

Supplementary Material

Synthesis of bis-oxathiaaza[3.3.3]propellanes via nucleophilic addition of (1, ω -alkanediyl)bis(*N'*-organylthioureas) on dicyanomethylene-1,3-indanedione

Alaa A. Hassan,^{a*} Kamal M. A. El-Shaieb,^a Amal S. Abd El-Aal,^a Stefan Bräse,^b and Martin Nieger^c

^a Chemistry Department, Faculty of Science, Minia University, 61519 El-Minia, Egypt.

^b Institute of Organic Chemistry, Karlsruhe Institute of Technology, Fritz-Haber-Weg 6, 76131 Karlsruhe, Germany.

^c Laboratory of Inorganic Chemistry, Department of Chemistry, University of Helsinki P.O Box 55 (A. I. Virtasen aukio 1), 00014 Helsinki, Finland
E-mail: alaahassan2001@mu.edu.eg

Single crystal X-ray diffraction study was carried out on an Agilent Super Nova diffractometer at 173 K with EOS-detector and MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). Direct Methods (SHELXS-97 [Sheldrick, G.M., *Acta Crystallogr.*, **2008**, A64, 112-122.]) were used for structure solution and refinement was carried out using SHELXL-2013 [Sheldrick, G.M., *Acta Crystallogr.*, **2008**, A64, 112-122.] (full-matrix least-squares on F^2). Hydrogen atoms were localized by difference Fourier Synthesis map and refined using a riding model [H(N) free]. A semi-empirical absorption correction and an extinction correction were applied.

Compound **7a**: C₄₀H₂₆N₈O₄S₂ · 2 (C₃H₇NO), Mr = 893.00 g mol⁻¹, colorless plates, crystal size 0.30 × 0.02 × 0.10 mm, triclinic, P-1 (no. 2), a = 9.7515 (5) Å, b = 9.8024 (8) Å, c = 13.4038 (8) Å, $\alpha = 90.279 (6)^\circ$, $\beta = 102.516 (5)^\circ$, $\gamma = 118.268 (7)^\circ$, V = 1092.98(14) Å³, Z = 1, D_{calcd} = 1.357 Mg m⁻³, F(000) = 466, $\mu = 0.184 \text{ mm}^{-1}$, T = 173 K, 7638 measured reflections ($2\theta_{\text{max}} = 55^\circ$), 4954 independent reflections ($R_{\text{int}} = 0.016$) 298 parameters, 2 restraints, R1 (for 3998 I > 2 σ (I)) = 0.044, wR² (for all data) = 0.107, S = 1.03, largest diff. peak and hole = 0.35 eÅ⁻³, -0.300 eÅ⁻³.

Supplementary data

Crystallographic data for the structure reported in this work have been deposited with Cambridge Crystallographic Data Center on supplementary publication no CCDC-1417181 Copies of the data

can be obtained free of charge on publication to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44(1223)336033; e-mail: deposit@ccdc.cam.ac.uk).

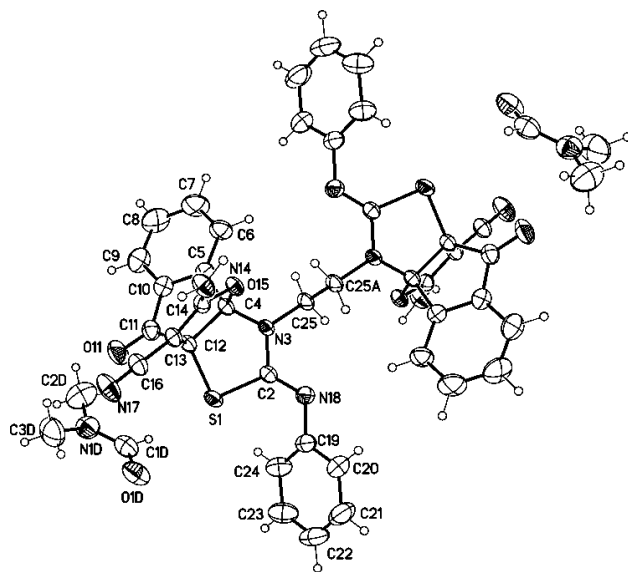


Figure 1. Molecular structure of **7a** in the crystal (displacement parameters are drawn at 50% probability level). The crystallographic numbering does not reflect the systematic IUPAC numbering (SB651_HY).

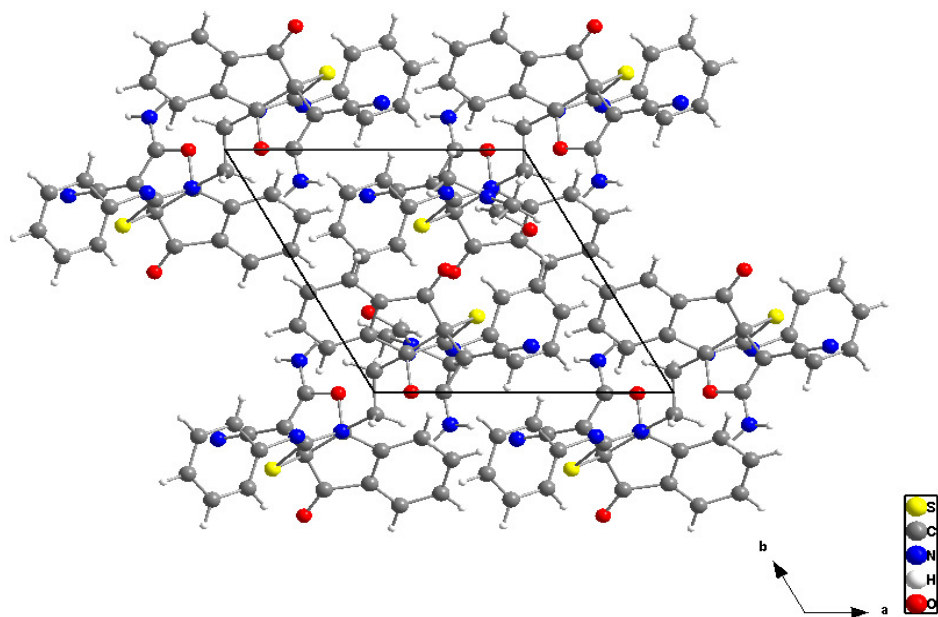


Figure 2. Crystal packing of **7a** (in the voids are DMF).

