

Three-component synthesis of new γ -piperidone-incorporating aza-14-crown-4 compounds

Anh T. T. Dang, Nhung T. Dao, Dat T. Nguyen, Alexander A. Titov, Hanh M. T. Luong, Victor N. Khrustalev, Van T. T. Tran and Anh T. Le

Table of Contents

1. Chemicals and solvents	S2
2. Instrument.....	S2
3. Spectral data analysis of the synthesized compounds	S2
4. Spectral data	S6
4.1. Compound 5a	S6
4.2. Compound 5b.....	S7
4.3. Compound 5c	S11
4.4. Compound 5d.....	S14
4.5. Compound 5e	S18
4.6. Compound 5g.....	S19
4.7. Compound 5h.....	S20
4.8. Compound 6	S22
5. Single X-ray diffraction of compounds 5c and 7	S23
Crystal data of compound 7	S34

1. Chemicals and solvents

Reagents purchased from commercial sources (Sigma-Aldrich) were used without any additional purification.

2. Instrument

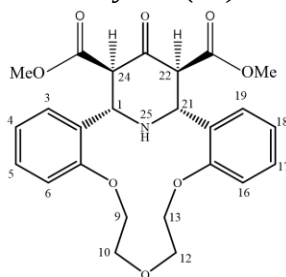
Melting points were determined in open capillary tubes on a digital Stuart SMP3 apparatus. Elemental analysis was conducted on Euro Vector EA-3000 analyser. IR spectra were recorded in FTIR Affinity – 1S SHIMADZU. ¹H and ¹³C NMR spectra were recorded in CDCl₃ solutions at 25°C, using a BRUKER 500 MHz NMR spectrometer at VNU University of Science and TMS as internal standard and chemical shifts are given in parts per million (δ). Signal of the residual protons of the solvent (7.26 ppm for CHCl₃) was used as the reference in ¹H-NMR spectra, while solvents signals (77.2 ppm for CDCl₃) was used as the reference in ¹³C-NMR spectra. Mass spectra were recorded on LTQ Orbitrap XL using electrospray ionization source and on instruments Finnigan MAT 95 XL (EI, ionizing energy 70 eV) at VNU University of Science.

3. Spectral data analysis of the synthesized compounds

Table S1. The reaction conditions for the synthesis of the target compounds

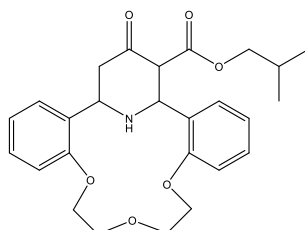
Entry	Comp.	Temperature	Reaction time (hours)	Yield (%)
1.	5a	60 °C	2	76
2.	5b	Refluxed	5	40
3.	5c	Refluxed	5	38
4.	5d	Refluxed	5	34
5.	5e	Refluxed	5	31
6.	5f	Refluxed	5	27
7.	5g	Refluxed	5	29
8.	5h	Refluxed	5	27
9.	6	80 °C	10	11
10.	7	r.t	24	45

3.1. Dimethyl 23-oxo-8,11,14-trioxa-25-azatetracyclo[19.3.1.0^{2,7}.0^{15,20}]pentacosa-2,4,6,15 (20),16,18-hexaen-22,24-dicarboxylate (**5a**)



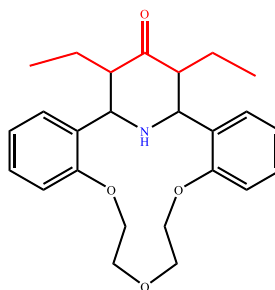
Yield: 7.9 g (76%), white crystals. M.p °C, R_f = 0.46 (ethyl acetate: hexane, 3:1). IR (KBr), ν , cm⁻¹: 3318 (NH), 1750 (O=CO), 1698 (C=O). Mass spectrum, m/z : 469 [M]⁺. ¹H, NMR (CDCl₃, 400MHz), δ , ppm: 3.26 (s, 6H, CH₃), 3.58 (m, 2H, H^{1,21}), 3.76 (d, 2H, J = 11.9 Hz, H^{22,24}), 3.97 (br.s., 1H, NH), 3.84 and 4.05 (both m, 4H each, 2xOCH₂CH₂O), 6.5, 6.56 and 6.90 (ABCD-system, 2H, 2H and 4H, respectively, J = 8.0 Hz, H^{aren}). ¹³C NMR, (CD₃CN, 100 MHz), δ , ppm: 52.6 (CH₃), 63.0 (C-22,24), 66.5 (C-1,21), 68.3 (C-9,13), 70.5 (C-10,12), 112.9 (C-6,16), 121.9 (C-4,18), 127.7 (C-2,20), 131.0 (C-5,17), 132.7 (C-3,19), 158.4 (C-7,15), 170.3 (COO), 202.4 (C-23). Found, %: C 63.75; H 5.81; N 2.80. C₂₅H₂₇NO₈. Calculated, %: C 63.96; H 5.80; N 2.98.

3.2. Isobutyl 2⁴-oxo-4,7,10-trioxa-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclodecaphane-2³-carboxylate (**5c**)



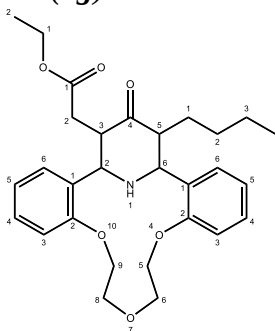
Yield 2.75 g (38 %), white crystals, M.p 217-219 °C (R_f = 0.46, H/E 3/1). IR (KBr), $\nu_{\max}$, cm⁻¹: 3323 (NH); 1740, 1697 (C=O); 1246 (C-O). ¹H NMR ([D₁] CDCl₃, 298 K) δ_{H} ppm: 0.759 (3H, d, J = 7 Hz, CH₃); 0.775 (3H, d, J = 7 Hz, CH₃); 1.739 (1H, m, -CH(CH₃)₂), 2.649 (1H, dd, J = 14 Hz; 3 Hz, CH_{piperidone}), 3.309 (1H, t, J = 13 Hz, CH_{piperidone}), 3.687 (1H, t, J = 7 Hz, CH_{piperidone}), 3.83 (1H, q, J = 7 Hz, CH_{piperidone}), 3.856-3.928 (2H, m, OCH₂), 4.055 (2H, d, J = 9,6 Hz, CH₂CH), 4.112-4.167 (5H, m, CH_{piperidone} & 2xOCH₂), 4.296 (2H, br.s, OCH₂), 6.785-6.849 (3H, m, H_{Ar}), 6.889 (1H, t, J = 7.8 Hz, H_{Ar}), 7.169-7.260 (4H, m, H_{Ar}); ¹³C-NMR, CDCl₃: δ_{C} ppm: 204.77 (-C=O), 169.18 (-C=O ester), 156.96, 156.85, 131.74, 130.51, 129.30, 129.13, 128.47, 126.58, 121.03, 120.84, 111.10, 111.05, 70.76, 69.93, 69.85, 66.48, 66.41, 62.67, 46.75, 27.60, 18.90.

3.3. 22,24-Diethyl-8,11,14-trioxa-25-azatetracyclo[19.3.1.0^{2,7}.0^{15,20}]pentacosa-2,4,6,15(20),16,18-hexaen-23-on (**5e**)



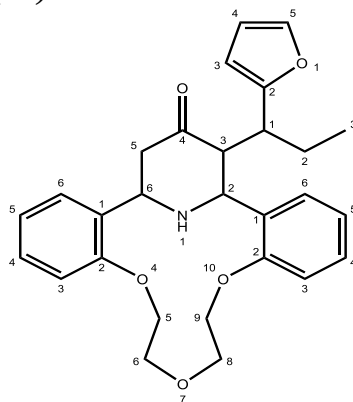
Yield: 2.82 g (31 %), white crystals. M.p. 159 – 160°C (from ethyl acetate), R_f = 0.55 (ethyl acetate). IR (KBr), ν , cm^{-1} : 3315 (NH), 1691 (C=O). Mass spectrum, m/z ($I_{\text{rel.}}\%$): 409 $[M]^+$ (25), 394 (11), 381 (17), 352 (25), 338 (60), 321 (8), 310 (18), 297 (62), 282 (7), 234 (11), 192 (12), 175 (12), 163 (26), 148 (51), 131 (75), 119 (68), 107 (47), 91 (100), 77 (74), 71 (49), 55 (73), 43 (69). ^1H NMR, (DMSO- d_6 , 400 MHz), δ , ppm: 0.65 (6H, t, J = 7.26 Hz, $2\times\text{CH}_2\text{CH}_3$), 0.91 and 1.44 (2H, both br. s, 2H each, $2\times\text{CH}_2\text{CH}_3$), 3.21 (2H, m, $\text{H}^{22,24}$), 3.49 (2H, t, J = 11.0 and 11.4 Hz, $\text{H}^{1,21}$), 3.81 – 4.15 (9H, m, $2\times\text{OCH}_2\text{CH}_2\text{O}$, NH), 6.83, 6.93, 7.11 and 7.22 (8H, J = 8.2, 8.1, 7.47 and 7.3 Hz, ABCD-system, H^{aren}). Found, %: C 72.38; H 7.04; N 3.43. $\text{C}_{25}\text{H}_{31}\text{NO}_4$. Calculated, %: C 72.42; H 7.13; N 3.67.

3.4. Ethyl 2-[24-butyl-23-oxo-8,11,14-trioxa-25-azatetracyclo[19.3.1.0^{2,7}.0^{15,20}]pentacosa-2,4,6,15(20),16,18-hexaen-22-yl]acetate (**5g**)



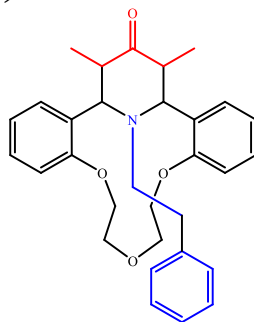
Yield: 3.2 g (29 %), white crystals. M.p. 171-173 °C, R_f = 0.69 (ethyl acetate). IR (KBr), ν , cm^{-1} : 3303 (NH), 1735 (OC=O) and 1699 (C=O). Mass spectrum, m/z ($I_{\text{rel.}}\%$): 495 $[M]^+$ (27), 480 (2), 452 (18), 438 (3), 410 (4), 366 (32), 310 (16), 297 (38), 159 (30), 131 (55), 105 (30), 91 (42), 77 (32), 55 (100), 43 (37). ^1H NMR, (CDCl_3 , 400 MHz), δ , (ppm): 0.71 (3H, t, J = 7.2 Hz, CH_3), 1.14 (3H, t, J = 7.1 Hz, CH_3), 0.98, 1.23 and 1.67 (m, 6H in total, CH_2), 1.94 and 2.62 (1H, both d.d, 1H each, J = 16.7 and 4.6; J = 16.7 и 8.0 Hz, respectively, $\text{C}^{22}\text{-CH}_2\text{C=O}$), 3.41 (1H, br. s, NH), 3.56 – 4.42 (16H, m, $\text{OCH}_2\text{CH}_2\text{O}$, $\text{O=CCH}_2\text{CH}_3$, $\text{-C}^{24}\text{-CH}_2\text{-}$, $\text{H}^{1,21,22,24}$), 6.76 – 7.12 (8H, m, H^{aren}). Found, %: C 70.03; H 7.48; N 2.69. $\text{C}_{29}\text{H}_{37}\text{NO}_6$. Calculated, %: C 70.28; H 7.52; N 2.83.

3.5. 24-[1-(2-Furyl)propyl]-8,11,14-trioxa-25-azatetracyclo[19.3.1.0^{2,7}.0^{15,20}]pentacos-2,4,6,15(20),16,18-hexaen-23-on (**5h**)



Yield: 2.8 g (27 %), white crystals. M.p. 178 – 180°C, *R*_f 0.74 (ethyl acetate). IR (KBr), ν , cm^{-1} : 3322 (NH), 1697 (C=O). Mass spectrum, *m/z* (*I*_{rel.} %): 461 [*M*]⁺ (53), 432 (100), 352 (16), 297 (31), 131 (13), 121 (43), 107 (9), 81 (9). ¹H NMR (CDCl₃, 400 MHz), δ , (ppm): 0.79 (3H, t, *J* = 7.3 Hz, CH₃), 1.7 (2H, octet, *J* = 6.8 and 5.8 Hz, CH₂CH₃), 2.36 (1H, d.d, *J* = 13.1 and 3.1 Hz, H²²), 2.88 (1H, br.t, *J* = 6.8 and 5.8 Hz, CHCH₂CH₃), 3.7 (1H, br. s, NH), 3.75 - 4.09 (12H, m, 2xOCH₂CH₂O, H^{1,21,24}), 5.5 (1H, br. s, *J* = 2.5 Hz, H^{fur}), 6.09 (1H, t, *J* = 2.5 and 2.0 Hz, H^{fur}), 6.71 – 7.23 (8H, m, H^{aren.}), 7.32 (1H, d, *J* = 2.0 Hz, H^{fur}). Found, %: C 72.71; H 6.54; N 2.91. C₂₈H₃₁NO₅. Calculated, %: C 72.86; H 6.77; N 3.03.

3.6. 22,24-Dimethyl-25-phenethyl-8,11,14-trioxa-25-azatetracyclo[19.3.1.0^{2,7}.0^{15,20}]pentacos-2,4,6,15(20),16,18-hezaen-23-one (**6**)



Yield 0.6 g (11%) of **6** was obtained as white crystals with M.p. 178-180°C by recrystallization from hexane – ethyl acetate (1:1, v/v.). *R*_f 0.67 (SiO₂, ethyl acetate). IR (KBr, ν , cm^{-1}): 1702 (C=O). Mass-spectrum (MS), *m/z* (*I*_{relat.}, %): 485 [*M*]⁺ (0), 394 (12), 338 (3), 205 (1), 173 (3), 161 (6), 146 (16), 133 (19), 119 (21), 105 (44), 91 (100), 77 (31), 65 (19), 55 (12), 43 (33). ¹H NMR, (CDCl₃, 400 MHz), δ , (ppm): 0.79 (d, 6H, *J* 6.6 Hz, CH₃), 2.41 (m, 4H, H^{22,24}), 3.48 (d, 2H, *J* 10.3 Hz, H^{1,21}), 3.78 – 4.12 (m, 8H and 4H, OCH₂CH₂O, CH₂), 6.26 – 7.33 (m, 13H, H^{aren.}). Found, %: C 71.81; H 7.75; N 3.12. C₃₁H₃₅NO₄. Calculated, %: C 73.73; H 7.85; N 3.31.

