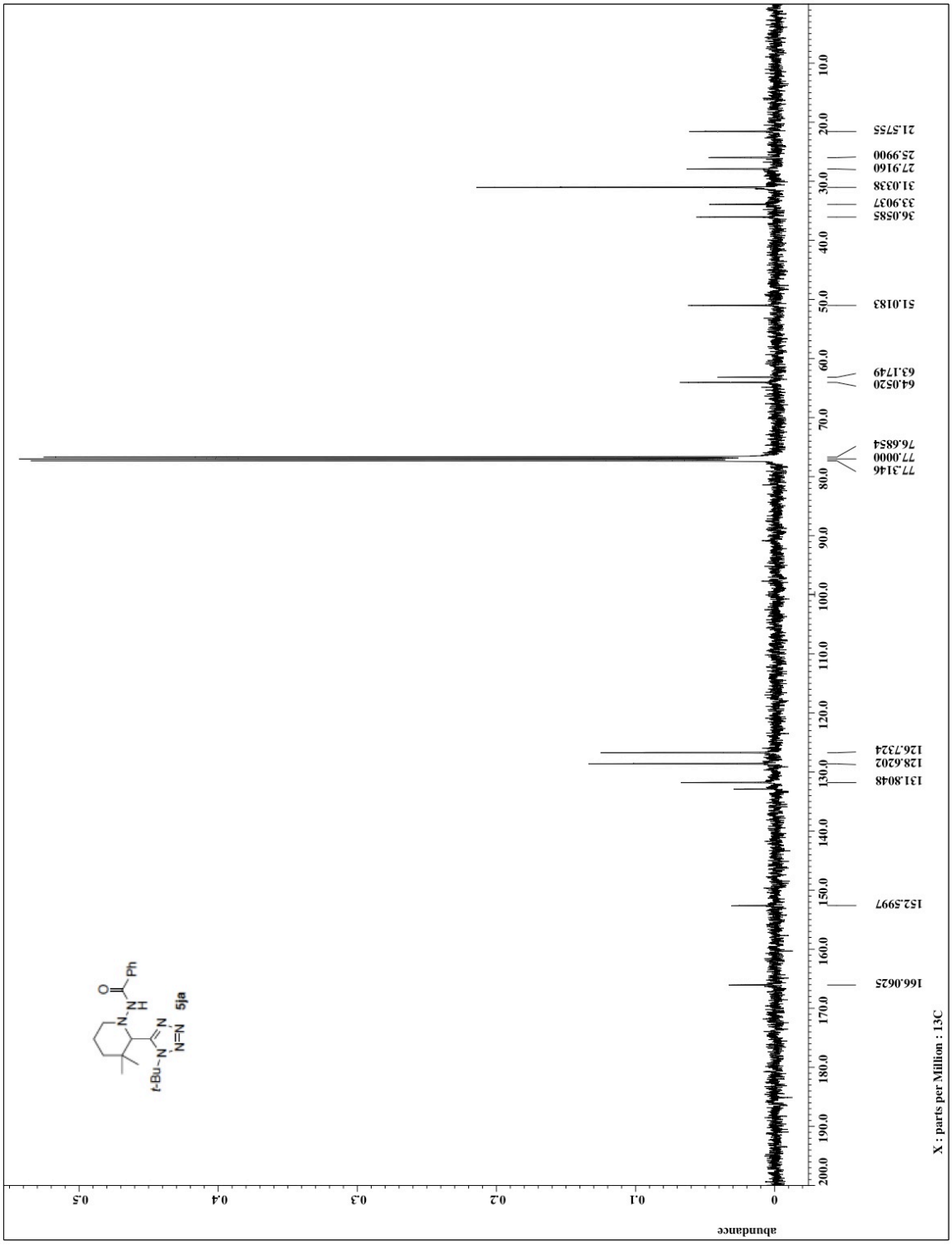

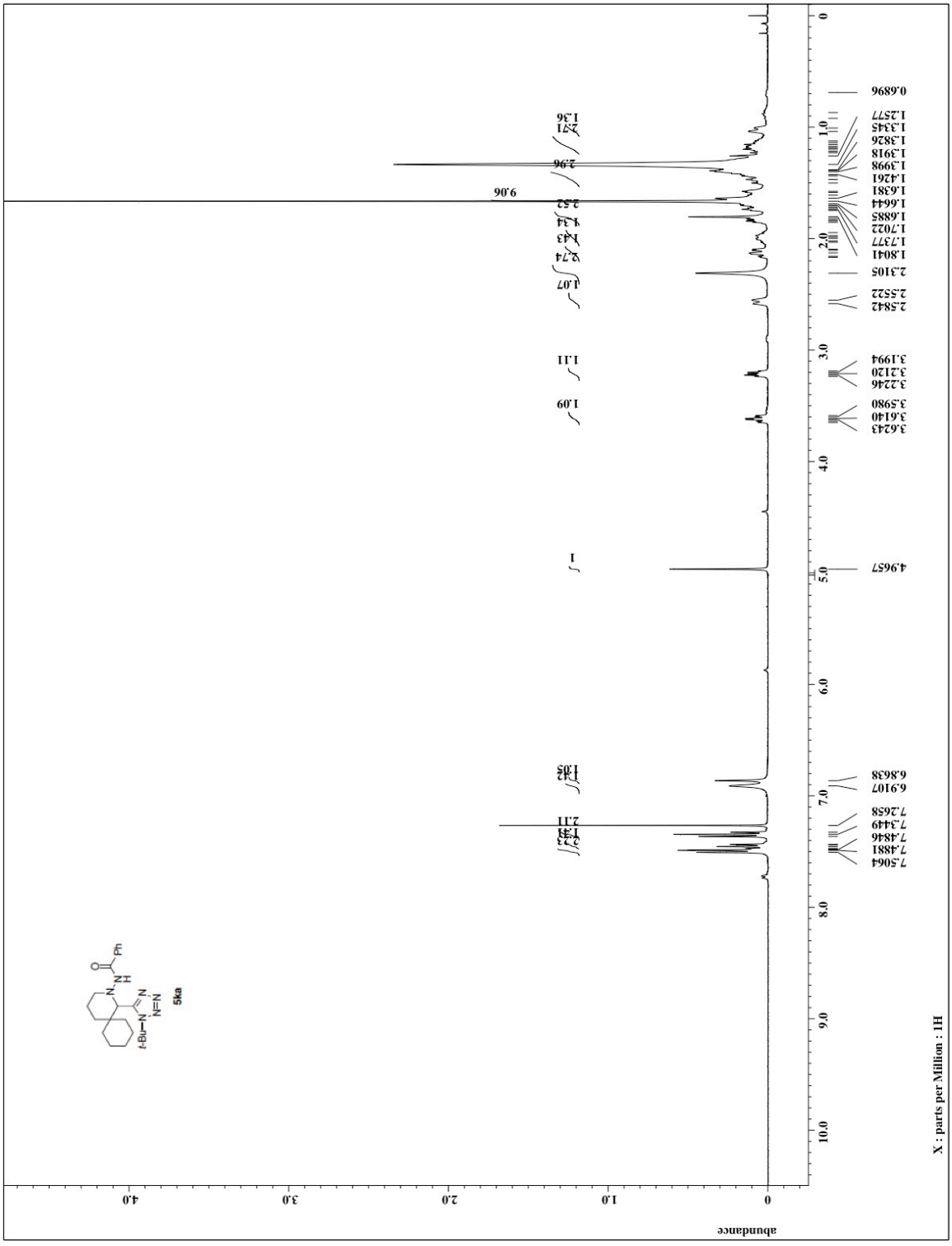

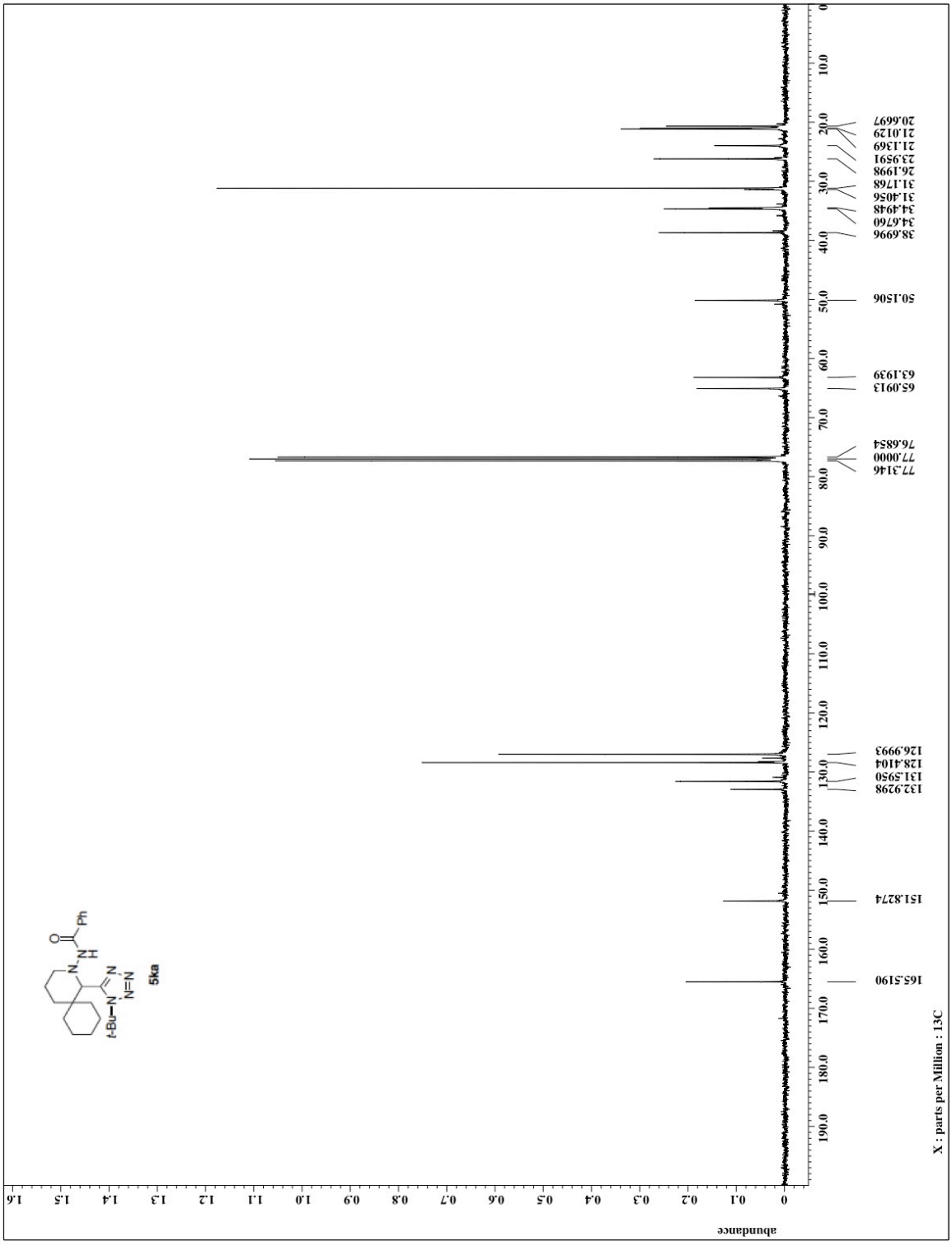

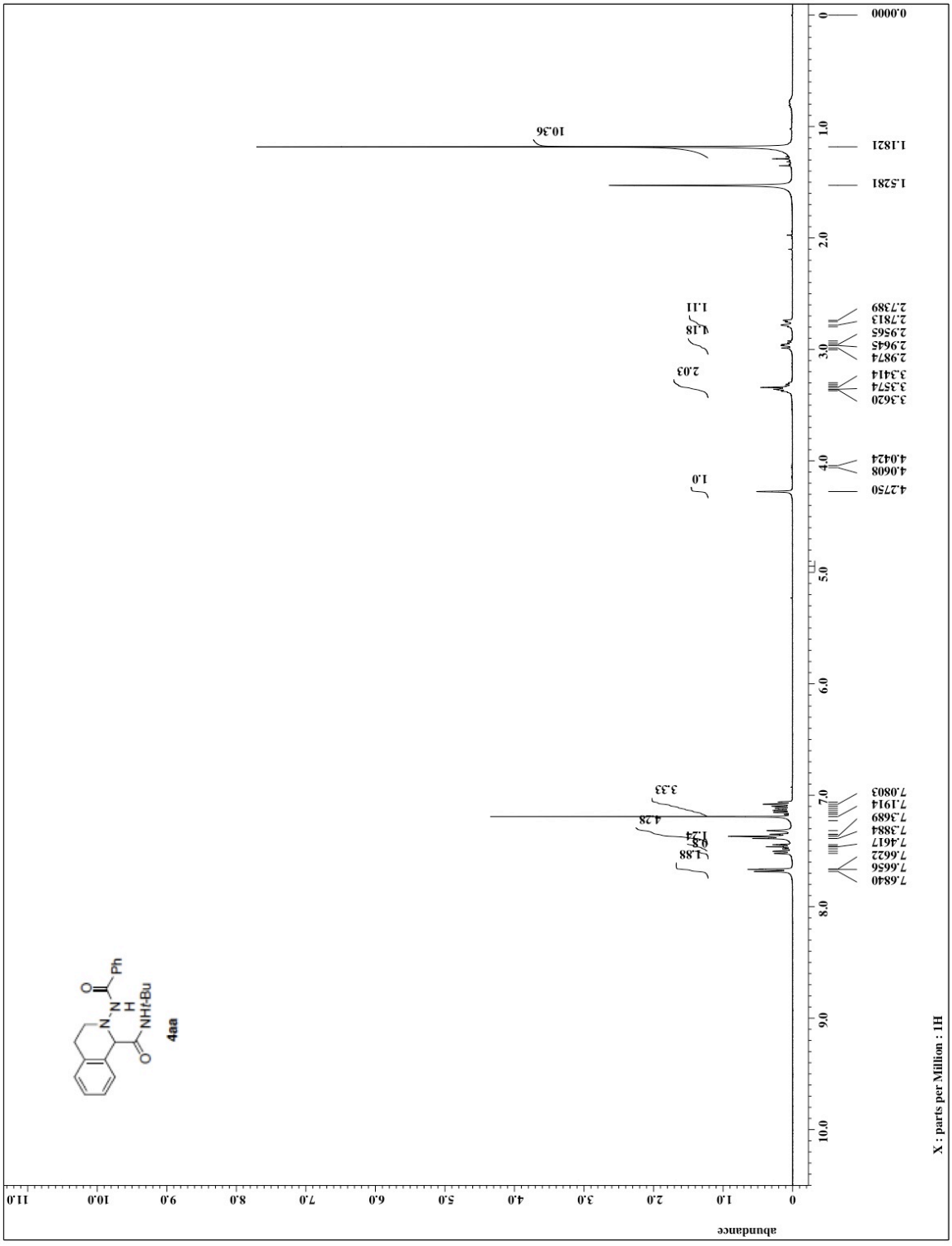

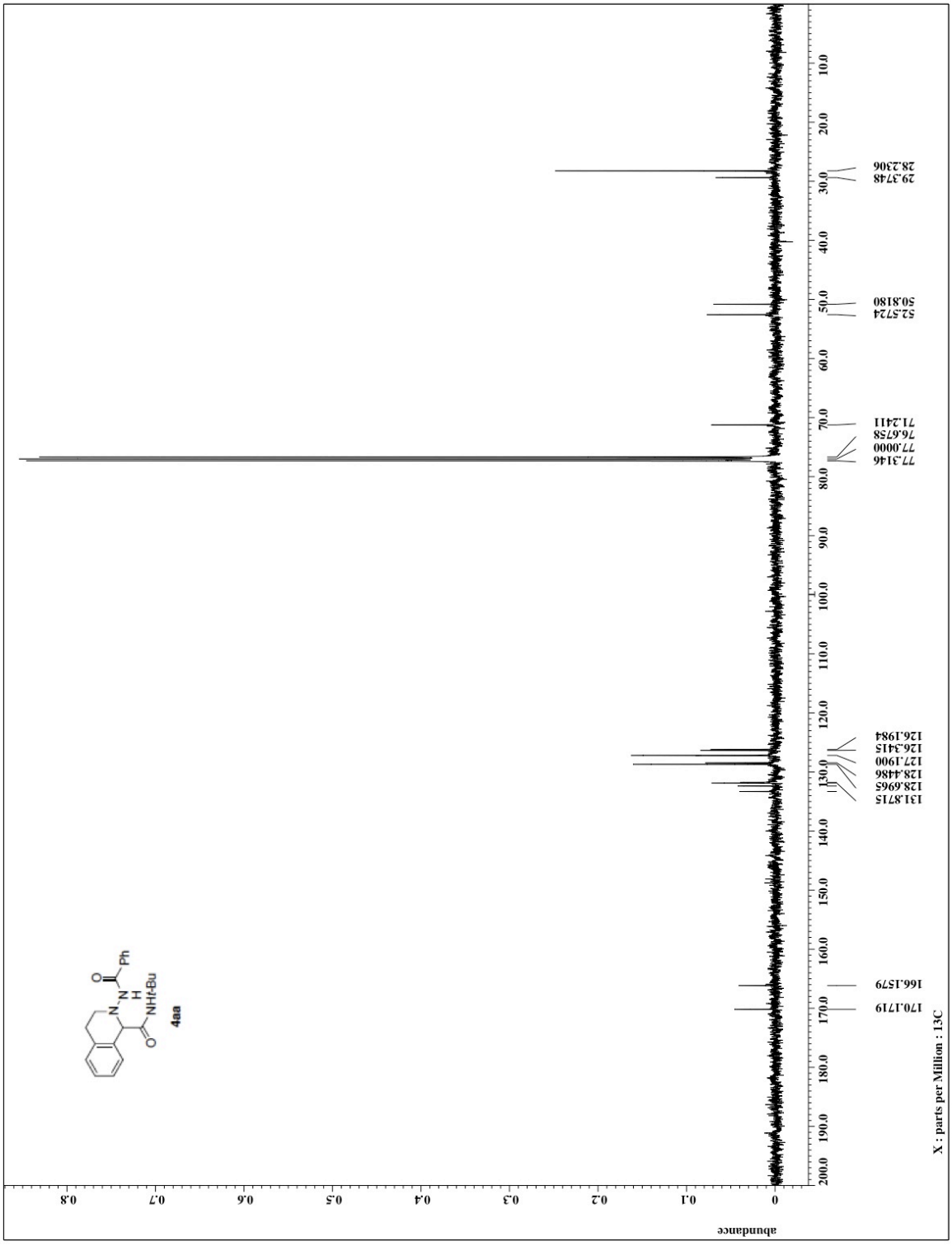

X-ray Structure Report

Fri May 13 2011

Experimental

Data Collection

A Colorless Prism crystal of $H_{24}C_{21}N_6O$ having approximate dimensions of 0.25 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku/MSC Mercury diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 10.2116(7) Å