Table 1. Crystal data for **7a**

$C_{40}H_{26}N_8O_4S_2 \cdot 2(C_3H_7NO)$	$Z = 1$
$M_r = 893.00$	$F(000) = 466$
Triclinic, $P-1$ (<i>no.2</i>)	$D_x = 1.357 \text{ Mg m}^{-3}$
$a = 9.7515$ (5) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.8024$ (8) Å	Cell parameters from 2581 reflections
$c = 13.4038$ (8) Å	$\theta = 2.4\text{--}29.6^\circ$
$\alpha = 90.279$ (6) $^\circ$	$\mu = 0.18 \text{ mm}^{-1}$
$\beta = 102.516$ (5) $^\circ$	$T = 173 \text{ K}$
$\gamma = 118.268$ (7) $^\circ$	Plates, colourless
$V = 1092.98$ (14) Å ³	$0.30 \times 0.20 \times 0.10$ mm

Table 2. Data collection for **7a**

Agilent, SuperNova, Single source at offset, Eos diffractometer	4954 independent reflections
Radiation source: SuperNova (Mo) X-ray Source	3998 reflections with $I > 2\sigma(I)$
Detector resolution: 32.0214 pixels mm ⁻¹	$R_{\text{int}} = 0.016$
rotation in ω , 1 $^\circ$ scans	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan <i>CrysAlis PRO</i> , Agilent Technologies. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.858$, $T_{\text{max}} = 1.000$	$k = -12 \rightarrow 12$
7638 measured reflections	$l = -13 \rightarrow 17$

Table 3. Refinement data for **7a**

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 0.4217P]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.03$	$(\Delta/\sigma)_{\max} = 0.001$
4954 reflections	$\Delta_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
298 parameters	$\Delta_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$
2 restraints	Extinction correction: <i>SHELXL</i> , $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0088 (15)

Table 4. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for **7a**

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.50133 (5)	0.68657 (5)	0.32202 (3)	0.03351 (14)
C2	0.66191 (18)	0.79297 (18)	0.43225 (12)	0.0266 (3)
N3	0.80959 (15)	0.84120 (15)	0.41067 (10)	0.0259 (3)
C4	0.81228 (18)	0.83774 (18)	0.30497 (12)	0.0242 (3)
C5	0.9020 (2)	0.7625 (2)	0.27211 (13)	0.0288 (4)
C6	1.0661 (2)	0.8146 (2)	0.29775 (16)	0.0419 (5)
H6	1.1392	0.9092	0.3417	0.050*
C7	1.1193 (3)	0.7243 (3)	0.2572 (2)	0.0590 (6)
H7	1.2311	0.7573	0.2745	0.071*
C8	1.0146 (3)	0.5867 (3)	0.1920 (2)	0.0611 (6)
H8	1.0552	0.5265	0.1663	0.073*
C9	0.8521 (3)	0.5367 (2)	0.16434 (17)	0.0482 (5)
H9	0.7798	0.4435	0.1188	0.058*
C10	0.7968 (2)	0.6265 (2)	0.20494 (14)	0.0331 (4)
C11	0.6311 (2)	0.6013 (2)	0.18278 (14)	0.0333 (4)
O11	0.51103 (17)	0.49477 (16)	0.12920 (12)	0.0537 (4)

C12	0.63788 (18)	0.74375 (19)	0.23738 (12)	0.0267 (3)
C13	0.62873 (19)	0.85742 (19)	0.16584 (12)	0.0280 (4)
C14	0.76816 (19)	0.99725 (19)	0.19299 (12)	0.0262 (3)
N14	0.81327 (19)	1.13119 (18)	0.15477 (12)	0.0359 (4)
H14A	0.914 (2)	1.207 (2)	0.1778 (17)	0.054*
H14B	0.747 (2)	1.137 (3)	0.1019 (14)	0.054*
O15	0.87662 (12)	0.99695 (12)	0.27543 (8)	0.0269 (3)
C16	0.4923 (2)	0.8296 (2)	0.08880 (14)	0.0346 (4)
N17	0.3808 (2)	0.8088 (2)	0.02660 (14)	0.0505 (5)
N18	0.64954 (17)	0.82089 (17)	0.52141 (11)	0.0337 (3)
C19	0.4960 (2)	0.7639 (2)	0.54239 (14)	0.0350 (4)
C20	0.4663 (3)	0.8708 (3)	0.58898 (16)	0.0484 (5)
H20	0.5455	0.9780	0.6030	0.058*
C21	0.3219 (3)	0.8216 (3)	0.61498 (19)	0.0618 (7)
H21	0.3014	0.8956	0.6454	0.074*
C22	0.2078 (3)	0.6667 (3)	0.59722 (19)	0.0601 (6)
H22	0.1098	0.6332	0.6169	0.072*
C23	0.2358 (3)	0.5605 (3)	0.5510 (2)	0.0570 (6)
H23	0.1562	0.4535	0.5377	0.068*
C24	0.3794 (2)	0.6080 (2)	0.52361 (17)	0.0455 (5)
H24	0.3978	0.5335	0.4918	0.055*
C25	0.95697 (18)	0.91077 (19)	0.49401 (12)	0.0275 (4)
H25A	1.0305	0.8742	0.4799	0.033*
H25B	0.9299	0.8748	0.5595	0.033*
O1D	0.14507	0.33006	0.22949	0.0611 (4)

	(17)	(18)	(14)	
C1D	0.2159 (2)	0.2568 (3)	0.22229 (18)	0.0496 (5)
H1D	0.2730	0.2426	0.2847	0.060*
N1D	0.2202 (2)	0.1958 (2)	0.13559 (15)	0.0516 (5)
C2D	0.3156 (4)	0.1194 (3)	0.1349 (2)	0.0793 (8)
H2D1	0.3617	0.1117	0.2059	0.119*
H2D2	0.4025	0.1803	0.1016	0.119*
H2D3	0.2474	0.0145	0.0968	0.119*
C3D	0.1552 (3)	0.2305 (4)	0.0375 (2)	0.0821 (9)
H3D1	0.0988	0.2875	0.0482	0.123*
H3D2	0.0798	0.1329	-0.0079	0.123*
H3D3	0.2429	0.2947	0.0057	0.123*

Table 7. Atomic displacement parameters \AA^2 for **7a**.