4. Spectral data

4.1. Compound 5a

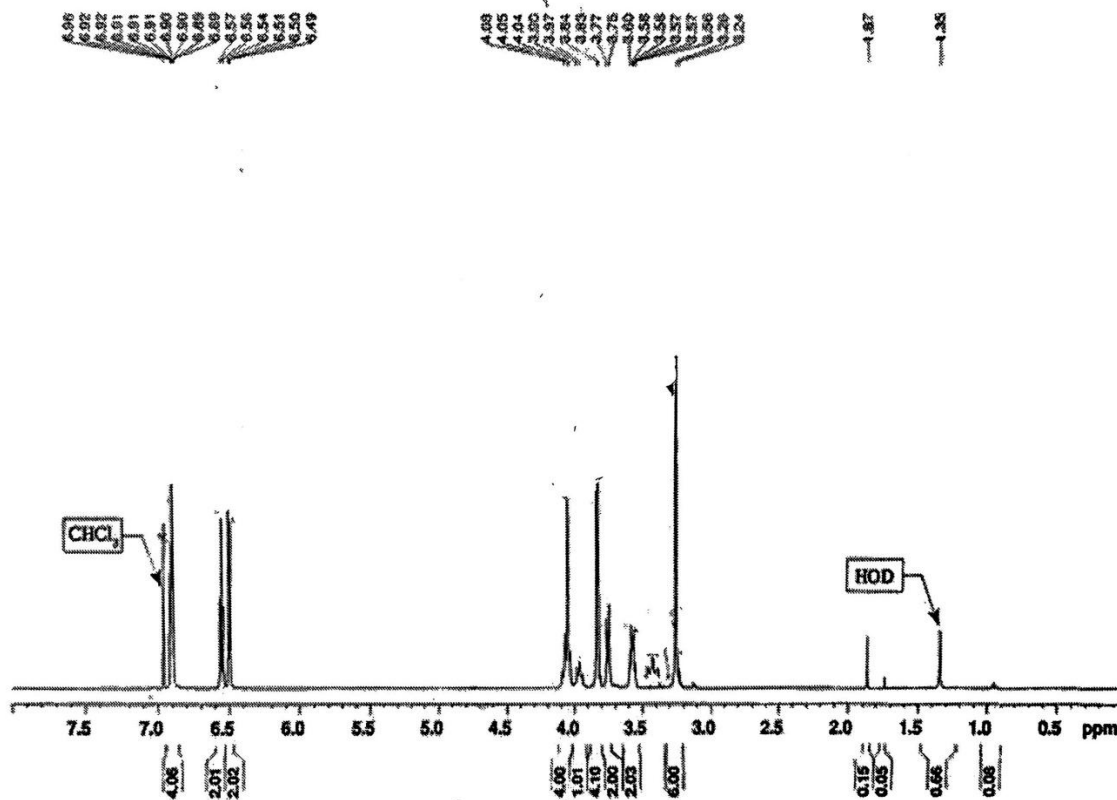


Fig S1. ¹H-NMR of 5a

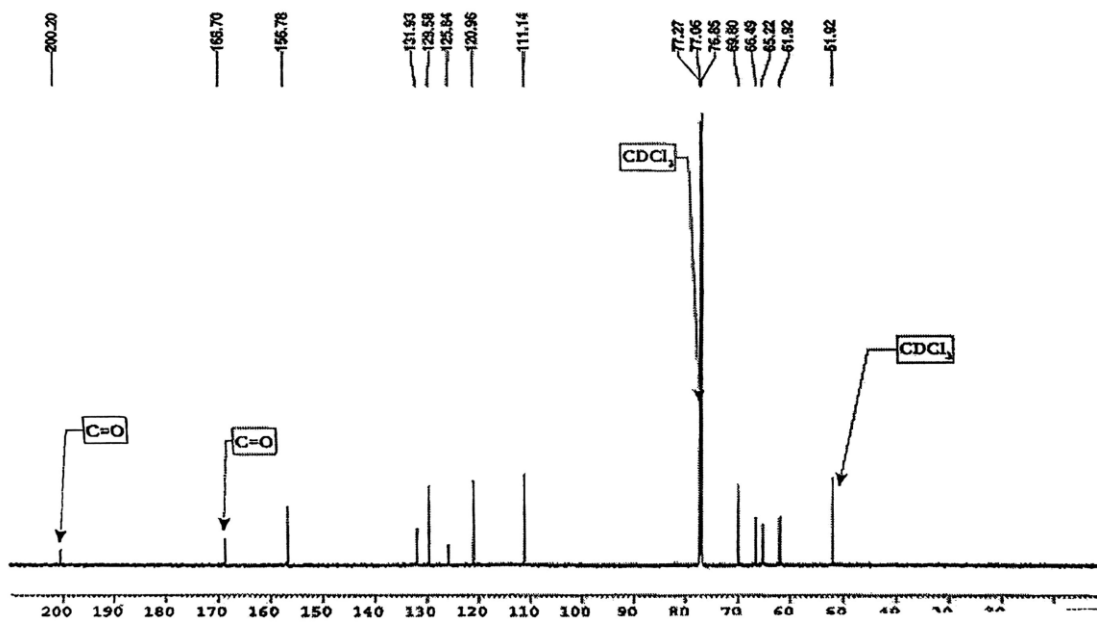


Fig S2. ¹³C-NMR of 5a

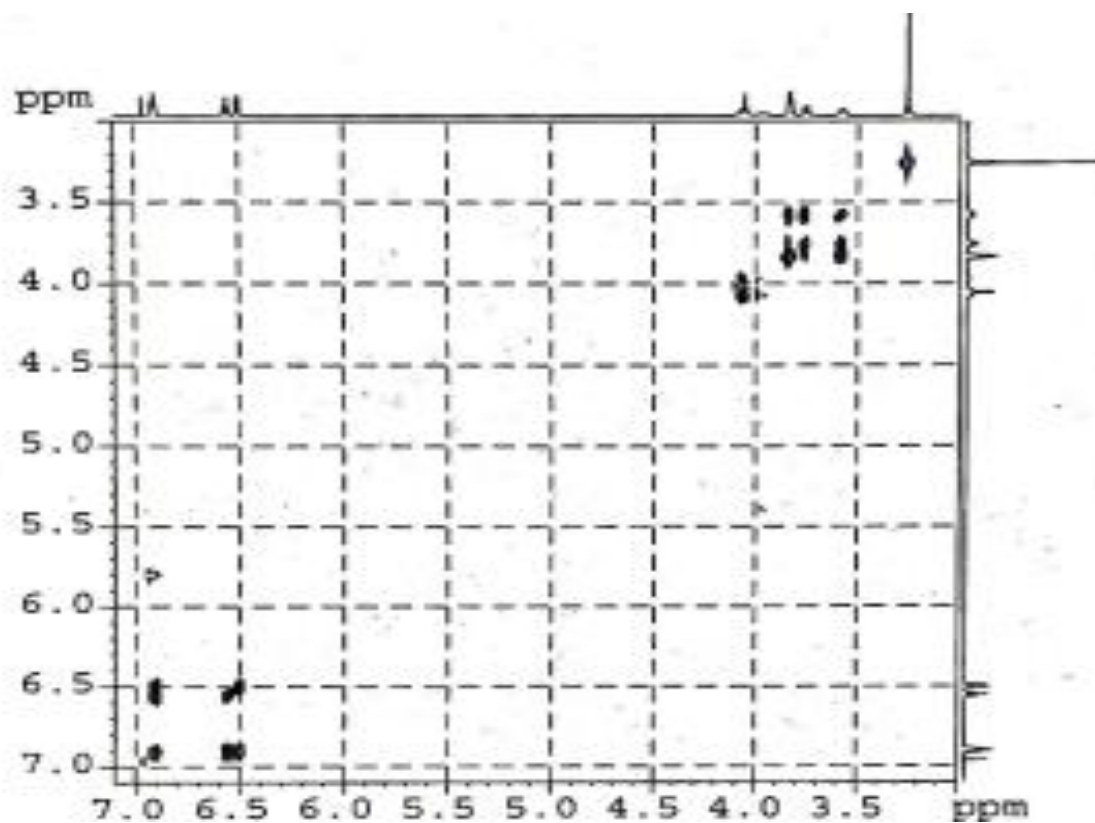
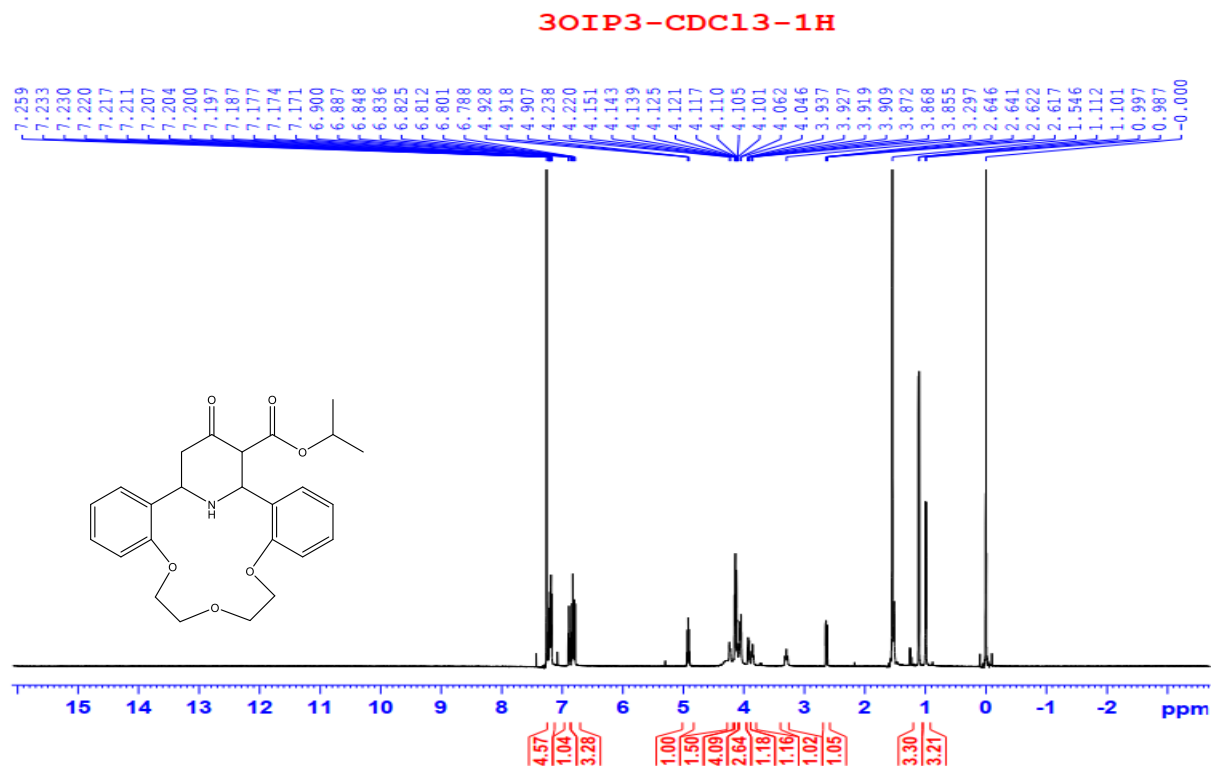
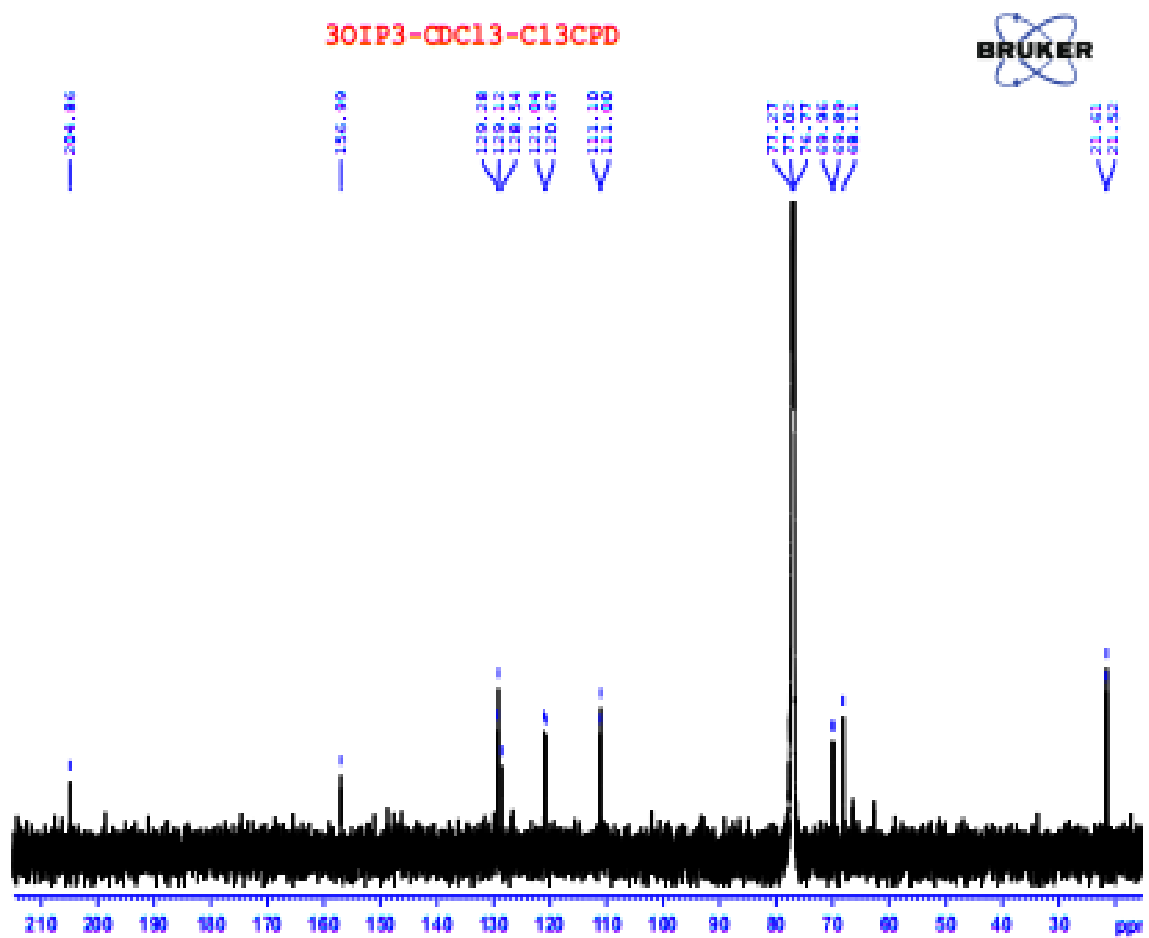
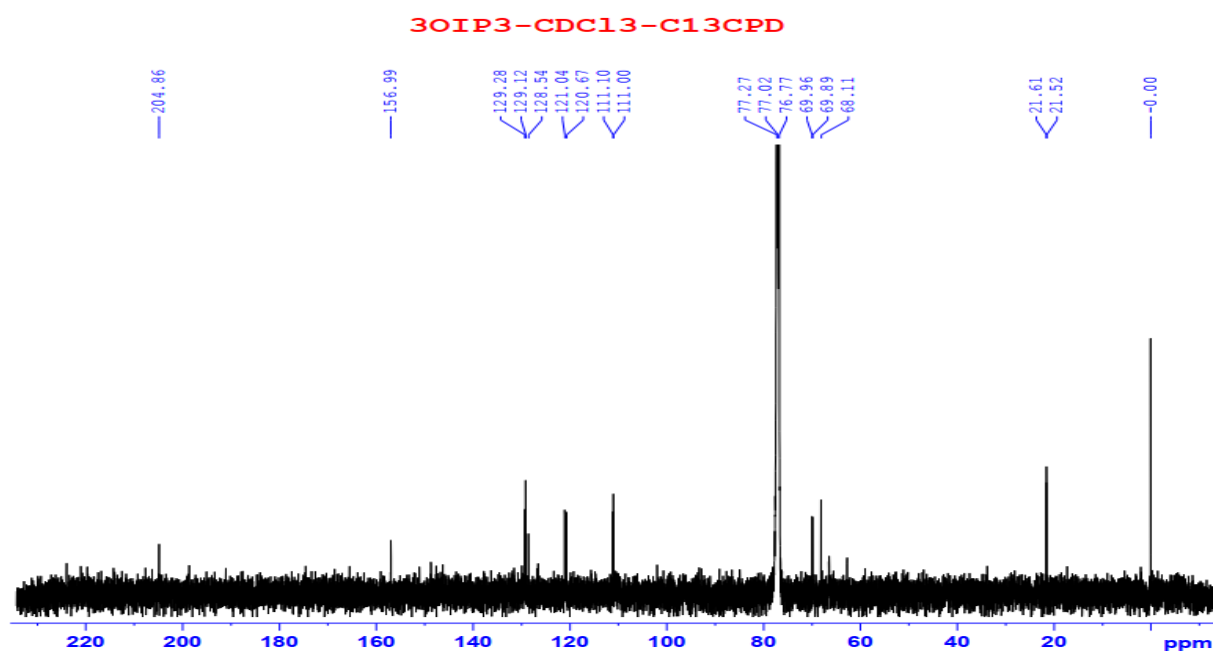