b = 19.269(1) Å β = 113.424(1)°
c = 10.7192(7) Å
V = 1935.4(2) Å^3

For Z = 4 and F.W. = 376.46, the calculated density is 1.29 g/cm^3. The systematic absences of:

h0l: l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -150 ± 1°C to a maximum 2θ value of 55.0°.A total of 720 oscillation images were collected. A first sweep of data was done using ω

scans from -80.0 to 100.0° in 0.50° step, at χ=45.0° and ϕ=0.0°. The exposure rate was 50.0 [sec./°]. The detector swing angle was 10.1°. The crystal-to-detector distance was 34.96 mm. A second sweep of data was done using ω scans from -80.0 to 100.0° in 0.50° step, at χ=45.0° and ϕ=90.0°. The exposure rate was 50.0 [sec./°]. The detector swing angle was 10.1°. The crystal-to-detector distance was 34.96 mm.

Data Reduction

Of the 4503 reflections which were collected, 4367 were unique (R_{int} = 0.024); equivalent reflections were merged. Data were collected and processed using the CrystalClear program (Rigaku). The linear absorption coefficient, μ, for Mo-Kα radiation is 0.8 cm^{-1}. was applied which resulted in transmission factors ranging from 0.81 to 0.98. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 3362 observed reflections ($I > 3.00\sigma(I)$, $2\theta < 0.00$) and 253 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.049$$

$$R_W = [(\Sigma \, w \, (|Fo| - |Fc|)^2 \, / \, \Sigma \, w \, Fo^2)]^{1/2} = 0.074$$

$$R_W = [(S \, w \, (Fo^2 - Fc^2)^2 \, / \, S \, w \, (Fo^2)^2)]^{1/2} = 0.000 \text{ for all data}$$

The standard deviation of an observation of unit weight[4] was 1.29. The weighting scheme was based on counting statistics and included a factor (p = 0.089) to downweight the intense reflections. Plots of $\Sigma \, w \, (|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin

θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.34 and -0.17 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma\, w(|F_o|-|F_c|)^2$ where