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0190 (2)	0.0430 (3)	0.0300 (2)	0.01033 (19)	0.00122 (16)	0.00262 (18)
C2	0.0230 (8)	0.0271 (8)	0.0272 (8)	0.0118 (7)	0.0022 (6)	0.0038 (6)
N3	0.0182 (6)	0.0319 (7)	0.0219 (7)	0.0096 (6)	0.0006 (5)	0.0017 (5)
C4	0.0201 (7)	0.0251 (8)	0.0237 (8)	0.0103 (6)	0.0002 (6)	0.0029 (6)
C5	0.0310 (8)	0.0331 (9)	0.0275 (8)	0.0199 (7)	0.0066 (7)	0.0076 (7)
C6	0.0321 (9)	0.0472 (11)	0.0512 (12)	0.0242 (9)	0.0080 (8)	0.0053 (9)
C7	0.0450 (12)	0.0678 (15)	0.0812 (18)	0.0408 (12)	0.0159 (12)	0.0055 (13)
C8	0.0687 (15)	0.0613 (15)	0.0809 (18)	0.0498 (14)	0.0273 (13)	0.0080 (13)
C9	0.0618 (13)	0.0385 (11)	0.0534 (13)	0.0307 (11)	0.0161 (11)	0.0026 (9)
C10	0.0400 (10)	0.0297 (9)	0.0337 (9)	0.0201 (8)	0.0096 (8)	0.0061 (7)
C11	0.0353	0.0285	0.0311 (9)	0.0123 (8)	0.0062 (7)	0.0025

	(9)	(9)				(7)
O11	0.0417 (8)	0.0395 (8)	0.0584 (10)	0.0085 (7)	-0.0005 (7)	-0.0164 (7)
C12	0.0212 (7)	0.0292 (8)	0.0251 (8)	0.0109 (7)	0.0002 (6)	0.0003 (6)
C13	0.0248 (8)	0.0325 (9)	0.0240 (8)	0.0144 (7)	-0.0006 (6)	0.0026 (6)
C14	0.0257 (8)	0.0308 (8)	0.0232 (8)	0.0168 (7)	0.0011 (6)	0.0012 (6)
N14	0.0314 (8)	0.0329 (8)	0.0364 (9)	0.0147 (7)	-0.0029 (7)	0.0091 (7)
O15	0.0224 (5)	0.0265 (6)	0.0262 (6)	0.0106 (5)	-0.0016 (4)	0.0045 (4)
C16	0.0310 (9)	0.0349 (9)	0.0306 (9)	0.0137 (8)	-0.0009 (7)	0.0035 (7)
N17	0.0396 (9)	0.0489 (10)	0.0458 (10)	0.0178 (8)	-0.0130 (8)	0.0069 (8)
N18	0.0291 (7)	0.0423 (9)	0.0285 (8)	0.0165 (7)	0.0073 (6)	0.0047 (6)
C19	0.0315 (9)	0.0501 (11)	0.0285 (9)	0.0230 (9)	0.0092 (7)	0.0120 (8)
C20	0.0482 (12)	0.0570 (13)	0.0451 (12)	0.0270 (11)	0.0178 (10)	0.0010 (10)
C21	0.0644 (15)	0.0829 (18)	0.0587 (15)	0.0461 (15)	0.0293 (12)	0.0060 (13)
C22	0.0475 (13)	0.0885 (19)	0.0621 (15)	0.0397 (14)	0.0311 (11)	0.0280 (13)
C23	0.0453 (12)	0.0561 (14)	0.0752 (17)	0.0237 (11)	0.0277 (12)	0.0289 (12)
C24	0.0425 (11)	0.0471 (12)	0.0579 (13)	0.0265 (10)	0.0216 (10)	0.0195 (10)
C25	0.0211 (7)	0.0334 (9)	0.0237 (8)	0.0126 (7)	-0.0012 (6)	0.0034 (6)
O1D	0.0389 (8)	0.0463 (9)	0.0858 (13)	0.0160 (7)	0.0035 (8)	-0.0015 (8)
C1D	0.0381 (11)	0.0503 (12)	0.0526 (13)	0.0182 (10)	0.0041 (9)	0.0114 (10)
N1D	0.0502 (10)	0.0527 (11)	0.0491 (11)	0.0213 (9)	0.0157 (9)	0.0153 (9)
C2D	0.092 (2)	0.0783 (19)	0.089 (2)	0.0499 (17)	0.0427 (18)	0.0208 (16)
C3D	0.0672 (17)	0.106 (2)	0.0490 (15)	0.0279 (17)	0.0022 (13)	0.0184 (15)

Geometric parameters (Å, °) for **7a**

S1—C2	1.7771 (16)	N14—H14B	0.871 (15)
S1—C12	1.8245 (17)	C16—N17	1.146 (2)
C2—N18	1.268 (2)	N18—C19	1.423 (2)
C2—N3	1.385 (2)	C19—C24	1.385 (3)
N3—C4	1.423 (2)	C19—C20	1.390 (3)
N3—C25	1.4652 (18)	C20—C21	1.381 (3)
C4—O15	1.4739 (18)	C20—H20	0.9500
C4—C5	1.509 (2)	C21—C22	1.372 (4)
C4—C12	1.550 (2)	C21—H21	0.9500
C5—C6	1.388 (2)	C22—C23	1.370 (3)
C5—C10	1.390 (2)	C22—H22	0.9500
C6—C7	1.378 (3)	C23—C24	1.385 (3)
C6—H6	0.9500	C23—H23	0.9500
C7—C8	1.387 (3)	C24—H24	0.9500
C7—H7	0.9500	C25—C25 ⁱ	1.529 (3)
C8—C9	1.378 (3)	C25—H25A	0.9900
C8—H8	0.9500	C25—H25B	0.9900
C9—C10	1.390 (3)	O1D—C1D	1.225 (3)
C9—H9	0.9500	C1D—N1D	1.322 (3)
C10—C11	1.474 (2)	C1D—H1D	0.9500
C11—O11	1.203 (2)	N1D—C2D	1.445 (3)
C11—C12	1.540 (2)	N1D—C3D	1.447 (3)
C12—C13	1.495 (2)	C2D—H2D1	0.9800
C13—C14	1.369 (2)	C2D—H2D2	0.9800
C13—C16	1.403 (2)	C2D—H2D3	0.9800
C14—N14	1.318 (2)	C3D—H3D1	0.9800
C14—O15	1.3551 (18)	C3D—H3D2	0.9800
N14—H14A	0.888 (15)	C3D—H3D3	0.9800
C2—S1—C12	92.11 (7)	C14—N14— H14B	118.1 (14)
N18—C2—N3	122.07	H14A—N14—	123 (2)