Fig S3. COSY NMR of 5a

4.2. Compound 5b





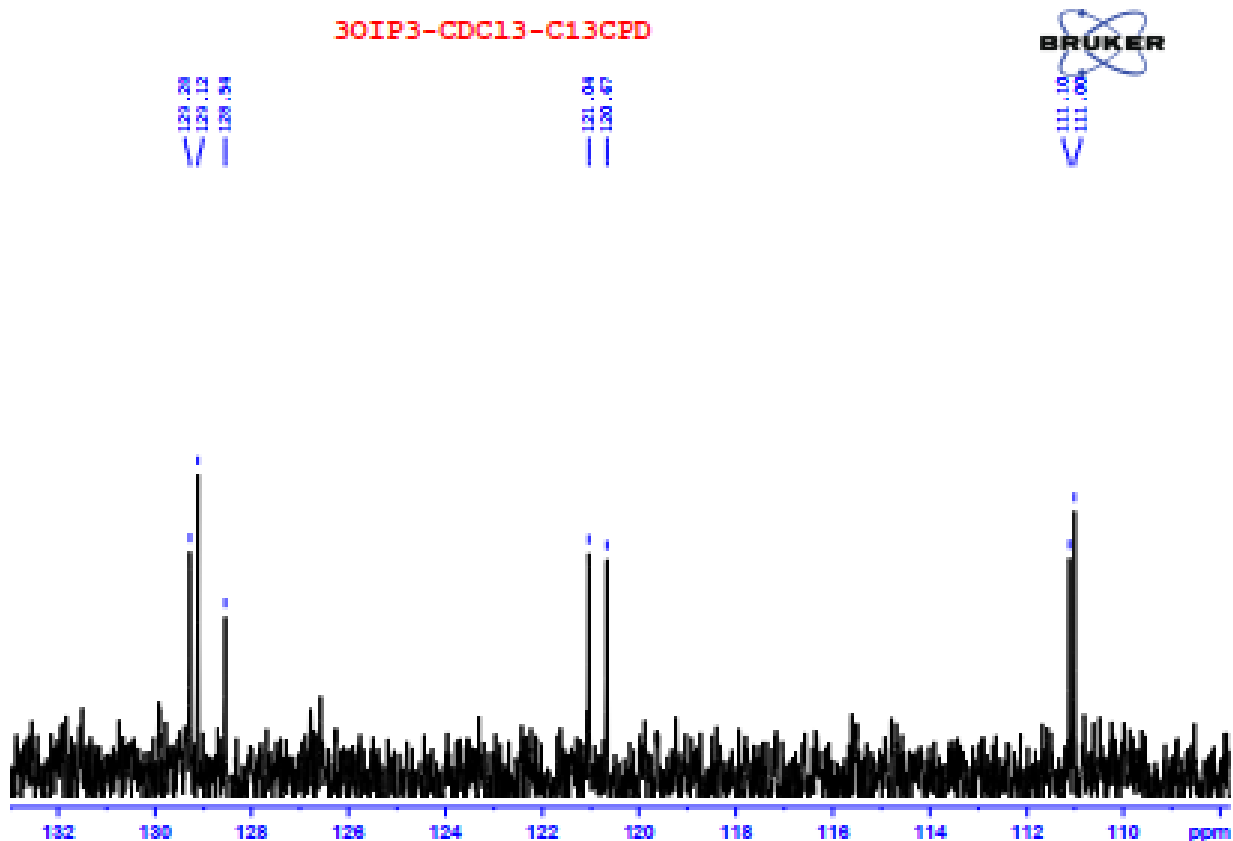


Fig S5. ^{13}C -NMR of **5b**

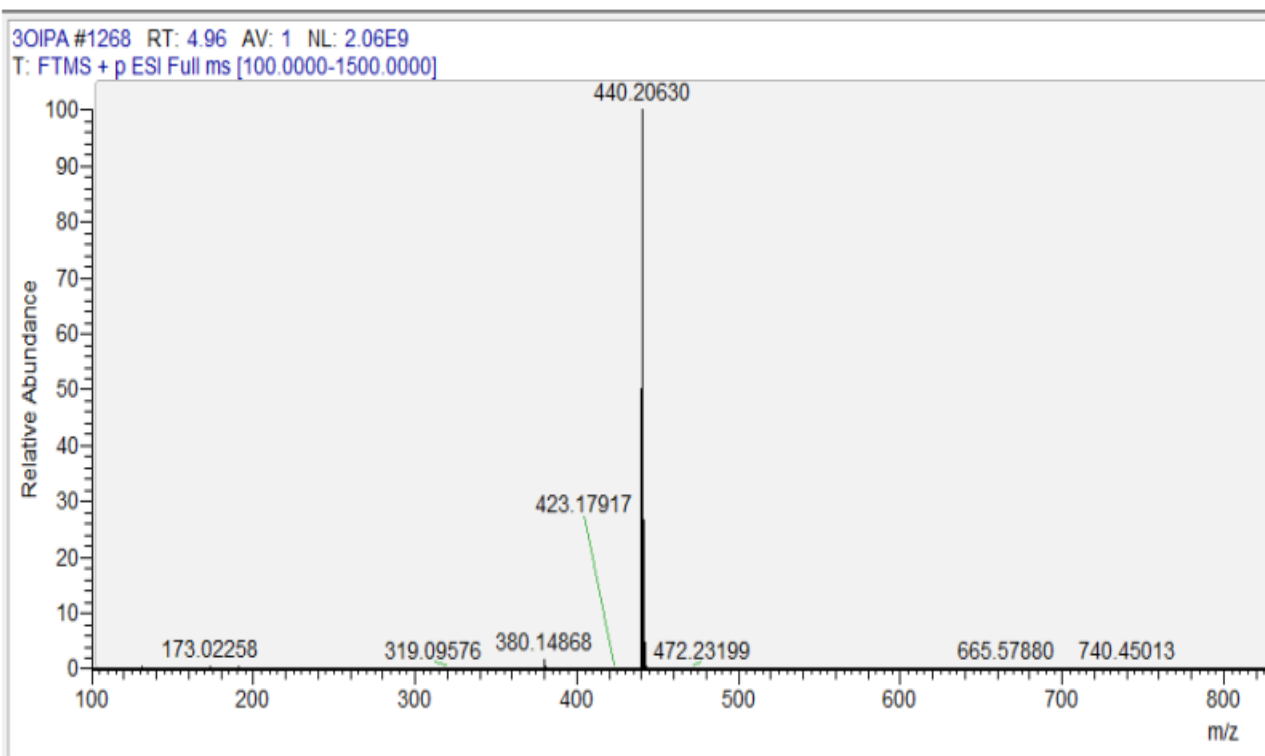


Fig S6. HRMS of **5b**

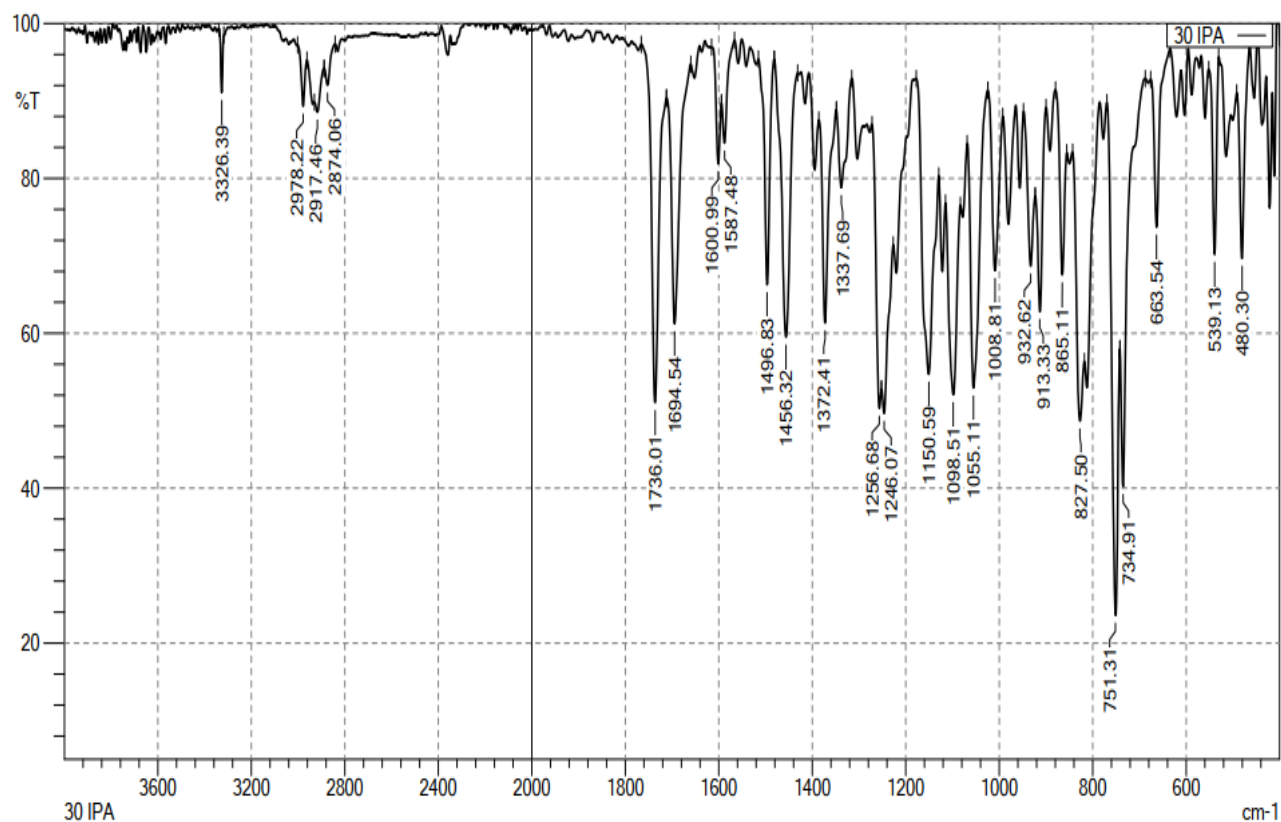
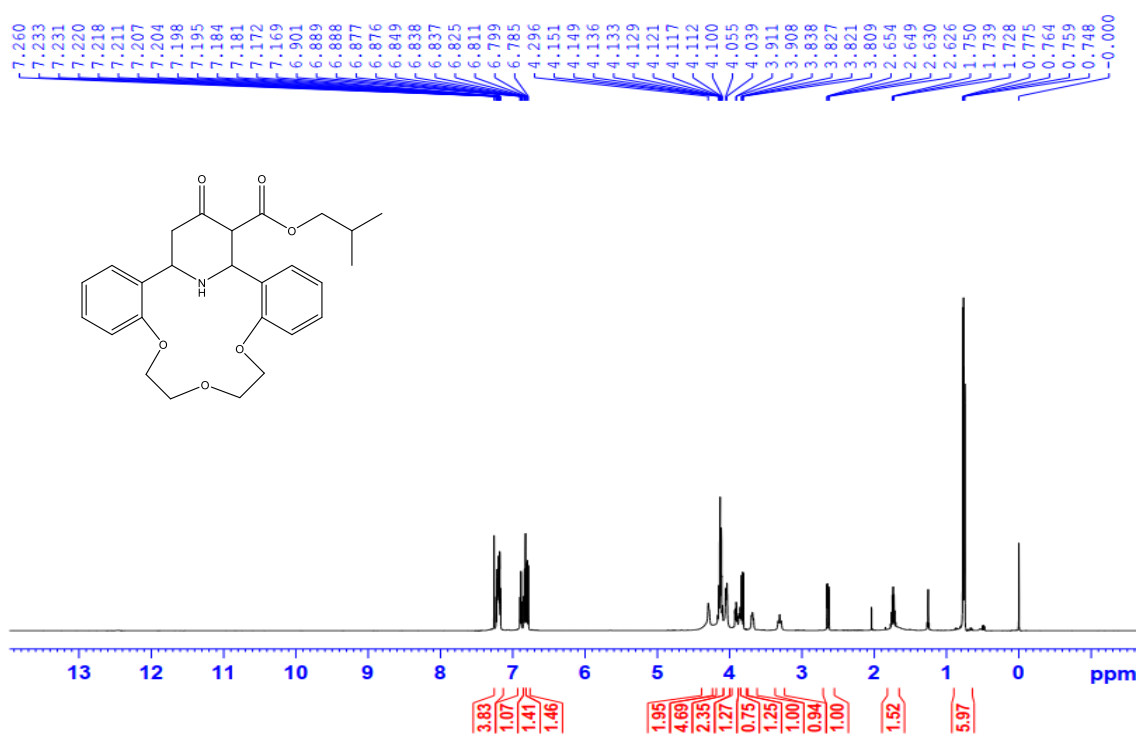