$w = 1/[\sigma^2(Fo)] = [\sigma^2_c(Fo) + p^2Fo^2/4]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$[\Sigma\, w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_V = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$H_{24}C_{21}N_6O$
Formula Weight	376.46
Crystal Color, Habit	Colorless, Prism
Crystal Dimensions	0.25 X 0.20 X 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	7622 (6.1 - 55.0°)
Lattice Parameters	a = 10.2116(7) Å b = 19.269(1) Å c = 10.7192(7) Å β = 113.424(1)° V = 1935.4(2) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4

D_{calc}	1.292 g/cm^3
F_{000}	800.00
μ(MoKα)	0.84 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku/MSC Mercury CCD
Radiation	MoKα (λ = 0.71070 Å) graphite monochromated
Temperature	-150.0 ºC
Detector Aperture	70 mm x 70 mm
Data Images	720 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	-80.0 - 100.0º
ω oscillation Range (χ=45.0, ϕ=90.0)	-80.0 - 100.0º
Exposure Rate	50.0 sec./º
Detector Swing Angle	10.07º
Detector Position	34.96 mm

$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 4503 Unique: 4367 (R_{int} = 0.024)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.8063 - 0.9833)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (|Fo| - |Fc|)^2
Least Squares Weights	$1/\sigma^2(Fo) = 4Fo^2/\sigma^2(Fo^2)$
p-factor	0.0890
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I>3.00\sigma(I)$, $2\theta < 0.00^{o}$)	3362
No. Variables	253

Reflection/Parameter Ratio	13.29
Residuals: R; Rw	0.049 ; 0.074
Goodness of Fit Indicator	1.29
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.34 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.17 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	1.1059(2)	0.07405(7)	0.1174(1)	2.29(3)
N(1)	0.8910(2)	0.05913(8)	0.2033(1)	1.55(3)
N(2)	1.0168(2)	0.01856(8)	0.2539(1)	1.65(3)
N(3)	1.0255(2)	0.08477(8)	0.5526(1)	1.58(3)
N(4)	1.1402(2)	0.12239(8)	0.6327(1)	1.72(3)
N(5)	1.1632(2)	0.17243(8)	0.5634(1)	1.68(3)
N(6)	1.0619(1)	0.16882(7)	0.4351(1)	1.41(3)
C(1)	0.8536(2)	0.08068(9)	0.3164(2)	1.45(3)
C(2)	0.7191(2)	0.12449(9)	0.2646(2)	1.51(3)
C(3)	0.6889(2)	0.1677(1)	0.3542(2)	1.93(4)
C(4)	0.5656(2)	0.2073(1)	0.3096(2)	2.19(4)
C(5)	0.4707(2)	0.2037(1)	0.1749(2)	2.20(4)
C(6)	0.4988(2)	0.1601(1)	0.0860(2)	2.04(4)
C(7)	0.6233(2)	0.11996(9)	0.1291(2)	1.70(3)
C(8)	0.6545(2)	0.0742(1)	0.0295(2)	2.12(4)
C(9)	0.7706(2)	0.02137(10)	0.1014(2)	1.91(4)
C(10)	0.9783(2)	0.11349(9)	0.4298(2)	1.38(3)
C(11)	1.0717(2)	0.22559(9)	0.3408(2)	1.50(3)
C(12)	1.2140(2)	0.21462(10)	0.3272(2)	1.86(3)
C(13)	0.9477(2)	0.22110(10)	0.2025(2)	1.88(3)
C(14)	1.0684(2)	0.29487(10)	0.4088(2)	2.03(4)
C(15)	1.1173(2)	0.02986(9)	0.2025(2)	1.63(3)
C(16)	1.2470(2)	-0.01536(9)	0.2612(2)	1.62(3)
C(17)	1.3788(2)	0.0138(1)	0.2810(2)	2.19(4)
C(18)	1.5013(2)	-0.0260(1)	0.3332(2)	2.65(4)
C(19)	1.4933(2)	-0.0956(1)	0.3626(2)	2.74(4)
C(20)	1.3632(2)	-0.1250(1)	0.3424(2)	2.53(4)
C(21)	1.2399(2)	-0.08483(10)	0.2928(2)	1.99(4)
H(1)	0.8394	0.0366	0.3673	7.2