	(14)	H14B	
N18—C2—S1	126.61 (13)	C14—O15—C4	108.30 (11)
N3—C2—S1	111.30 (12)	N17—C16—C13	178.9 (2)
C2—N3—C4	117.03 (12)	C2—N18—C19	120.80 (15)
C2—N3—C25	120.23 (13)	C24—C19—C20	118.90 (18)
C4—N3—C25	122.28 (13)	C24—C19—N18	123.59 (17)
N3—C4—O15	110.09 (12)	C20—C19—N18	117.43 (17)
N3—C4—C5	116.58 (13)	C21—C20—C19	120.2 (2)
O15—C4—C5	108.85 (13)	C21—C20—H20	119.9
N3—C4—C12	109.35 (13)	C19—C20—H20	119.9
O15—C4— C12	106.09 (12)	C22—C21—C20	120.5 (2)
C5—C4—C12	105.29 (13)	C22—C21—H21	119.7
C6—C5—C10	120.52 (17)	C20—C21—H21	119.7
C6—C5—C4	128.68 (16)	C23—C22—C21	119.7 (2)
C10—C5—C4	110.76 (14)	C23—C22—H22	120.2
C7—C6—C5	117.71 (19)	C21—C22—H22	120.2
C7—C6—H6	121.1	C22—C23—C24	120.5 (2)
C5—C6—H6	121.1	C22—C23—H23	119.7
C6—C7—C8	122.0 (2)	C24—C23—H23	119.7
C6—C7—H7	119.0	C19—C24—C23	120.2 (2)
C8—C7—H7	119.0	C19—C24—H24	119.9
C9—C8—C7	120.4 (2)	C23—C24—H24	119.9
C9—C8—H8	119.8	N3—C25—C25 ⁱ	112.39 (16)
C7—C8—H8	119.8	N3—C25— H25A	109.1
C8—C9—C10	118.1 (2)	C25 ⁱ —C25—	109.1

		H25A	
C8—C9—H9	120.9	N3—C25— H25B	109.1
C10—C9—H9	120.9	C25 ⁱ —C25— H25B	109.1
C5—C10—C9	121.17 (17)	H25A—C25— H25B	107.9
C5—C10— C11	110.85 (15)	O1D—C1D— N1D	126.0 (2)
C9—C10— C11	127.92 (17)	O1D—C1D— H1D	117.0
O11—C11— C10	128.27 (18)	N1D—C1D— H1D	117.0
O11—C11— C12	124.49 (17)	C1D—N1D— C2D	121.7 (2)
C10—C11— C12	107.17 (14)	C1D—N1D— C3D	120.2 (2)
C13—C12— C11	113.09 (14)	C2D—N1D— C3D	117.0 (2)
C13—C12— C4	102.01 (12)	N1D—C2D— H2D1	109.5
C11—C12— C4	104.96 (13)	N1D—C2D— H2D2	109.5
C13—C12—S1	115.58 (12)	H2D1—C2D— H2D2	109.5
C11—C12—S1	111.88 (12)	N1D—C2D— H2D3	109.5
C4—C12—S1	108.13 (11)	H2D1—C2D— H2D3	109.5
C14—C13— C16	125.02 (16)	H2D2—C2D— H2D3	109.5
C14—C13— C12	109.73 (13)	N1D—C3D— H3D1	109.5
C16—C13— C12	125.00 (15)	N1D—C3D— H3D2	109.5
N14—C14— O15	115.07 (14)	H3D1—C3D— H3D2	109.5
N14—C14— C13	131.58 (15)	N1D—C3D— H3D3	109.5
O15—C14— C13	113.34 (14)	H3D1—C3D— H3D3	109.5
C14—N14— H14A	118.6 (15)	H3D2—C3D— H3D3	109.5

C12—S1— C2—N18	170.23 (16)	C5—C4—C12— C13	-108.46 (14)
C12—S1— C2—N3	-11.52 (12)	N3—C4—C12— C11	-116.30 (14)
N18—C2— N3—C4	-165.20 (16)	O15—C4— C12—C11	125.00 (13)
S1—C2—N3— C4	16.45 (17)	C5—C4—C12— C11	9.69 (16)
N18—C2— N3—C25	7.3 (2)	N3—C4—C12— S1	3.26 (15)
S1—C2—N3— C25	-171.07 (11)	O15—C4— C12—S1	-115.44 (12)
C2—N3— C4—O15	103.49 (15)	C5—C4—C12— S1	129.26 (12)
C25—N3— C4—O15	-68.83 (18)	C2—S1—C12— C13	-109.09 (12)
C2—N3— C4—C5	-131.93 (15)	C2—S1—C12— C11	119.56 (12)
C25—N3— C4—C5	55.8 (2)	C2—S1—C12— C4	4.45 (12)
C2—N3— C4—C12	-12.70 (19)	C11—C12— C13—C14	-116.71 (16)
C25—N3— C4—C12	174.99 (13)	C4—C12— C13—C14	-4.52 (18)
N3—C4— C5—C6	-68.6 (2)	S1—C12— C13—C14	112.51 (14)
O15—C4— C5—C6	56.6 (2)	C11—C12— C13—C16	68.8 (2)
C12—C4— C5—C6	169.97 (18)	C4—C12— C13—C16	-178.97 (17)
N3—C4— C5—C10	113.55 (16)	S1—C12— C13—C16	-61.9 (2)
O15—C4— C5—C10	-121.23 (14)	C16—C13— C14—N14	-3.7 (3)
C12—C4— C5—C10	-7.84 (18)	C12—C13— C14—N14	-178.10 (18)
C10—C5— C6—C7	-2.2 (3)	C16—C13— C14—O15	174.72 (16)
C4—C5— C6—C7	-179.79 (19)	C12—C13— C14—O15	0.3 (2)
C5—C6— C7—C8	0.7 (4)	N14—C14— O15—C4	-176.88 (15)
C6—C7—	0.9 (4)	C13—C14—	4.47 (18)