Fig S7. IR of 5b

4.3. Compound 5c

3OIBA-CDC13-1H



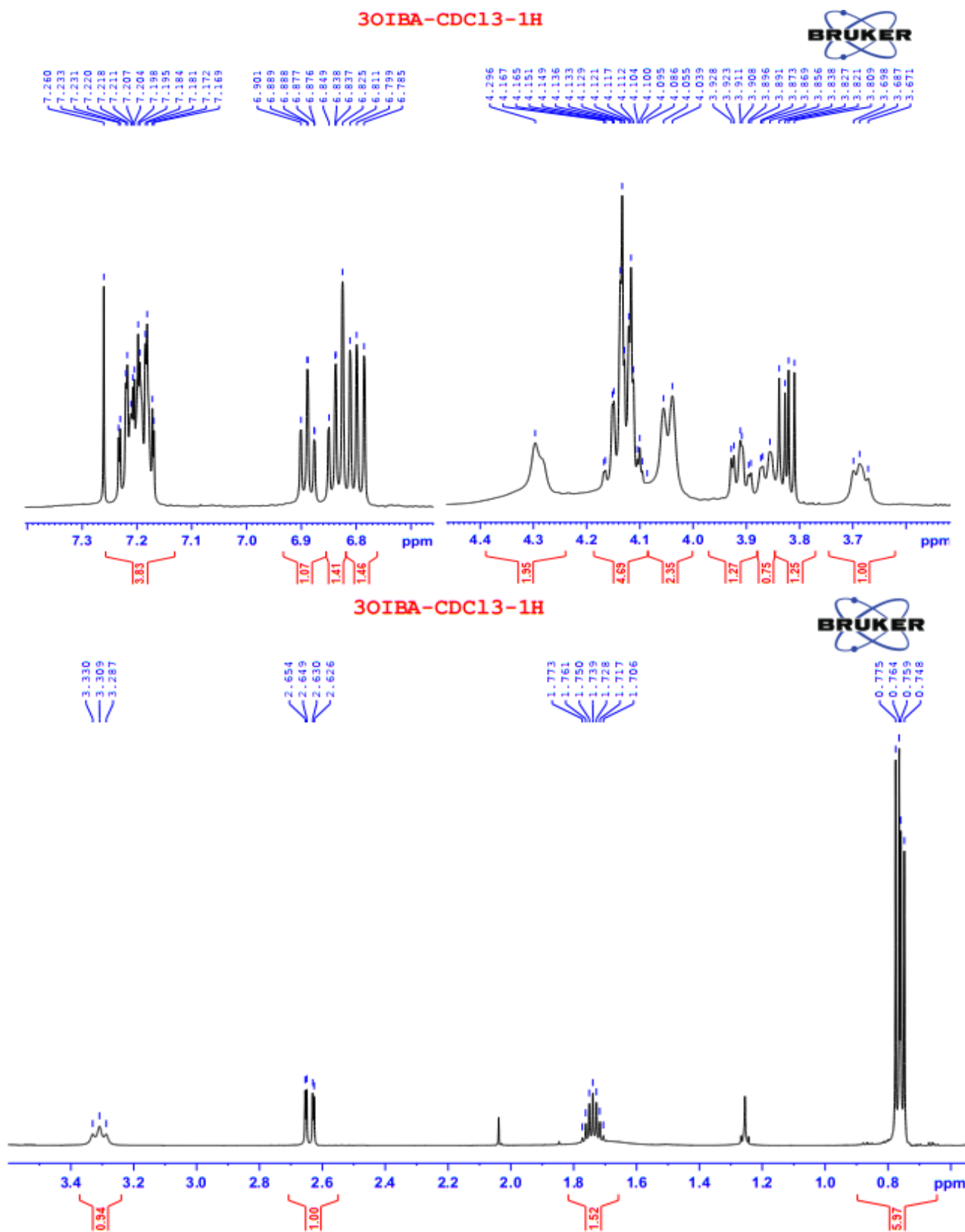


Fig S8. ¹H-NMR of 5c

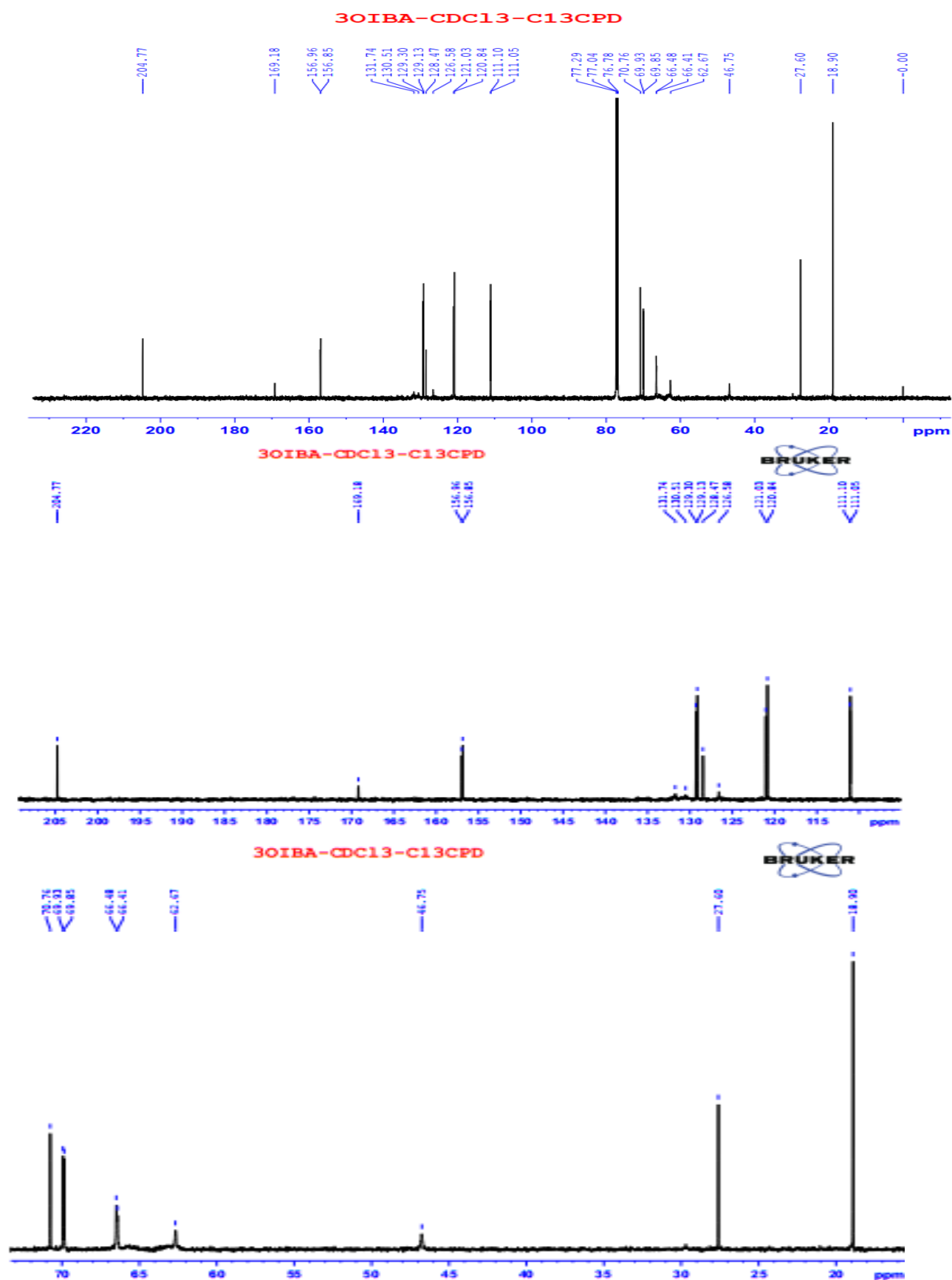


Fig S9. ¹³C-NMR of 5c

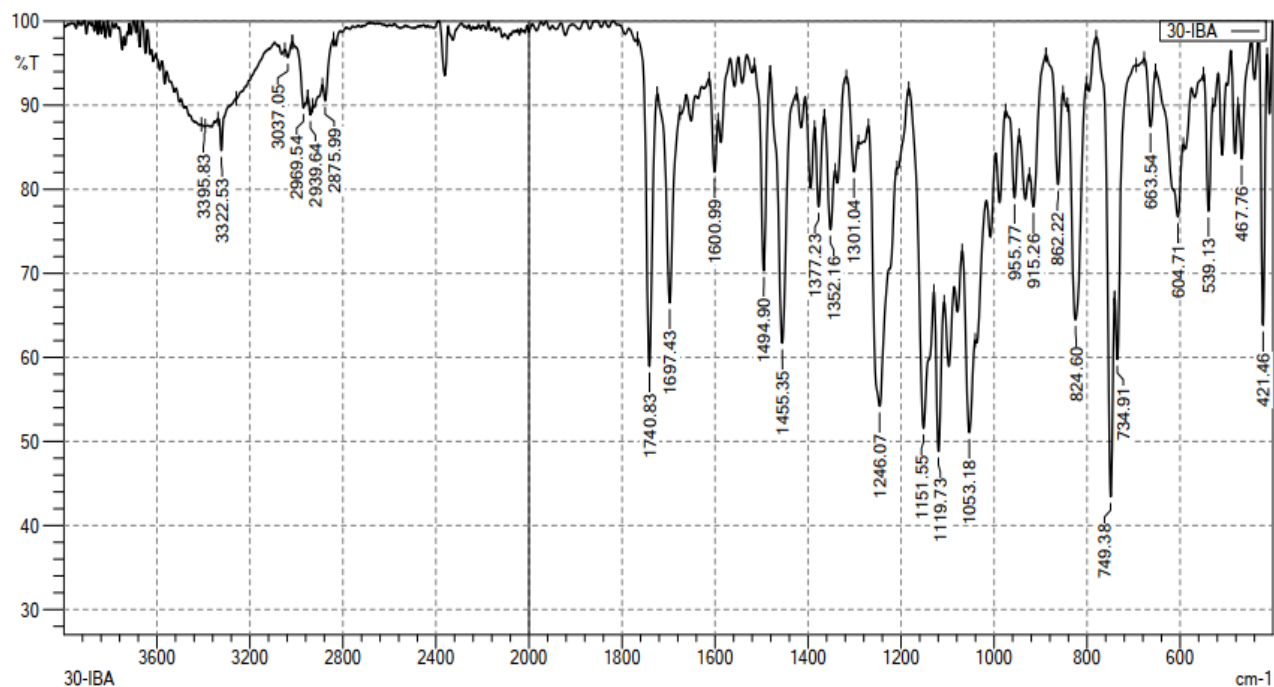
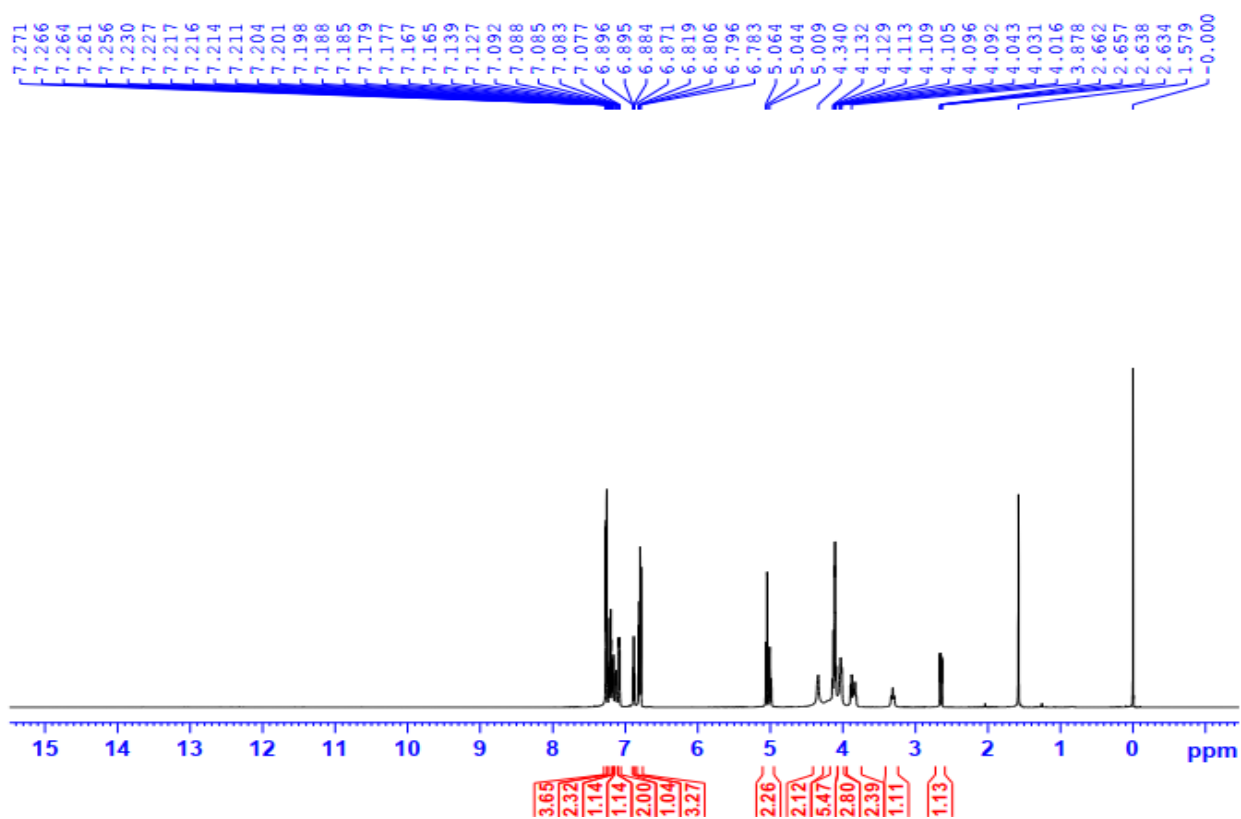