H(2)	0.7600	0.1705	0.4524	7.2
H(3)	0.5517	0.2374	0.3817	7.2
H(4)	0.3874	0.2329	0.1434	7.2
H(5)	0.4322	0.1546	-0.0090	7.2
H(6)	0.6893	0.1031	-0.0282	7.2
H(7)	0.5675	0.0513	-0.0321	7.2
H(8)	0.8078	0.0050	0.0350	7.2
H(9)	0.7393	-0.0166	0.1529	7.2
H(10)	1.2166	0.1682	0.2786	7.2
H(11)	1.2301	0.2546	0.2676	7.2
H(12)	1.2901	0.2160	0.4136	7.2
H(13)	0.9685	0.2651	0.1443	7.2
H(14)	0.8556	0.2340	0.2201	7.2
H(15)	0.9428	0.1782	0.1543	7.2
H(16)	0.9681	0.3016	0.4159	7.2
H(17)	1.0693	0.3318	0.3438	7.2
H(18)	1.1466	0.2991	0.5032	7.2
H(19)	1.0242	-0.0192	0.3163	7.2
H(20)	1.3814	0.0661	0.2561	7.2
H(21)	1.5901	0.0041	0.3491	7.2
H(22)	1.5837	-0.1184	0.3973	7.2
H(23)	1.3545	-0.1708	0.3730	7.2
H(24)	1.1504	-0.1059	0.2808	7.2

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0342(7)	0.0273(7)	0.0331(7)	0.0077(6)	0.0213(6)	0.0087(6)
N(1)	0.0168(7)	0.0230(7)	0.0186(7)	0.0044(6)	0.0065(6)	-0.0004(6)
N(2)	0.0201(7)	0.0215(8)	0.0233(7)	0.0050(6)	0.0107(6)	0.0040(6)
N(3)	0.0191(7)	0.0221(7)	0.0184(7)	0.0022(6)	0.0070(6)	0.0021(6)
N(4)	0.0215(7)	0.0233(8)	0.0197(7)	0.0017(6)	0.0073(6)	0.0014(6)
N(5)	0.0205(7)	0.0239(8)	0.0174(7)	0.0018(6)	0.0055(6)	0.0022(6)
N(6)	0.0153(7)	0.0207(7)	0.0169(7)	0.0013(5)	0.0056(6)	0.0012(5)
C(1)	0.0168(8)	0.0205(8)	0.0182(8)	0.0006(6)	0.0074(7)	0.0006(6)
C(2)	0.0156(8)	0.0207(8)	0.0223(8)	-0.0007(6)	0.0088(7)	0.0022(6)
C(3)	0.0196(9)	0.0290(10)	0.0244(9)	0.0019(7)	0.0083(7)	-0.0007(7)
C(4)	0.0247(9)	0.031(1)	0.0303(10)	0.0047(8)	0.0139(8)	-0.0003(8)
C(5)	0.0190(9)	0.032(1)	0.033(1)	0.0056(7)	0.0109(8)	0.0074(8)
C(6)	0.0191(9)	0.0312(10)	0.0259(9)	0.0006(7)	0.0075(7)	0.0053(8)
C(7)	0.0181(8)	0.0243(9)	0.0217(8)	-0.0012(6)	0.0076(7)	0.0025(7)
C(8)	0.0231(9)	0.034(1)	0.0202(9)	0.0045(7)	0.0053(7)	-0.0012(7)
C(9)	0.0246(9)	0.0254(9)	0.0204(8)	0.0013(7)	0.0065(7)	-0.0037(7)
C(10)	0.0163(8)	0.0188(8)	0.0190(8)	0.0031(6)	0.0089(7)	0.0014(6)
C(11)	0.0203(8)	0.0193(8)	0.0188(8)	0.0000(6)	0.0091(7)	0.0043(6)
C(12)	0.0204(9)	0.0277(9)	0.0240(9)	0.0019(7)	0.0104(7)	0.0029(7)
C(13)	0.0221(9)	0.0265(9)	0.0202(8)	-0.0003(7)	0.0057(7)	0.0040(7)
C(14)	0.0306(10)	0.0209(9)	0.0251(9)	0.0029(7)	0.0108(8)	0.0016(7)
C(15)	0.0233(9)	0.0200(8)	0.0208(8)	0.0010(7)	0.0111(7)	-0.0016(6)
C(16)	0.0208(9)	0.0240(9)	0.0185(8)	0.0014(7)	0.0098(7)	-0.0020(7)
C(17)	0.0242(9)	0.030(1)	0.0298(10)	-0.0033(8)	0.0121(8)	-0.0011(8)
C(18)	0.0218(9)	0.044(1)	0.035(1)	-0.0020(8)	0.0115(8)	0.0019(9)
C(19)	0.0246(10)	0.047(1)	0.034(1)	0.0123(9)	0.0126(9)	0.0104(9)
C(20)	0.033(1)	0.030(1)	0.040(1)	0.0080(8)	0.0212(9)	0.0102(8)
C(21)	0.0225(9)	0.0231(9)	0.0333(10)	0.0011(7)	0.0145(8)	0.0007(8)