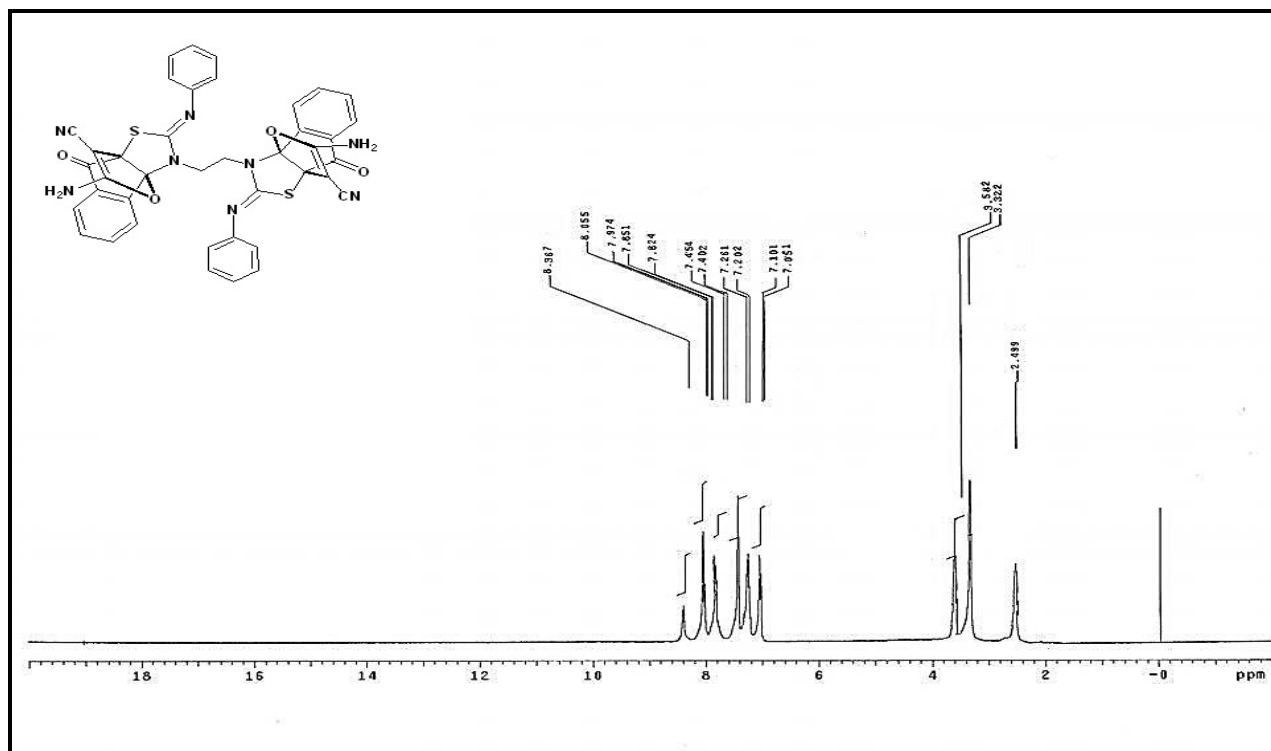
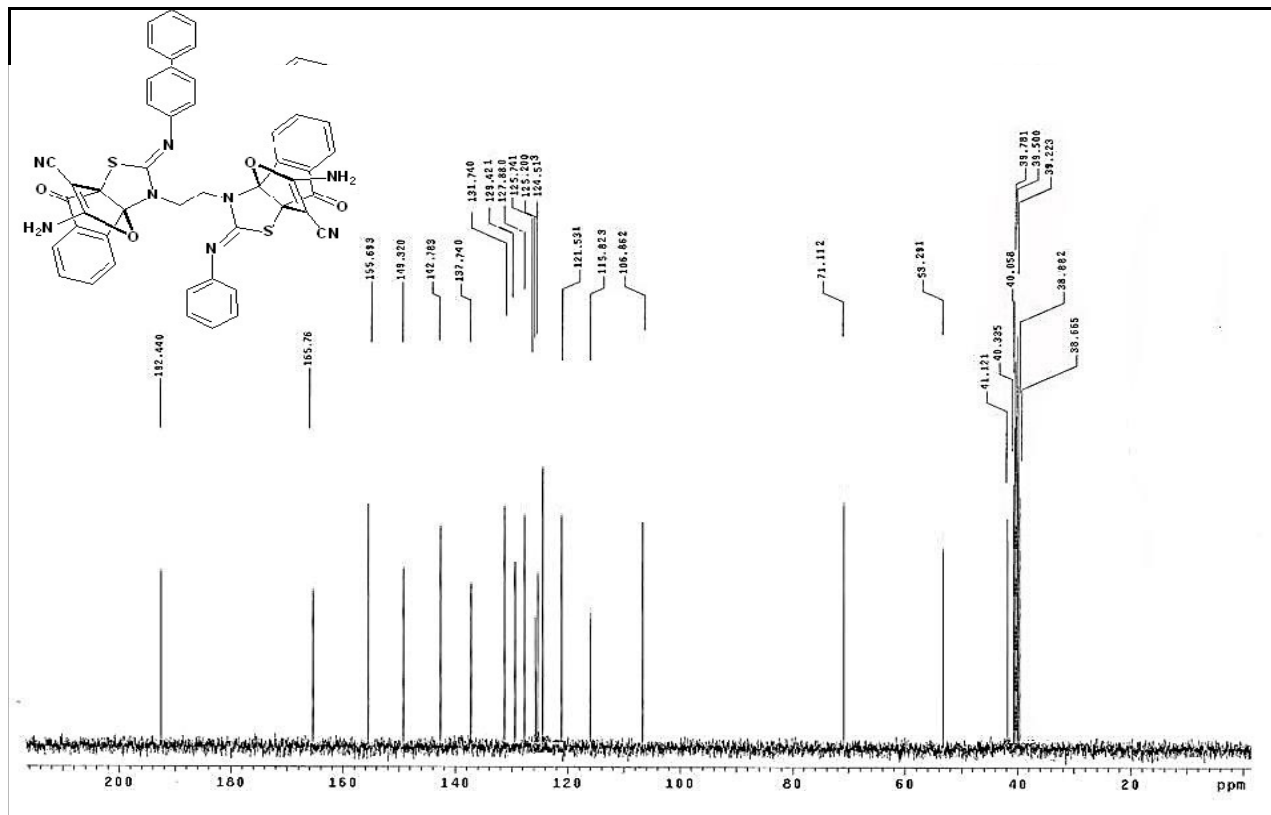
C8—C9		O15—C4	
C7—C8— C9—C10	-1.1 (4)	N3—C4—O15— C14	-125.32 (13)
C6—C5— C10—C9	2.0 (3)	C5—C4—O15— C14	105.76 (14)
C4—C5— C10—C9	-179.94 (16)	C12—C4— O15—C14	-7.11 (16)
C6—C5— C10—C11	-175.48 (16)	N3—C2—N18— C19	-177.20 (15)
C4—C5— C10—C11	2.5 (2)	S1—C2—N18— C19	0.9 (2)
C8—C9— C10—C5	-0.4 (3)	C2—N18— C19—C24	54.9 (2)
C8—C9— C10—C11	176.7 (2)	C2—N18— C19—C20	-128.4 (2)
C5—C10— C11—O11	-178.95 (19)	C24—C19— C20—C21	-0.6 (3)
C9—C10— C11—O11	3.7 (3)	N18—C19— C20—C21	-177.45 (19)
C5—C10— C11—C12	3.9 (2)	C19—C20— C21—C22	1.4 (4)
C9—C10— C11—C12	-173.37 (18)	C20—C21— C22—C23	-1.6 (4)
O11—C11— C12—C13	-75.3 (2)	C21—C22— C23—C24	1.0 (4)
C10—C11— C12—C13	101.90 (16)	C20—C19— C24—C23	0.0 (3)
O11—C11— C12—C4	174.29 (18)	N18—C19— C24—C23	176.68 (19)
C10—C11— C12—C4	-8.47 (18)	C22—C23— C24—C19	-0.2 (3)
O11—C11— C12—S1	57.3 (2)	C2—N3—C25— C25 ⁱ	-97.0 (2)
C10—C11— C12—S1	-125.50 (13)	C4—N3—C25— C25 ⁱ	75.1 (2)
N3—C4— C12—C13	125.55 (14)	O1D—C1D— N1D—C2D	176.1 (2)
O15—C4— C12—C13	6.84 (16)	O1D—C1D— N1D—C3D	8.5 (3)