Fig S10. IR of 5c

4.4. Compound 5d

30OPhe-CDCl₃-1H



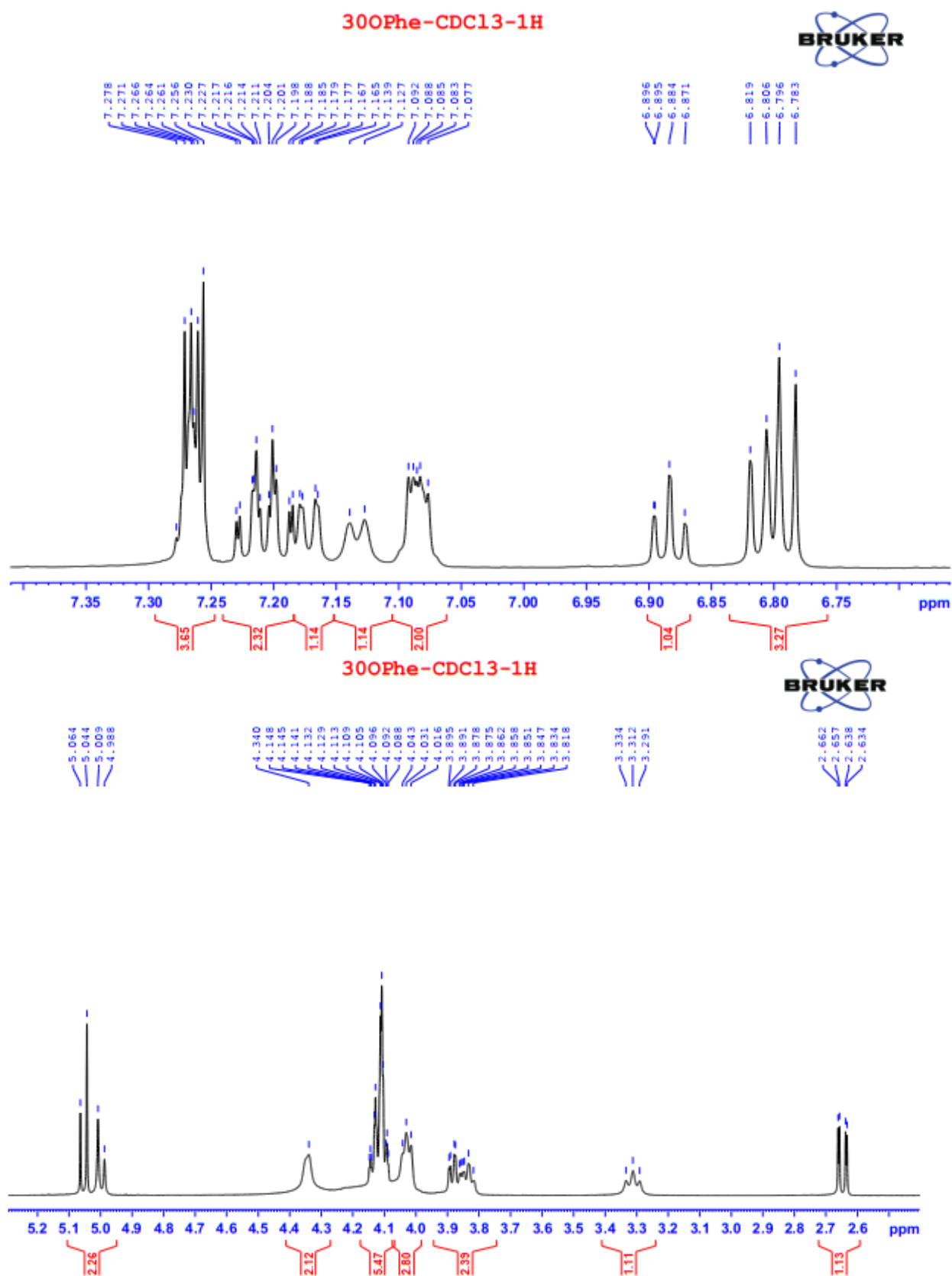


Fig S11. ¹H-NMR of **5d**

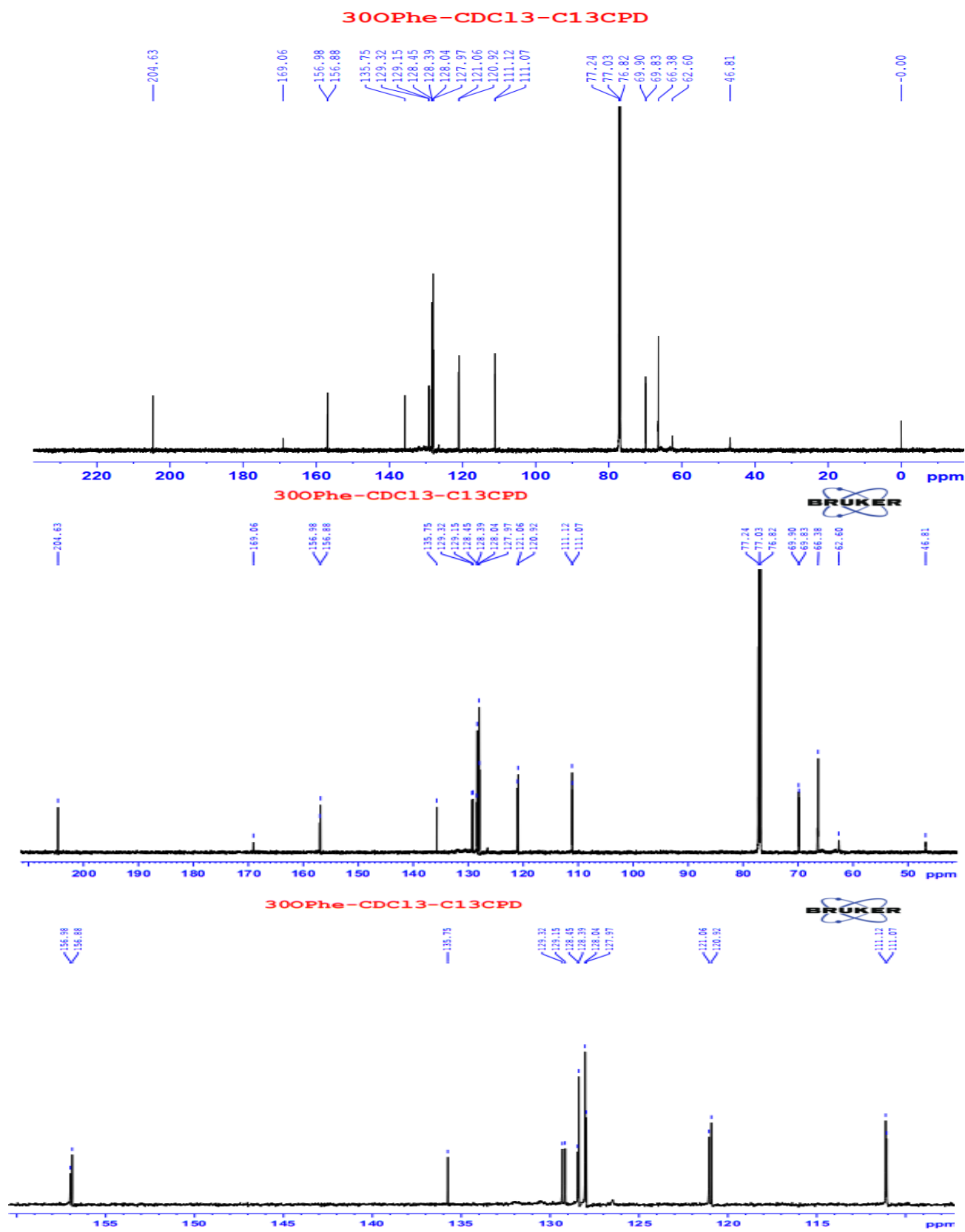


Fig S12. ¹³C-NMR of 5d

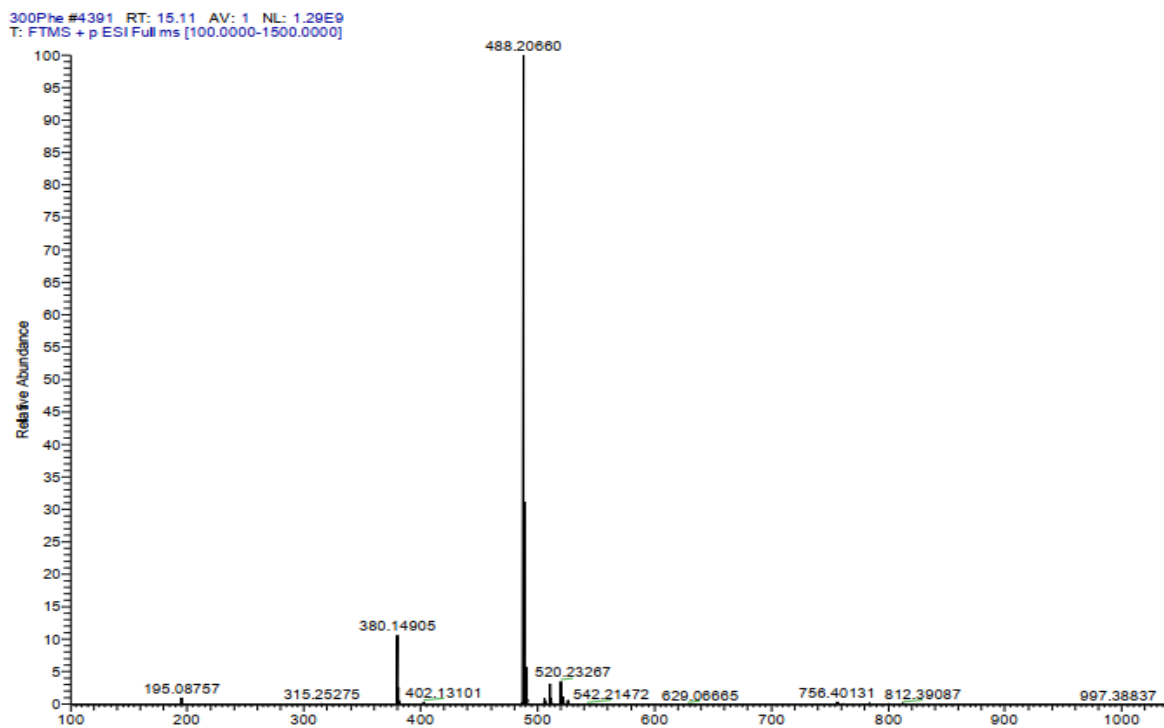


Fig S13. HRMS of 5d

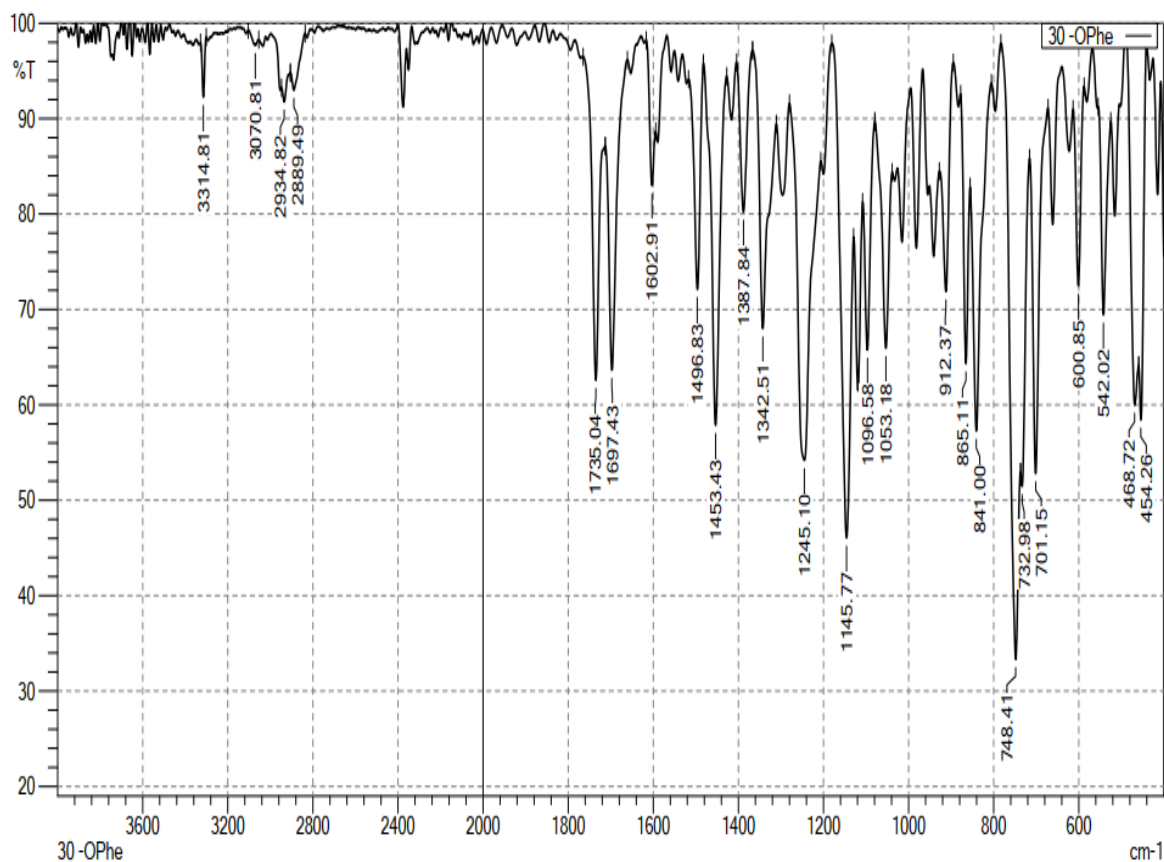


Fig S14. IR of 5d

4.5. Compound 5e

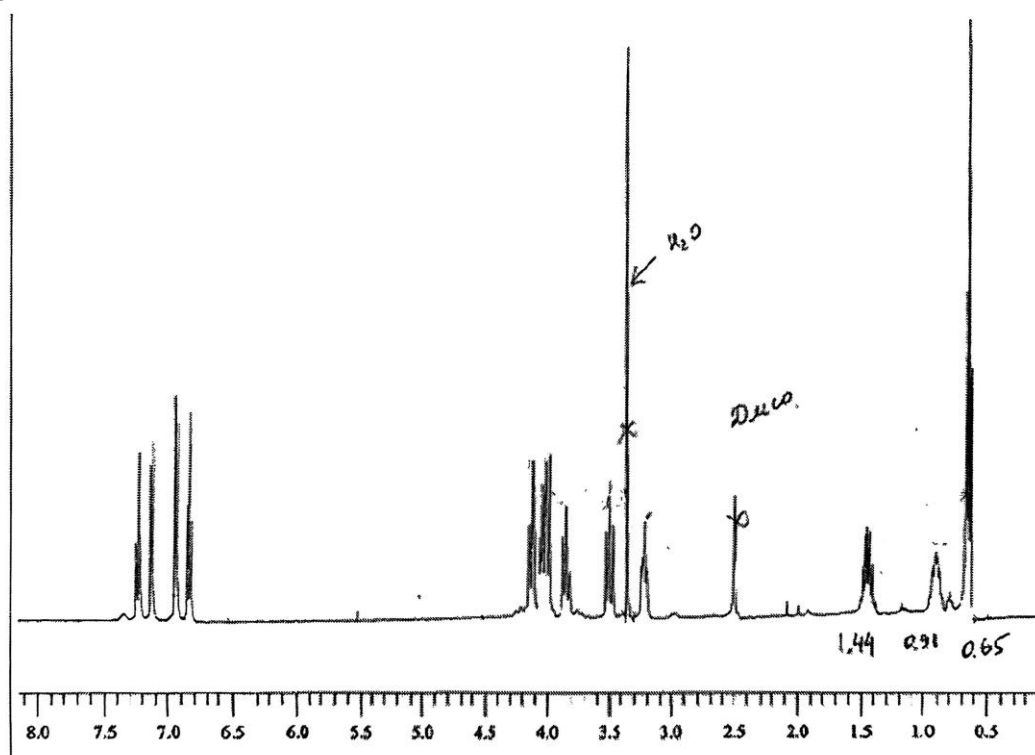


Fig S15. $^1\text{H-NMR}$ of 5e

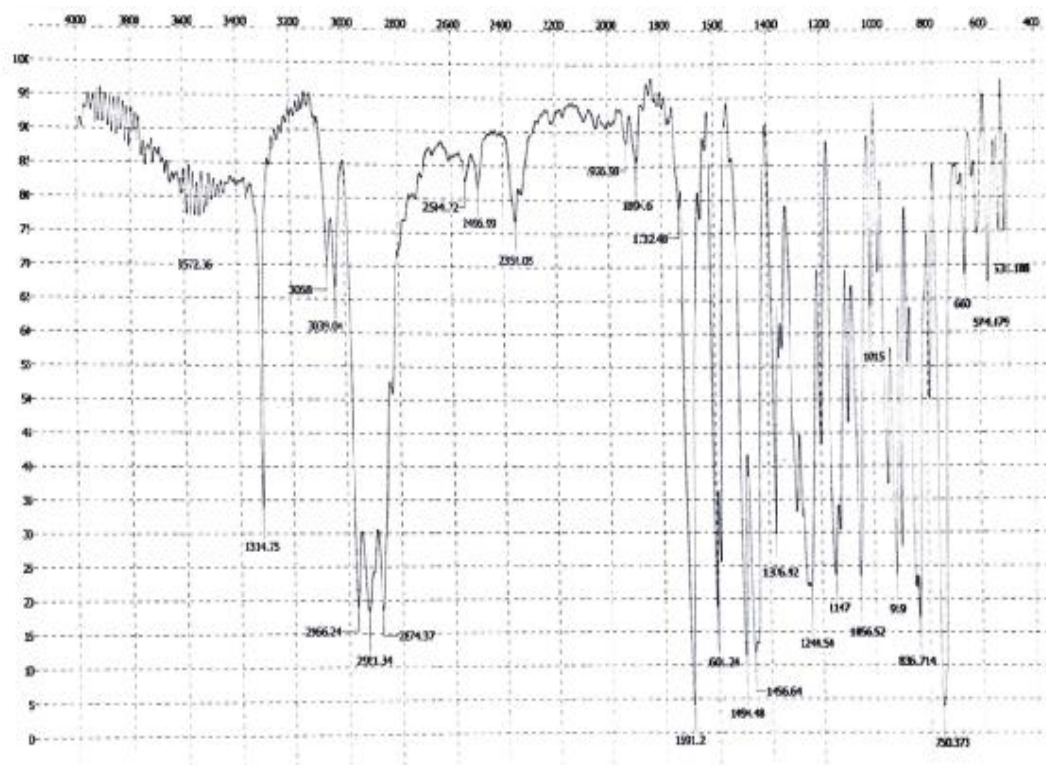


Fig S16. IR of 5e

4.6. Compound 5g

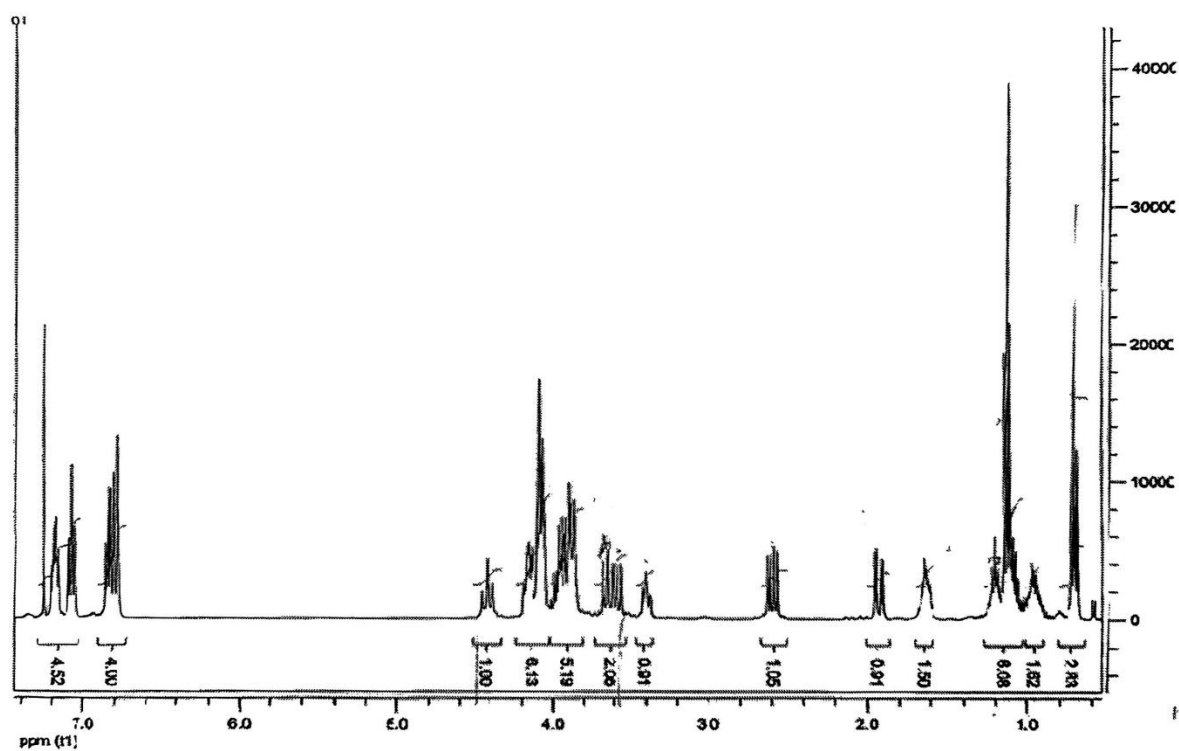


Fig S17. ¹H-NMR of 5g

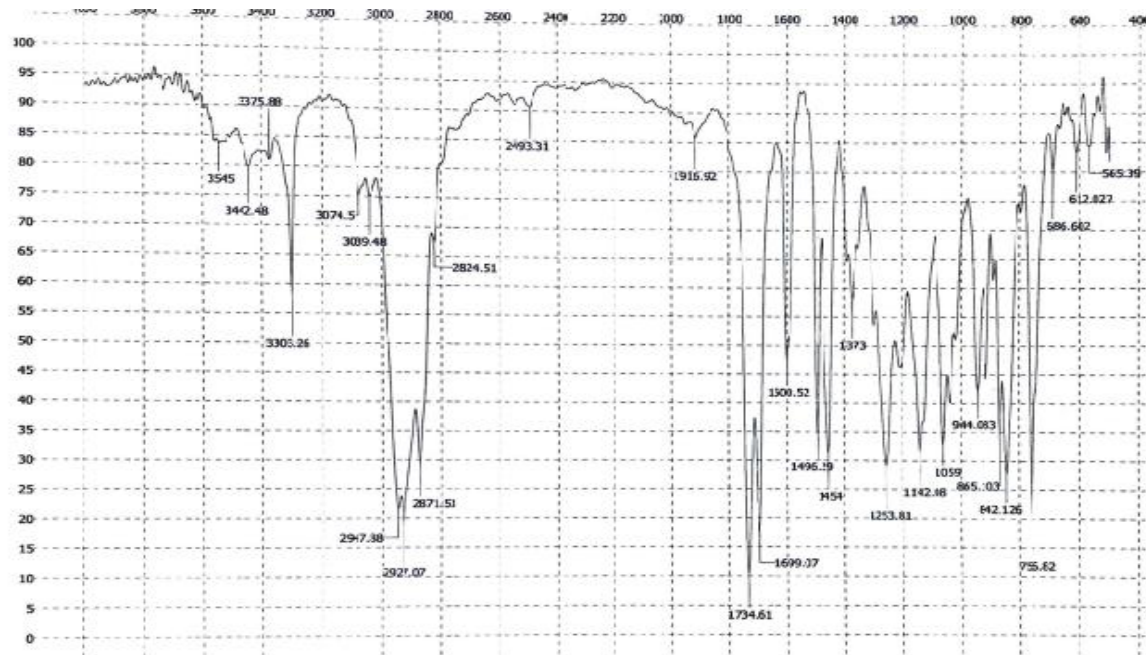


Fig S18. IR of 5g

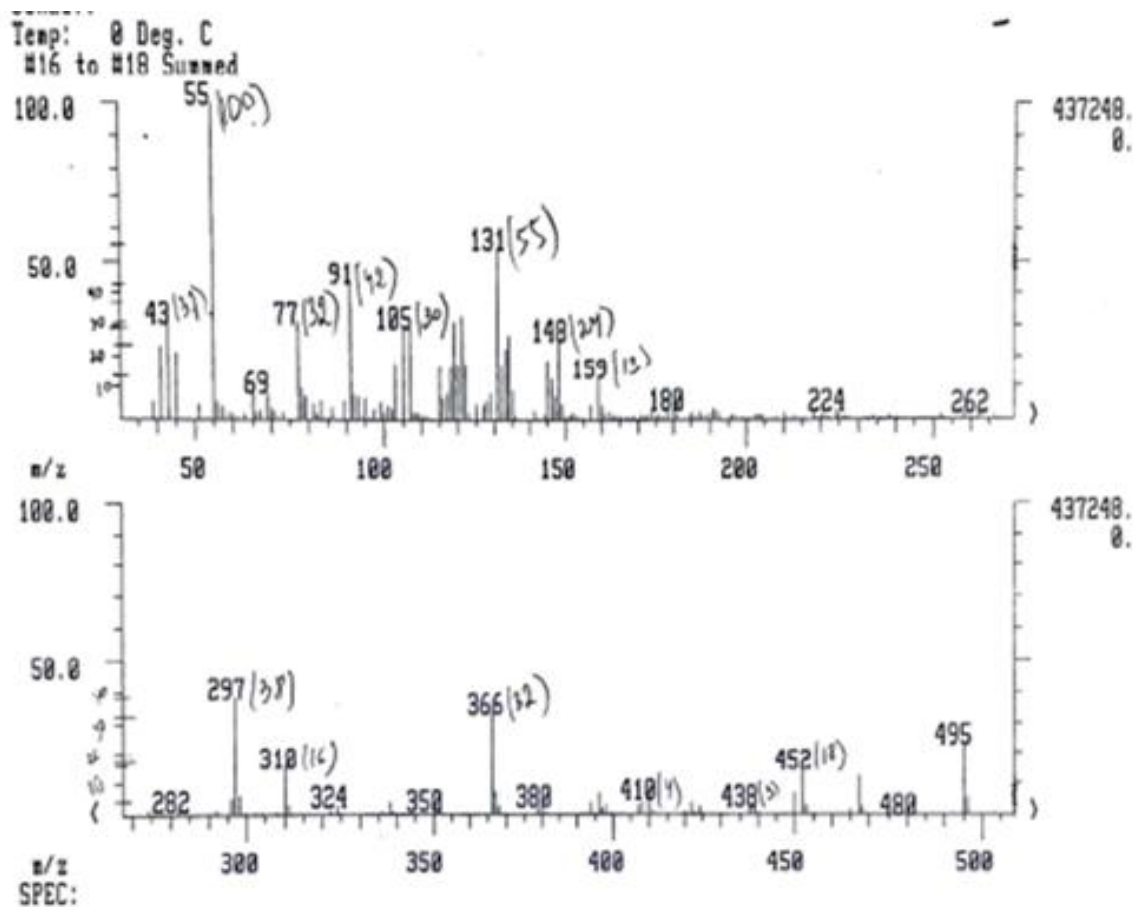