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O1	C15	1.220(2)	N1	N2	1.415(2)
N1	C1	1.469(2)	N1	C9	1.472(2)
N2	C15	1.361(2)	N3	N4	1.355(2)
N3	C10	1.329(2)	N4	N5	1.294(2)
N5	N6	1.355(2)	N6	C10	1.352(2)
N6	C11	1.520(2)	C1	C2	1.517(2)
C1	C10	1.506(2)	C2	C3	1.394(2)
C2	C7	1.395(2)	C3	C4	1.385(3)
C4	C5	1.385(3)	C5	C6	1.385(3)
C6	C7	1.400(2)	C7	C8	1.513(3)
C8	C9	1.519(3)	C11	C12	1.532(2)
C11	C13	1.522(2)	C11	C14	1.528(2)
C15	C16	1.499(2)	C16	C17	1.395(3)
C16	C21	1.390(3)	C17	C18	1.383(3)
C18	C19	1.386(3)	C19	C20	1.380(3)
C20	C21	1.391(3)			

Table 4. Bond Angles(º)

atom	atom	atom	angle	atom	atom	atom	angle
N2	N1	C1	109.5(1)	N2	N1	C9	112.0(1)
C1	N1	C9	109.7(1)	N1	N2	C15	118.2(1)
N4	N3	C10	106.6(1)	N3	N4	N5	110.1(1)
N4	N5	N6	107.7(1)	N5	N6	C10	107.3(1)
N5	N6	C11	114.4(1)	C10	N6	C11	138.3(1)
N1	C1	C2	110.5(1)	N1	C1	C10	111.8(1)
C2	C1	C10	114.5(1)	C1	C2	C3	119.7(1)
C1	C2	C7	120.3(1)	C3	C2	C7	119.9(2)
C2	C3	C4	120.7(2)	C3	C4	C5	119.7(2)
C4	C5	C6	119.8(2)	C5	C6	C7	121.2(2)
C2	C7	C6	118.6(2)	C2	C7	C8	120.9(2)
C6	C7	C8	120.5(2)	C7	C8	C9	111.9(1)
N1	C9	C8	107.3(1)	N3	C10	N6	108.2(1)
N3	C10	C1	118.8(1)	N6	C10	C1	133.0(1)
N6	C11	C12	106.3(1)	N6	C11	C13	111.0(1)
N6	C11	C14	107.0(1)	C12	C11	C13	110.6(1)
C12	C11	C14	111.6(1)	C13	C11	C14	110.3(1)
O1	C15	N2	123.4(2)	O1	C15	C16	121.7(1)
N2	C15	C16	114.8(1)	C15	C16	C17	117.9(2)
C15	C16	C21	122.6(2)	C17	C16	C21	119.5(2)
C16	C17	C18	120.0(2)	C17	C18	C19	120.2(2)
C18	C19	C20	120.1(2)	C19	C20	C21	120.0(2)
C16	C21	C20	120.1(2)				

Table 5. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O1	C14[1)]	3.293(2)	O1	C9[2)]	3.581(2)
N2	N3[3)]	3.028(2)	N3	N3[3)]	3.427(3)
N3	C1[3)]	3.505(2)	N5	C12[4)]	3.440(2)
N5	C19[5)]	3.594(2)	C3	C20[3)]	3.594(3)

Symmetry operations

(1)	X,-Y+1/2,Z-1/2	(2)	-X+2,-Y,-Z
(3)	-X+2,-Y,-Z+1	(4)	X,-Y+1/2,Z+1/2
(5)	-X+3,-Y,-Z+1		