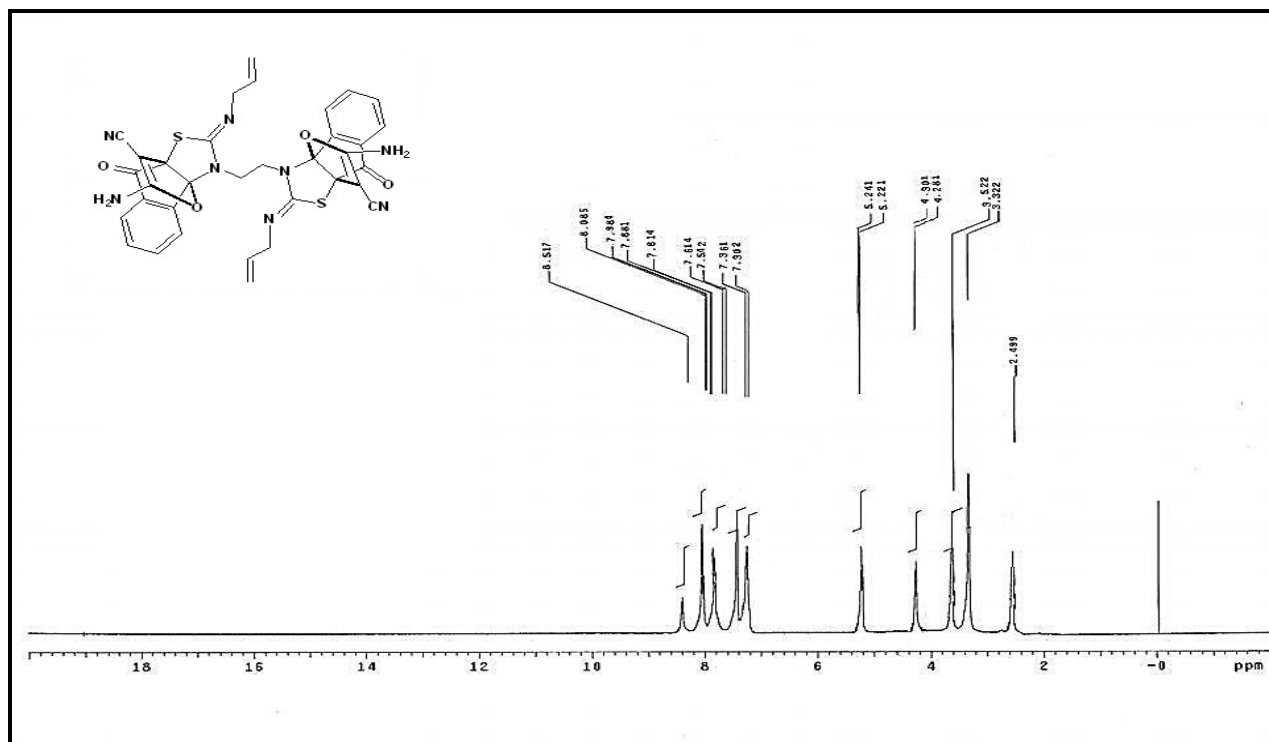
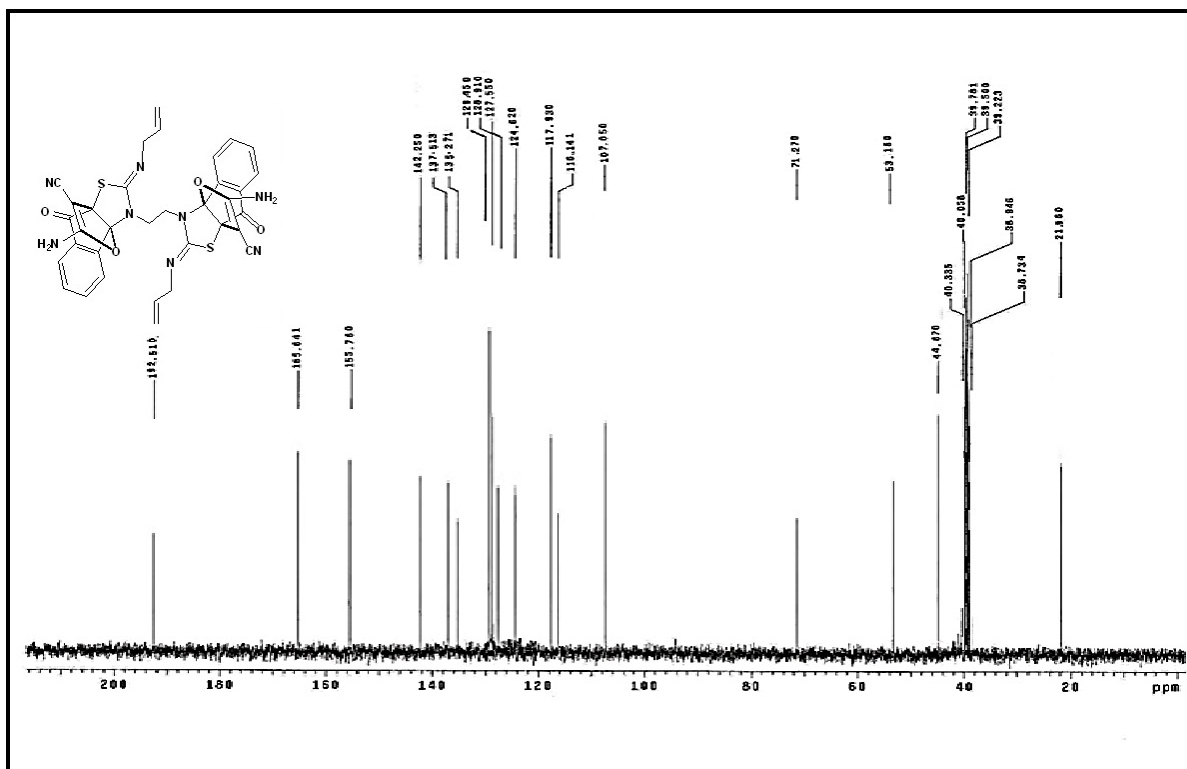
Symmetry code: (i) $-x+2, -y+2, -z+1$.