Fig S19. ESI-MS of 5g

4.7. Compound 5h

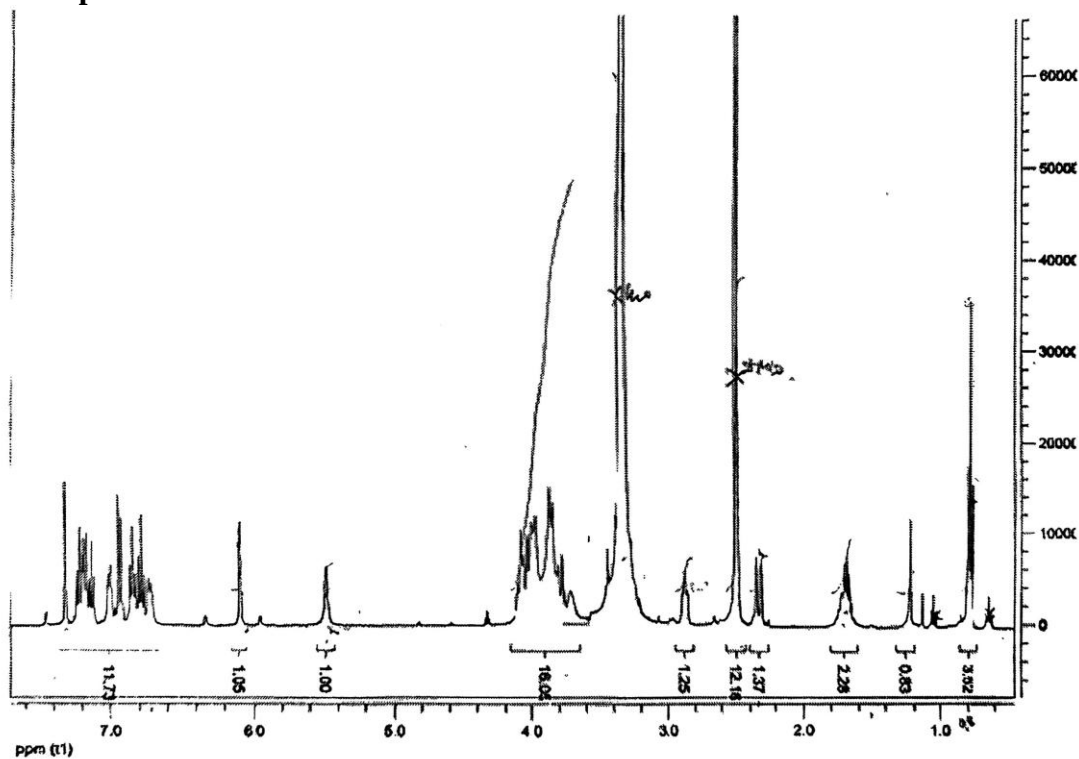


Fig S20. ^1H -NMR of 5h

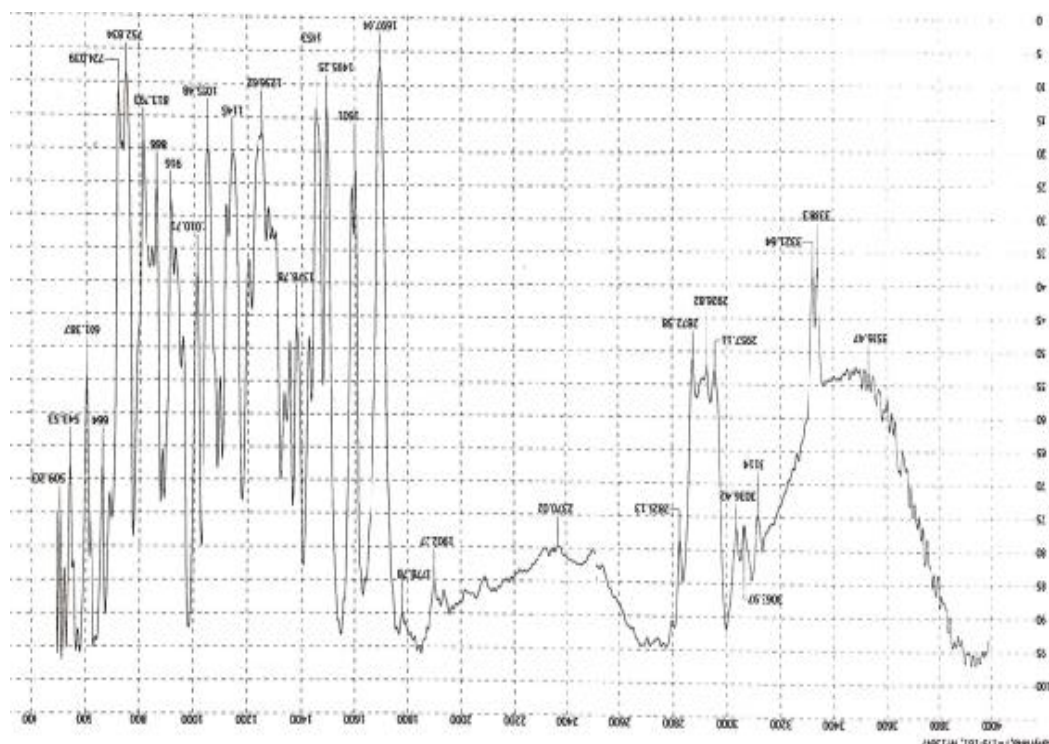


Fig S21. IR of 5h

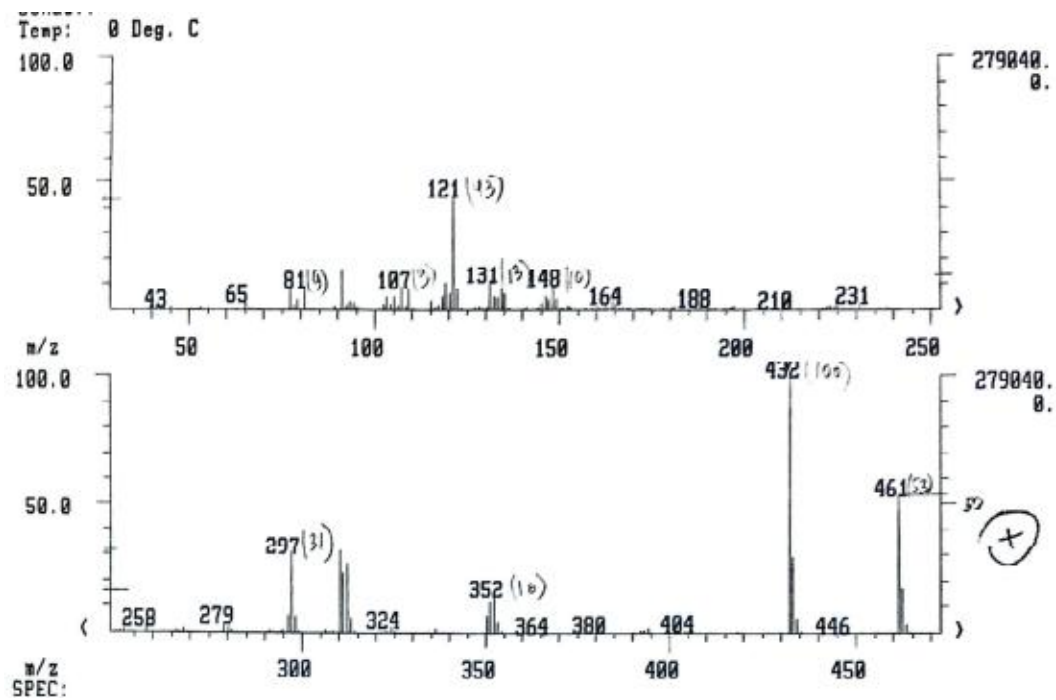


Fig S22. ESI - MS of 5h

4.8. Compound 6

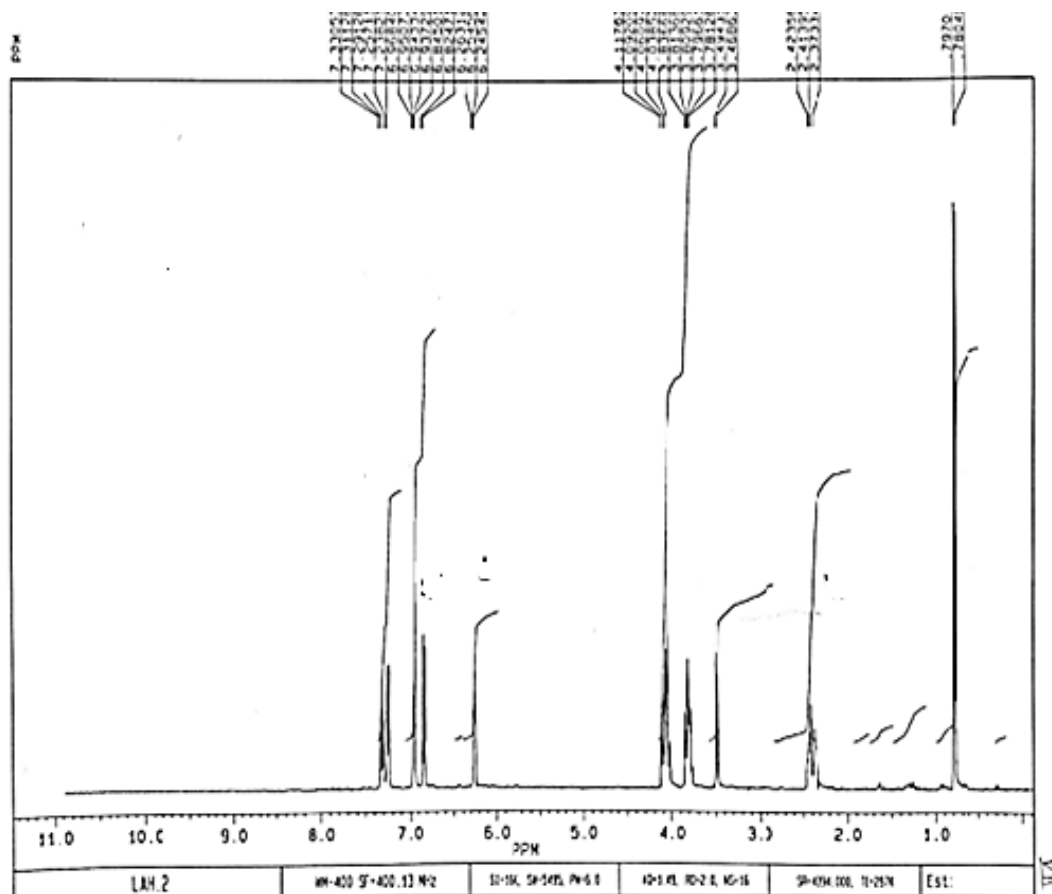


Fig S23. ¹H-NMR of 6

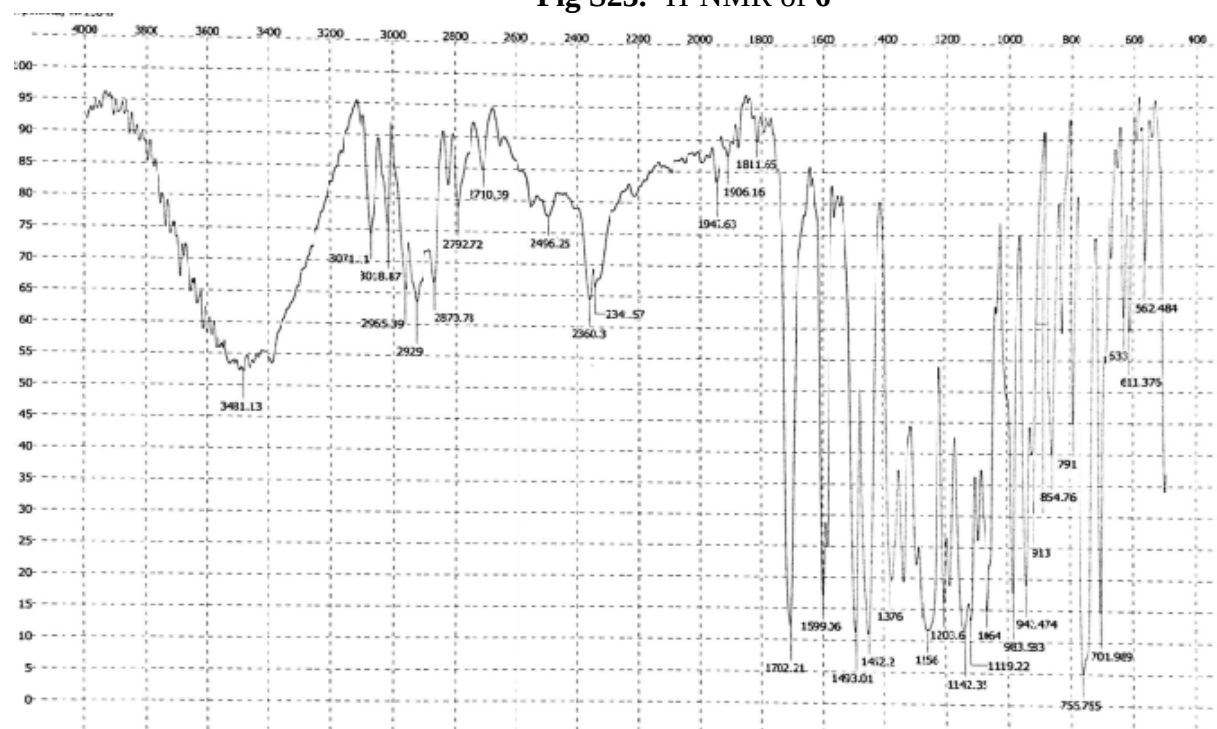


Fig S24. IR of 6

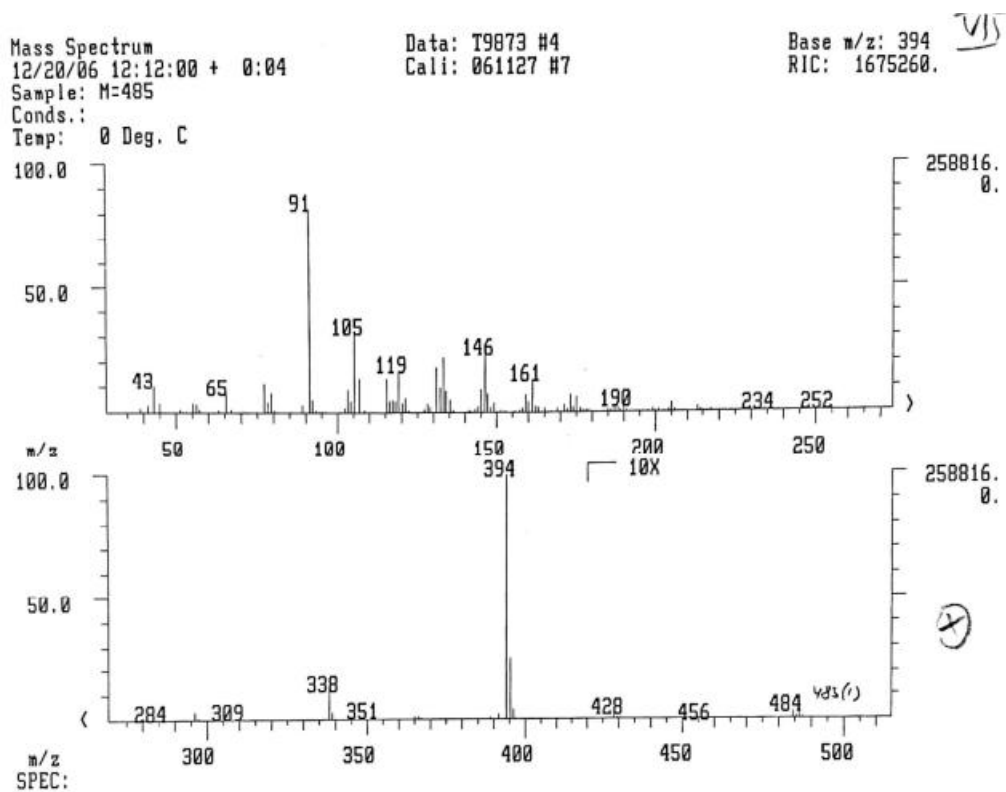


Fig S25. ESI-MS of **6**

5. Single X-ray diffraction of compounds **5c** and **7**

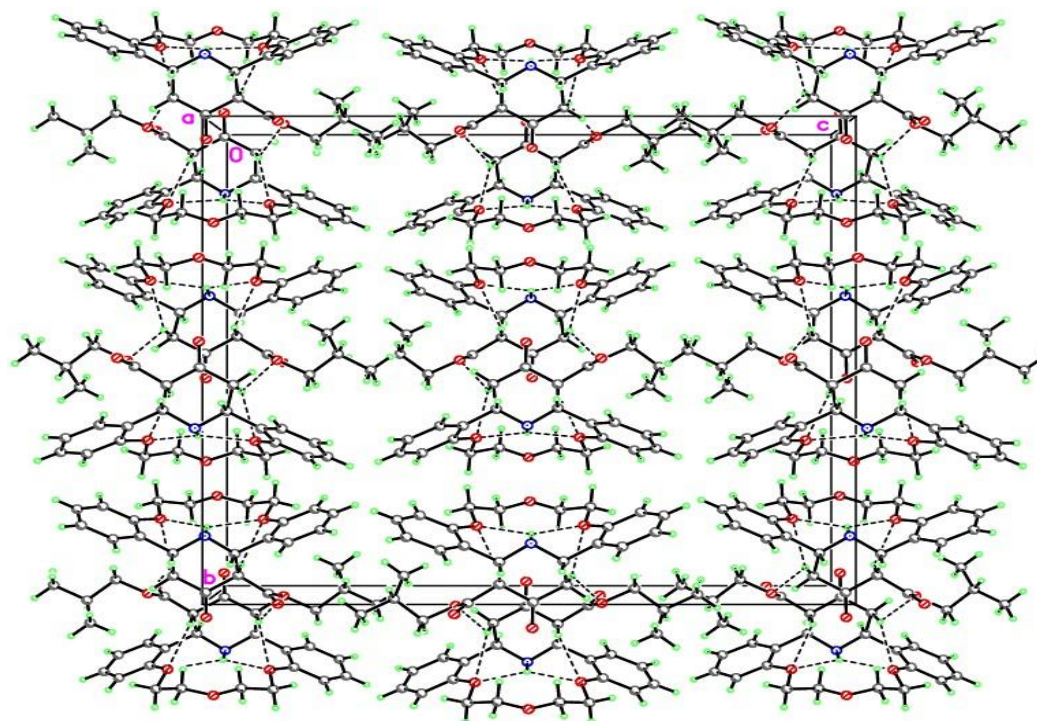


Fig S26. Crystal packing of **5c** along the crystallographic *a* axis.