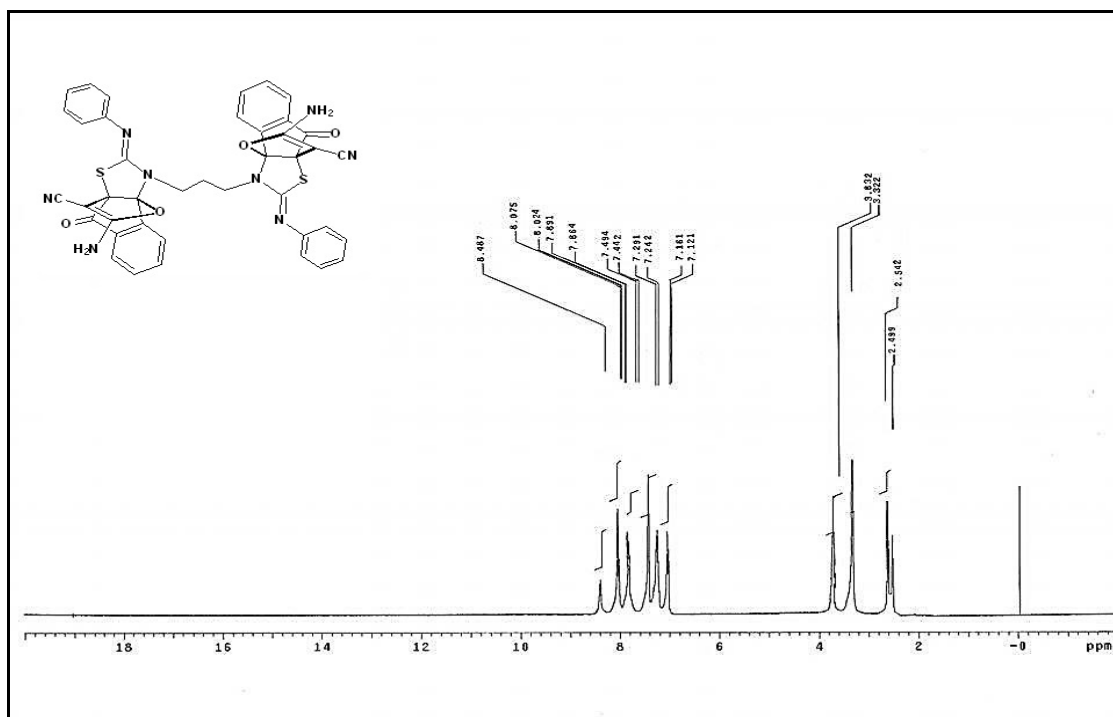
Hydrogen-bond geometry (Å, °) for **7a**.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N14— H14A \cdots O1D ⁱⁱ	0.89 (2)	1.94 (2)	2.806 (2)	166 (2)
N14— H14B \cdots N17 ⁱⁱⁱ	0.87 (2)	2.12 (2)	2.985 (2)	170 (2)
C25— H25B \cdots O15 ⁱ	0.99	2.44	3.0677 (19)	121
C3D— H3D3 \cdots O11	0.98	2.58	3.141 (3)	116

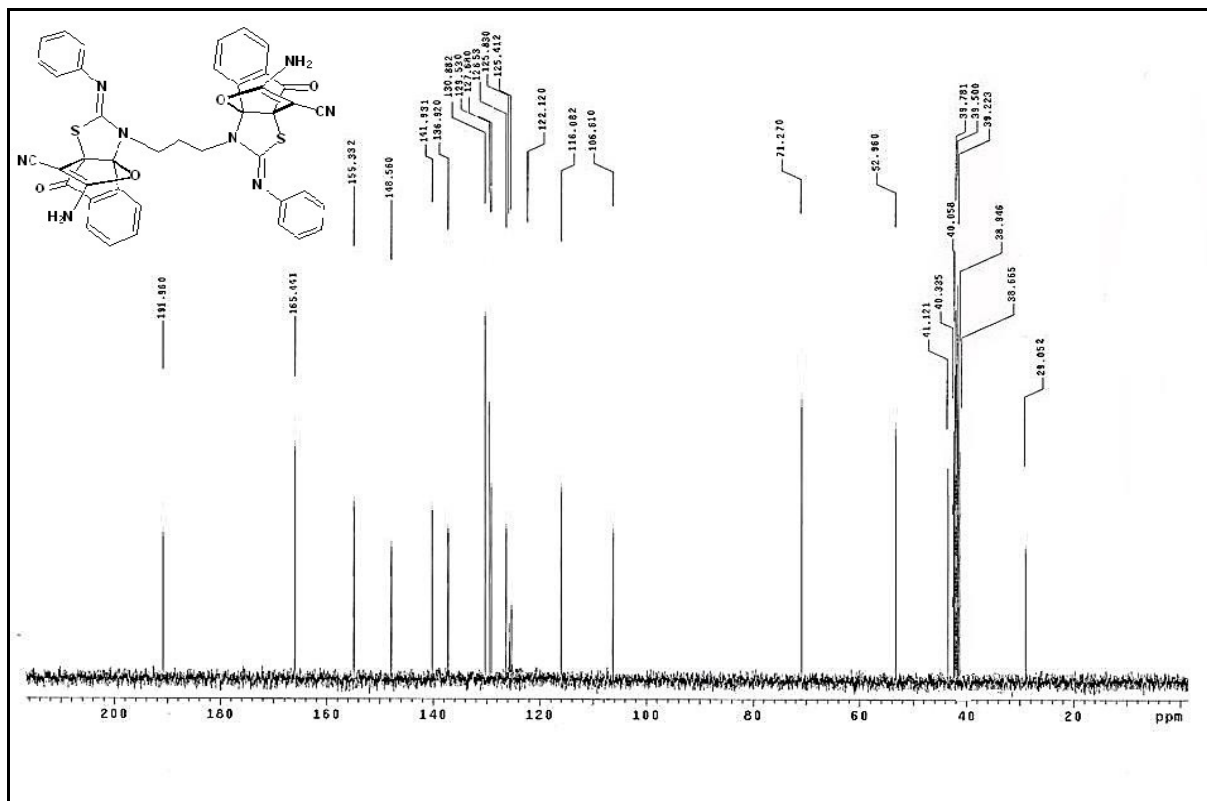
Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $x+1, y+1, z$; (iii) $-x+1, -y+2, -z$.