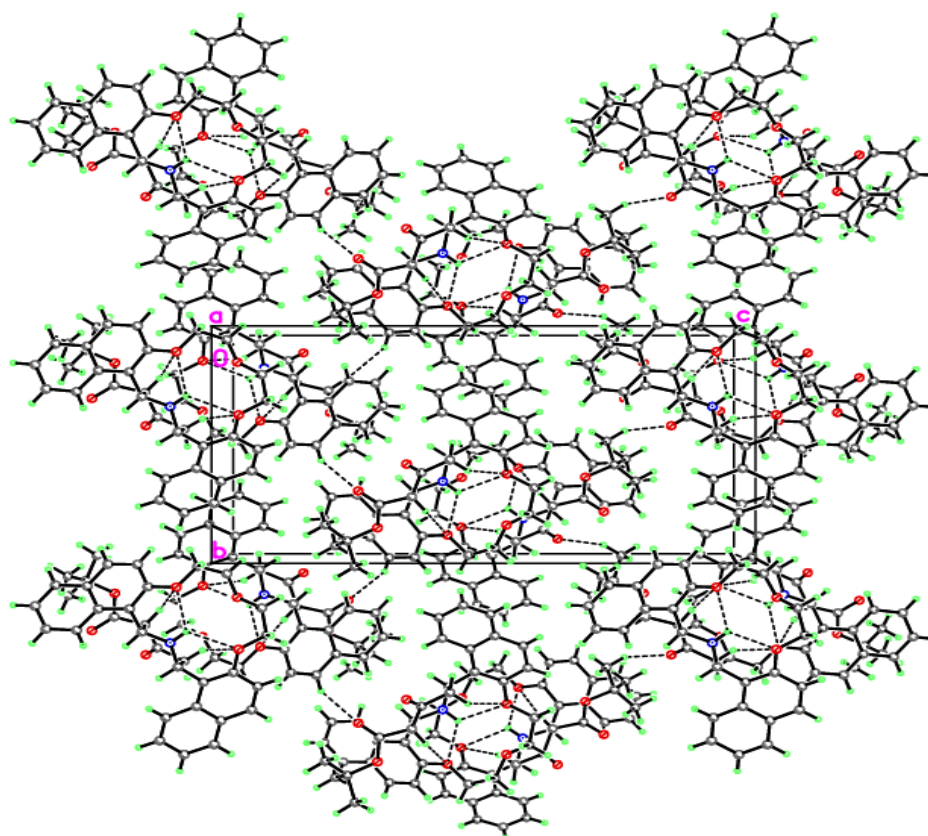


Figure S27. Crystal packing of **7** along the crystallographic **a** axis.

Table S2. Hydrogen bonds for **5c** and **7** [Å and °].

D*—H···A*	d(D—H)	d(H···A)	d(D···A)	∠DHA
Compound 5c				
C22—H22B···O14	0.99	2.41	3.002(3)	118
C24—H24A···O27 ^a	0.99	2.50	3.123(3)	121
C24—H24C···O8	1.00	2.38	3.055(3)	124
N25—H25···O8	0.82(3)	2.41(3)	2.942(3)	123(2)
N25—H25···O14	0.82(3)	2.47(3)	2.979(3)	121(2)
Compound 7				
C21—H21···O33 ^b	0.95	2.45	3.309(2)	150
C30—H30···O18	1.00	2.42	3.022(2)	118
C32—H32A···O12	0.99	2.31	2.928(2)	119
N33—H33···O12	0.90(2)	2.52(2)	3.065(2)	119(2)
N33—H33···O18	0.90(2)	2.33(2)	2.870(2)	118(2)
C37—H37B···O31 ^c	0.98	2.47	3.309(2)	144

* D – proton donor; A – proton acceptor;

Symmetry transformations used to generate equivalent atoms:

^a -x+1, -y+1, -z+1; ^b 1-x, y+½, -z+½; ^c 2-x, y+½, -z+½

Crystal data of compound 5c

$C_{26}H_{31}NO_6$	$D_x = 1.319 \text{ Mg m}^{-3}$
$M_r = 453.52$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
Orthorhombic, $Pbca$	Cell parameters from 17452 reflections
$a = 7.82055 (11) \text{ \AA}$	$\theta = 3.8\text{--}77.2^\circ$
$b = 23.2953 (2) \text{ \AA}$	$\mu = 0.76 \text{ mm}^{-1}$
$c = 25.0755 (5) \text{ \AA}$	$T = 100 \text{ K}$
$V = 4568.31 (12) \text{ \AA}^3$	Prism, colourless
$Z = 8$	$0.22 \times 0.15 \times 0.11 \text{ mm}$
$F(000) = 1936$	

Data collection

Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector diffractometer	4364 reflections with $I > 2\sigma(I)$
Radiation source: micro-focus sealed X-ray tube	$R_{\text{int}} = 0.062$
ϕ and ω scans	$\theta_{\text{max}} = 77.6^\circ$, $\theta_{\text{min}} = 3.5^\circ$
Absorption correction: multi-scan <i>CrysAlis PRO</i> 1.171.41.117a (Rigaku Oxford Diffraction, 2021) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.650$, $T_{\text{max}} = 1.000$	$k = -20 \rightarrow 29$
30624 measured reflections	$l = -31 \rightarrow 31$
4752 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: difference Fourier map
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.093$	Hydrogen site location: mixed
$wR(F^2) = 0.185$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 1P]$ where $P = (F_o^2 + 2F_c^2)/3$
4752 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
328 parameters	$\Delta_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
5 restraints	$\Delta_{\text{min}} = -0.36 \text{ e \AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$) for **5c**

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.8692 (3)	0.60114 (10)	0.44895 (9)	0.0252 (5)	
H1	0.9873	0.5849	0.4517	0.030*	
C2	0.8642 (4)	0.63578 (11)	0.39753 (11)	0.0369 (7)	
C3	1.0045 (6)	0.63438 (13)	0.36336 (13)	0.0537 (10)	
H3	1.1046	0.6141	0.3736	0.064*	
C4	0.9998 (8)	0.66242 (17)	0.31431 (15)	0.0839 (18)	
H4	1.0959	0.6613	0.2911	0.101*	
C5	0.8547 (9)	0.69157 (18)	0.29994 (16)	0.0863 (18)	
H5	0.8514	0.7106	0.2664	0.104*	
C6	0.7160 (7)	0.69406 (16)	0.33216 (17)	0.0711 (14)	
H6	0.6175	0.7150	0.3215	0.085*	
C7	0.7191 (5)	0.66549 (13)	0.38141 (13)	0.0499 (9)	
O8	0.5838 (3)	0.66443 (9)	0.41618 (11)	0.0573 (7)	
C9	0.4349 (5)	0.69824 (15)	0.4057 (2)	0.0832 (17)	
H9A	0.3807	0.6860	0.3719	0.100*	
H9B	0.4657	0.7393	0.4028	0.100*	
C10	0.3141 (4)	0.68886 (16)	0.4519 (2)	0.0874 (19)	
H10A	0.2063	0.7102	0.4458	0.105*	
H10B	0.2866	0.6475	0.4552	0.105*	
O11	0.3932 (3)	0.70845 (9)	0.49885 (15)	0.0778 (11)	
C12	0.3131 (4)	0.68936 (15)	0.5458 (2)	0.0836 (18)	
H12A	0.2823	0.6483	0.5424	0.100*	
H12B	0.2070	0.7116	0.5521	0.100*	
C13	0.4345 (4)	0.69753 (14)	0.5916 (2)	0.0772 (16)	
H13A	0.4674	0.7384	0.5949	0.093*	
H13B	0.3810	0.6850	0.6254	0.093*	
O14	0.5807 (3)	0.66307 (9)	0.57981 (11)	0.0540 (7)	
C15	0.7158 (5)	0.66181 (13)	0.61455 (13)	0.0468 (8)	
C16	0.7152 (6)	0.68825 (15)	0.66475 (16)	0.0665 (13)	
H16	0.6189	0.7096	0.6765	0.080*	
C17	0.8558 (8)	0.68258 (17)	0.69621 (15)	0.0753 (15)	
H17	0.8550	0.6999	0.7305	0.090*	
C18	0.9995 (7)	0.65282 (17)	0.68044 (15)	0.0717 (14)	
H18	1.0964	0.6500	0.7031	0.086*	
C19	0.9988 (5)	0.62715 (13)	0.63050 (12)	0.0489 (9)	
H19	1.0964	0.6063	0.6191	0.059*	

C20	0.8576 (4)	0.63136 (11)	0.59678 (11)	0.0349 (6)	
C21	0.8595 (3)	0.59923 (10)	0.54441 (9)	0.0237 (5)	
H21	0.9741	0.5804	0.5417	0.028*	
C22	0.7234 (3)	0.54956 (10)	0.54471 (10)	0.0262 (5)	
H22	0.6073	0.5676	0.5448	0.031*	0.85
H22A	0.7416	0.5247	0.5762	0.031*	0.15
H22B	0.6070	0.5660	0.5470	0.031*	0.15
C23	0.7399 (3)	0.51446 (10)	0.49440 (10)	0.0244 (5)	
O23	0.7449 (2)	0.46226 (7)	0.49538 (7)	0.0287 (4)	
C24	0.7458 (4)	0.54892 (10)	0.44394 (11)	0.0309 (6)	
H24A	0.6294	0.5629	0.4354	0.037*	0.85
H24B	0.7841	0.5240	0.4143	0.037*	0.85
H24C	0.6305	0.5675	0.4431	0.037*	0.15
N25	0.8417 (3)	0.63542 (8)	0.49722 (8)	0.0222 (4)	
H25	0.747 (4)	0.6509 (13)	0.4967 (11)	0.027*	
C26	0.7359 (4)	0.51235 (11)	0.59214 (11)	0.0226 (5)	0.85
O26	0.8681 (3)	0.49559 (9)	0.61128 (8)	0.0311 (5)	0.85
O27	0.5802 (3)	0.50050 (9)	0.61145 (8)	0.0269 (4)	0.85
C27	0.5681 (4)	0.47041 (13)	0.66191 (11)	0.0284 (6)	0.85
H27A	0.6748	0.4487	0.6685	0.034*	0.85
H27B	0.4720	0.4428	0.6607	0.034*	0.85
C28	0.5397 (4)	0.51302 (14)	0.70636 (12)	0.0295 (6)	0.85
H28A	0.6480	0.5351	0.7116	0.035*	0.85
C29	0.3978 (5)	0.55546 (16)	0.69353 (15)	0.0431 (8)	0.85
H29A	0.4261	0.5762	0.6607	0.065*	0.85
H29B	0.3858	0.5828	0.7230	0.065*	0.85
H29C	0.2899	0.5347	0.6887	0.065*	0.85
C30	0.5027 (4)	0.47986 (17)	0.75757 (13)	0.0406 (8)	0.85
H30A	0.5966	0.4531	0.7647	0.061*	0.85
H30B	0.3957	0.4584	0.7535	0.061*	0.85
H30C	0.4920	0.5068	0.7874	0.061*	0.85
C26'	0.7370 (17)	0.5135 (8)	0.4071 (7)	0.0360 (13)	0.15
O26'	0.8703 (14)	0.4967 (6)	0.3892 (5)	0.0360 (13)	0.15
O27'	0.5840 (16)	0.5024 (6)	0.3875 (5)	0.0360 (13)	0.15
C27'	0.569 (2)	0.4716 (6)	0.3384 (5)	0.0360 (13)	0.15
H27C	0.4714	0.4447	0.3402	0.043*	0.15
H27D	0.6744	0.4493	0.3316	0.043*	0.15
C28'	0.541 (2)	0.5152 (6)	0.2937 (5)	0.0360 (13)	0.15
H28B	0.6515	0.5356	0.2873	0.043*	0.15

C29'	0.404 (2)	0.5606 (7)	0.3048 (8)	0.0360 (13)	0.15
H29D	0.3873	0.5844	0.2730	0.054*	0.15
H29E	0.4407	0.5849	0.3346	0.054*	0.15
H29F	0.2961	0.5416	0.3141	0.054*	0.15
C30'	0.505 (2)	0.4768 (8)	0.2454 (7)	0.0360 (13)	0.15
H30D	0.5967	0.4483	0.2419	0.054*	0.15
H30E	0.5005	0.5004	0.2131	0.054*	0.15
H30F	0.3956	0.4571	0.2503	0.054*	0.15

Atomic displacement parameters ($\text{\AA}^2$) for 5c

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0297 (12)	0.0198 (10)	0.0263 (12)	-0.0033 (9)	-0.0042 (10)	-0.0011 (9)
C2	0.0560 (18)	0.0234 (12)	0.0313 (13)	-0.0118 (12)	-0.0092 (13)	0.0005 (10)
C3	0.091 (3)	0.0310 (14)	0.0393 (16)	-0.0130 (17)	0.0198 (18)	-0.0058 (13)
C4	0.170 (6)	0.047 (2)	0.0351 (18)	-0.032 (3)	0.027 (3)	-0.0018 (16)
C5	0.171 (6)	0.048 (2)	0.040 (2)	-0.048 (3)	-0.023 (3)	0.0102 (17)
C6	0.106 (4)	0.0471 (19)	0.060 (2)	-0.032 (2)	-0.052 (3)	0.0223 (18)
C7	0.065 (2)	0.0321 (14)	0.0522 (18)	-0.0237 (15)	-0.0297 (17)	0.0154 (13)
O8	0.0396 (12)	0.0378 (11)	0.0944 (19)	-0.0041 (10)	-0.0331 (13)	0.0278 (12)
C9	0.042 (2)	0.0434 (19)	0.164 (5)	-0.0160 (16)	-0.056 (3)	0.050 (2)
C10	0.0259 (17)	0.0439 (19)	0.192 (6)	-0.0019 (15)	-0.036 (3)	0.036 (3)
O11	0.0254 (11)	0.0261 (11)	0.182 (3)	-0.0037 (9)	-0.0011 (17)	0.0037 (15)
C12	0.0245 (16)	0.0370 (17)	0.189 (6)	0.0014 (14)	0.030 (3)	-0.024 (3)
C13	0.0382 (19)	0.0340 (16)	0.160 (5)	-0.0111 (14)	0.052 (2)	-0.038 (2)
O14	0.0374 (12)	0.0336 (11)	0.0909 (18)	-0.0003 (9)	0.0304 (13)	-0.0241 (11)
C15	0.059 (2)	0.0309 (14)	0.0502 (18)	-0.0204 (14)	0.0263 (16)	-0.0156 (13)
C16	0.097 (3)	0.0422 (18)	0.060 (2)	-0.024 (2)	0.049 (2)	-0.0154 (17)
C17	0.140 (5)	0.049 (2)	0.0376 (18)	-0.042 (3)	0.013 (3)	-0.0106 (16)
C18	0.123 (4)	0.055 (2)	0.0366 (18)	-0.026 (3)	-0.020 (2)	0.0014 (16)
C19	0.076 (2)	0.0390 (15)	0.0318 (15)	-0.0167 (16)	-0.0118 (15)	0.0032 (12)
C20	0.0484 (16)	0.0250 (12)	0.0313 (13)	-0.0112 (12)	0.0094 (12)	-0.0016 (10)
C21	0.0236 (11)	0.0216 (11)	0.0258 (12)	-0.0036 (9)	0.0038 (9)	-0.0009 (9)
C22	0.0227 (12)	0.0210 (11)	0.0350 (13)	-0.0022 (9)	0.0082 (10)	-0.0026 (9)
C23	0.0165 (10)	0.0187 (10)	0.0380 (13)	-0.0012 (8)	-0.0012 (10)	-0.0012 (9)
O23	0.0322 (9)	0.0175 (7)	0.0364 (9)	-0.0006 (7)	-0.0003 (8)	-0.0003 (7)
C24	0.0357 (14)	0.0225 (11)	0.0345 (13)	-0.0041 (10)	-0.0126 (11)	0.0003 (10)
N25	0.0190 (9)	0.0179 (9)	0.0299 (10)	0.0007 (8)	0.0006 (8)	-0.0004 (8)
C26	0.0226 (13)	0.0220 (12)	0.0233 (13)	-0.0009 (10)	0.0025 (11)	-0.0035 (10)