¹H-NMR of (7a):-**¹³C-NMR of (7a):-**

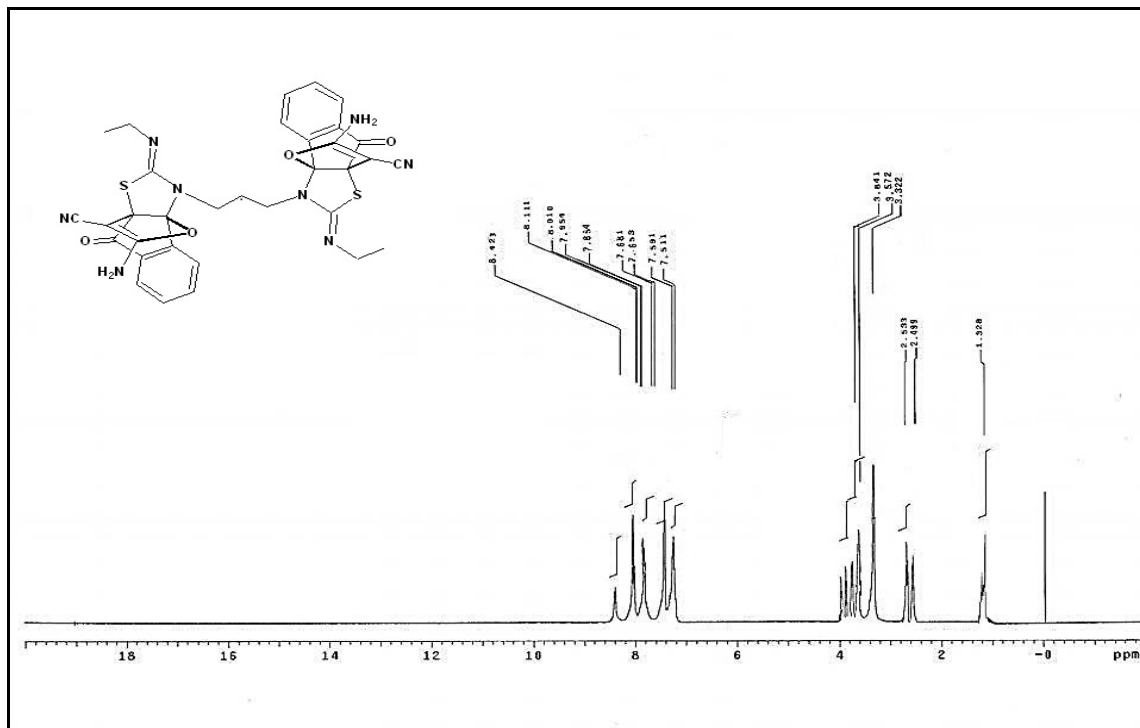
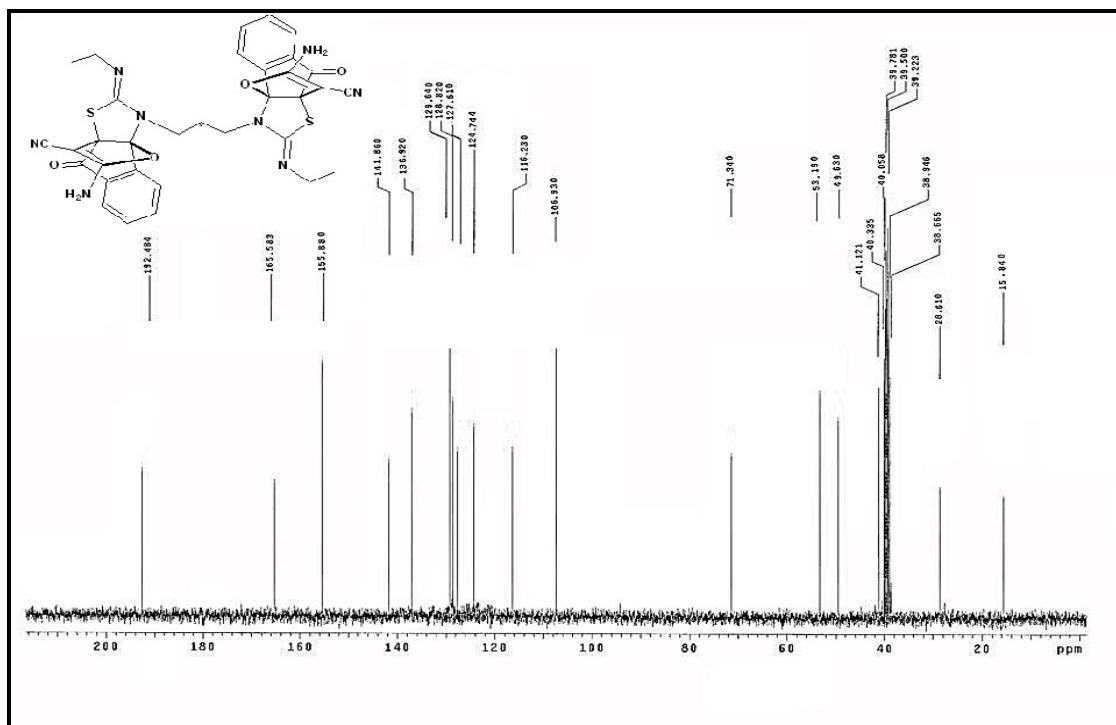
¹H-NMR of (7b):-**¹³C-NMR of (7b):-**

1 $^1\text{H-NMR}$ of (7c):-

2

3 $^{13}\text{C-NMR}$ of (7c):-

4

10
11 **$^1\text{H-NMR}$ of (7e):-**12
13 **$^{13}\text{C-NMR}$ of (7e):-**14
15