O26	0.0263 (11)	0.0363 (11)	0.0308 (11)	0.0033 (9)	-0.0015 (9)	0.0066 (9)
O27	0.0263 (10)	0.0324 (10)	0.0220 (9)	-0.0049 (8)	0.0016 (8)	0.0049 (8)
C27	0.0328 (15)	0.0312 (14)	0.0212 (13)	-0.0054 (12)	0.0026 (12)	0.0058 (11)
C28	0.0251 (14)	0.0366 (16)	0.0268 (15)	-0.0023 (13)	0.0017 (12)	0.0043 (12)
C29	0.0406 (19)	0.0452 (19)	0.0436 (19)	0.0046 (16)	0.0079 (16)	0.0027 (16)
C30	0.0266 (16)	0.069 (2)	0.0263 (16)	-0.0014 (16)	0.0043 (13)	0.0095 (16)
C26'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)
O26'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)
O27'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)
C27'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)
C28'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)
C29'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)
C30'	0.029 (3)	0.040 (3)	0.039 (3)	0.000 (3)	-0.003 (3)	-0.004 (3)

Geometric parameters (Å, °) for 5c

C1—N25	1.466 (3)	C21—H21	1.0000
C1—C2	1.521 (3)	C22—C26	1.475 (4)
C1—C24	1.558 (3)	C22—C23	1.509 (3)
C1—H1	1.0000	C22—H22	1.0000
C2—C7	1.390 (5)	C22—H22A	0.9900
C2—C3	1.393 (5)	C22—H22B	0.9900
C3—C4	1.393 (5)	C23—O23	1.217 (3)
C3—H3	0.9500	C23—C24	1.499 (3)
C4—C5	1.371 (8)	C24—C26'	1.240 (17)
C4—H4	0.9500	C24—H24A	0.9900
C5—C6	1.354 (7)	C24—H24B	0.9900
C5—H5	0.9500	C24—H24C	1.0000
C6—C7	1.403 (5)	N25—H25	0.82 (3)
C6—H6	0.9500	C26—O26	1.205 (3)
C7—O8	1.371 (5)	C26—O27	1.339 (3)
O8—C9	1.430 (4)	O27—C27	1.450 (3)
C9—C10	1.511 (7)	C27—C28	1.509 (4)
C9—H9A	0.9900	C27—H27A	0.9900
C9—H9B	0.9900	C27—H27B	0.9900
C10—O11	1.405 (6)	C28—C29	1.521 (5)
C10—H10A	0.9900	C28—C30	1.526 (4)
C10—H10B	0.9900	C28—H28A	1.0000
O11—C12	1.405 (6)	C29—H29A	0.9800

C12—C13	1.503 (7)	C29—H29B	0.9800
C12—H12A	0.9900	C29—H29C	0.9800
C12—H12B	0.9900	C30—H30A	0.9800
C13—O14	1.428 (4)	C30—H30B	0.9800
C13—H13A	0.9900	C30—H30C	0.9800
C13—H13B	0.9900	C26'—O26'	1.201 (3)
O14—C15	1.370 (4)	C26'—O27'	1.32 (2)
C15—C20	1.390 (4)	O27'—C27'	1.429 (3)
C15—C16	1.401 (5)	C27'—C28'	1.530 (3)
C16—C17	1.360 (7)	C27'—H27C	0.9900
C16—H16	0.9500	C27'—H27D	0.9900
C17—C18	1.378 (7)	C28'—C29'	1.530 (3)
C17—H17	0.9500	C28'—C30'	1.531 (3)
C18—C19	1.388 (5)	C28'—H28B	1.0000
C18—H18	0.9500	C29'—H29D	0.9800
C19—C20	1.394 (5)	C29'—H29E	0.9800
C19—H19	0.9500	C29'—H29F	0.9800
C20—C21	1.512 (3)	C30'—H30D	0.9800
C21—N25	1.459 (3)	C30'—H30E	0.9800
C21—C22	1.572 (3)	C30'—H30F	0.9800
N25—C1—C2	114.02 (19)	C21—C22—H22	107.8
N25—C1—C24	113.6 (2)	C23—C22—H22A	109.7
C2—C1—C24	109.3 (2)	C21—C22—H22A	109.7
N25—C1—H1	106.4	C23—C22—H22B	109.7
C2—C1—H1	106.4	C21—C22—H22B	109.7
C24—C1—H1	106.4	H22A—C22—H22B	108.2
C7—C2—C3	118.4 (3)	O23—C23—C24	123.4 (2)
C7—C2—C1	122.1 (3)	O23—C23—C22	121.8 (2)
C3—C2—C1	119.3 (3)	C24—C23—C22	114.71 (19)
C4—C3—C2	120.8 (4)	C26'—C24—C23	105.7 (9)
C4—C3—H3	119.6	C26'—C24—C1	127.8 (8)
C2—C3—H3	119.6	C23—C24—C1	111.7 (2)
C5—C4—C3	119.1 (5)	C23—C24—H24A	109.3
C5—C4—H4	120.5	C1—C24—H24A	109.3
C3—C4—H4	120.5	C23—C24—H24B	109.3
C6—C5—C4	121.8 (4)	C1—C24—H24B	109.3
C6—C5—H5	119.1	H24A—C24—H24B	107.9
C4—C5—H5	119.1	C26'—C24—H24C	102.8

C5—C6—C7	119.4 (4)	C23—C24—H24C	102.8
C5—C6—H6	120.3	C1—C24—H24C	102.8
C7—C6—H6	120.3	C21—N25—C1	109.93 (18)
O8—C7—C2	115.9 (3)	C21—N25—H25	111 (2)
O8—C7—C6	123.7 (4)	C1—N25—H25	111 (2)
C2—C7—C6	120.4 (4)	O26—C26—O27	124.7 (2)
C7—O8—C9	120.1 (3)	O26—C26—C22	124.7 (3)
O8—C9—C10	106.8 (3)	O27—C26—C22	110.6 (2)
O8—C9—H9A	110.4	C26—O27—C27	118.3 (2)
C10—C9—H9A	110.4	O27—C27—C28	109.6 (2)
O8—C9—H9B	110.4	O27—C27—H27A	109.7
C10—C9—H9B	110.4	C28—C27—H27A	109.7
H9A—C9—H9B	108.6	O27—C27—H27B	109.7
O11—C10—C9	108.7 (3)	C28—C27—H27B	109.7
O11—C10—H10A	110.0	H27A—C27—H27B	108.2
C9—C10—H10A	110.0	C27—C28—C29	112.3 (3)
O11—C10—H10B	110.0	C27—C28—C30	108.4 (3)
C9—C10—H10B	110.0	C29—C28—C30	111.6 (3)
H10A—C10—H10B	108.3	C27—C28—H28A	108.1
C10—O11—C12	113.7 (3)	C29—C28—H28A	108.1
O11—C12—C13	108.6 (3)	C30—C28—H28A	108.1
O11—C12—H12A	110.0	C28—C29—H29A	109.5
C13—C12—H12A	110.0	C28—C29—H29B	109.5
O11—C12—H12B	110.0	H29A—C29—H29B	109.5
C13—C12—H12B	110.0	C28—C29—H29C	109.5
H12A—C12—H12B	108.4	H29A—C29—H29C	109.5
O14—C13—C12	106.1 (3)	H29B—C29—H29C	109.5
O14—C13—H13A	110.5	C28—C30—H30A	109.5
C12—C13—H13A	110.5	C28—C30—H30B	109.5
O14—C13—H13B	110.5	H30A—C30—H30B	109.5
C12—C13—H13B	110.5	C28—C30—H30C	109.5
H13A—C13—H13B	108.7	H30A—C30—H30C	109.5
C15—O14—C13	119.9 (3)	H30B—C30—H30C	109.5
O14—C15—C20	115.0 (3)	O26'—C26'—C24	116.6 (15)
O14—C15—C16	124.0 (3)	O26'—C26'—O27'	125.7 (16)
C20—C15—C16	121.0 (4)	C24—C26'—O27'	117.3 (10)
C17—C16—C15	118.4 (4)	C26'—O27'—C27'	119.6 (13)
C17—C16—H16	120.8	O27'—C27'—C28'	108.1 (11)
C15—C16—H16	120.8	O27'—C27'—H27C	110.1

C16—C17—C18	122.8 (3)	C28'—C27'—H27C	110.1
C16—C17—H17	118.6	O27'—C27'—H27D	110.1
C18—C17—H17	118.6	C28'—C27'—H27D	110.1
C17—C18—C19	118.2 (4)	H27C—C27'—H27D	108.4
C17—C18—H18	120.9	C27'—C28'—C29'	115.2 (14)
C19—C18—H18	120.9	C27'—C28'—C30'	102.6 (12)
C18—C19—C20	121.4 (4)	C29'—C28'—C30'	114.9 (14)
C18—C19—H19	119.3	C27'—C28'—H28B	107.9
C20—C19—H19	119.3	C29'—C28'—H28B	107.9
C15—C20—C19	118.3 (3)	C30'—C28'—H28B	107.9
C15—C20—C21	122.6 (3)	C28'—C29'—H29D	109.5
C19—C20—C21	119.0 (3)	C28'—C29'—H29E	109.5
N25—C21—C20	114.67 (19)	H29D—C29'—H29E	109.5
N25—C21—C22	111.4 (2)	C28'—C29'—H29F	109.5
C20—C21—C22	110.7 (2)	H29D—C29'—H29F	109.5
N25—C21—H21	106.5	H29E—C29'—H29F	109.5
C20—C21—H21	106.5	C28'—C30'—H30D	109.5
C22—C21—H21	106.5	C28'—C30'—H30E	109.5
C26—C22—C23	110.5 (2)	H30D—C30'—H30E	109.5
C26—C22—C21	113.0 (2)	C28'—C30'—H30F	109.5
C23—C22—C21	109.70 (19)	H30D—C30'—H30F	109.5
C26—C22—H22	107.8	H30E—C30'—H30F	109.5
C23—C22—H22	107.8		
N25—C1—C2—C7	-61.7 (3)	C19—C20—C21—C22	112.3 (3)
C24—C1—C2—C7	66.7 (3)	N25—C21—C22—C26	-179.4 (2)
N25—C1—C2—C3	123.3 (3)	C20—C21—C22—C26	-50.5 (3)
C24—C1—C2—C3	-108.3 (3)	N25—C21—C22—C23	56.8 (3)
C7—C2—C3—C4	0.4 (4)	C20—C21—C22—C23	-174.3 (2)
C1—C2—C3—C4	175.6 (3)	C26—C22—C23—O23	6.8 (3)
C2—C3—C4—C5	0.0 (5)	C21—C22—C23—O23	132.1 (2)
C3—C4—C5—C6	0.2 (6)	C26—C22—C23—C24	-174.4 (2)
C4—C5—C6—C7	-0.8 (6)	C21—C22—C23—C24	-49.1 (3)
C3—C2—C7—O8	178.7 (3)	O23—C23—C24—C26'	7.2 (7)
C1—C2—C7—O8	3.6 (4)	C22—C23—C24—C26'	-171.6 (6)
C3—C2—C7—C6	-1.0 (4)	O23—C23—C24—C1	-135.7 (2)
C1—C2—C7—C6	-176.1 (3)	C22—C23—C24—C1	45.5 (3)
C5—C6—C7—O8	-178.5 (3)	N25—C1—C24—C26'	178.3 (10)
C5—C6—C7—C2	1.2 (5)	C2—C1—C24—C26'	49.7 (11)

C2—C7—O8—C9	173.9 (3)	N25—C1—C24—C23	-49.1 (3)
C6—C7—O8—C9	-6.4 (5)	C2—C1—C24—C23	-177.7 (2)
C7—O8—C9—C10	-178.3 (3)	C20—C21—N25—C1	172.1 (2)
O8—C9—C10—O11	62.0 (4)	C22—C21—N25—C1	-61.2 (2)
C9—C10—O11—C12	-165.0 (3)	C2—C1—N25—C21	-176.4 (2)
C10—O11—C12—C13	163.3 (3)	C24—C1—N25—C21	57.4 (3)
O11—C12—C13—O14	-61.1 (4)	C23—C22—C26—O26	81.4 (3)
C12—C13—O14—C15	179.7 (3)	C21—C22—C26—O26	-41.9 (4)
C13—O14—C15—C20	-174.1 (3)	C23—C22—C26—O27	-99.5 (2)
C13—O14—C15—C16	6.5 (4)	C21—C22—C26—O27	137.1 (2)
O14—C15—C16—C17	178.2 (3)	O26—C26—O27—C27	6.7 (4)
C20—C15—C16—C17	-1.2 (5)	C22—C26—O27—C27	-172.4 (2)
C15—C16—C17—C18	1.1 (6)	C26—O27—C27—C28	97.3 (3)
C16—C17—C18—C19	-0.7 (6)	O27—C27—C28—C29	48.4 (3)
C17—C18—C19—C20	0.4 (5)	O27—C27—C28—C30	172.2 (2)
O14—C15—C20—C19	-178.6 (2)	C23—C24—C26'—O26'	-93.1 (17)
C16—C15—C20—C19	0.8 (4)	C1—C24—C26'—O26'	42 (2)
O14—C15—C20—C21	-3.3 (4)	C23—C24—C26'—O27'	93.9 (16)
C16—C15—C20—C21	176.2 (3)	C1—C24—C26'—O27'	-131.4 (12)
C18—C19—C20—C15	-0.4 (4)	O26'—C26'—O27'—C27'	-5 (3)
C18—C19—C20—C21	-176.0 (3)	C24—C26'—O27'—C27'	167.0 (14)
C15—C20—C21—N25	64.0 (3)	C26'—O27'—C27'—C28'	-98.4 (19)
C19—C20—C21—N25	-120.7 (3)	O27'—C27'—C28'—C29'	-48 (2)
C15—C20—C21—C22	-63.1 (3)	O27'—C27'—C28'—C30'	-173.3 (14)

Crystal data of compound 7

$C_{34}H_{35}NO_6$	$D_x = 1.301 \text{ Mg m}^{-3}$
$M_r = 553.63$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
Orthorhombic, $P2_12_12_1$	Cell parameters from 20483 reflections
$a = 9.88859 (8) \text{ \AA}$	$\theta = 3.7\text{--}76.9^\circ$
$b = 12.11532 (9) \text{ \AA}$	$\mu = 0.72 \text{ mm}^{-1}$
$c = 23.59512 (18) \text{ \AA}$	$T = 100 \text{ K}$
$V = 2826.78 (4) \text{ \AA}^3$	Prism, colourless
$Z = 4$	$0.19 \times 0.16 \times 0.11 \text{ mm}$
$F(000) = 1176$	

Data collection

Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector diffractometer	5726 reflections with $I > 2\sigma(I)$
Radiation source: micro-focus sealed X-ray tube	$R_{\text{int}} = 0.031$
ϕ and ω scans	$\theta_{\text{max}} = 77.8^\circ$, $\theta_{\text{min}} = 3.8^\circ$
Absorption correction: multi-scan <i>CrysAlis PRO</i> 1.171.41.117a (Rigaku Oxford Diffraction, 2021). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	$h = -12 \rightarrow 11$
$T_{\text{min}} = 0.794$, $T_{\text{max}} = 1.000$	$k = -15 \rightarrow 14$
25254 measured reflections	$l = -29 \rightarrow 20$
5850 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2 + 0.7028P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.072$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
5850 reflections	$\Delta_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
377 parameters	Extinction correction: <i>SHELXL</i> , $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.00050 (5)
Primary atom site location: difference Fourier map	Absolute structure: Flack x determined using 2363 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons and Flack (2004), <i>Acta Cryst. A</i> 60, s61).
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.09 (5)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$) for 7

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.60817 (17)	0.55471 (13)	0.43323 (7)	0.0197 (3)
H1	0.5898	0.5077	0.3992	0.024*
C2	0.54426 (17)	0.49539 (14)	0.48306 (7)	0.0201 (3)
C3	0.46009 (17)	0.40052 (14)	0.47434 (7)	0.0214 (3)
C4	0.42872 (18)	0.35699 (14)	0.41976 (7)	0.0233 (3)
H4	0.4691	0.3892	0.3872	0.028*
C5	0.3416 (2)	0.26975 (15)	0.41316 (8)	0.0280 (4)
H5	0.3224	0.2432	0.3762	0.034*
C6	0.2804 (2)	0.21897 (15)	0.46020 (9)	0.0332 (4)
H6	0.2199	0.1588	0.4551	0.040*
C7	0.3089 (2)	0.25716 (15)	0.51319 (8)	0.0319 (4)
H7	0.2687	0.2223	0.5451	0.038*
C8	0.39742 (19)	0.34789 (15)	0.52171 (8)	0.0260 (4)
C9	0.4212 (2)	0.39019 (16)	0.57666 (8)	0.0313 (4)
H9	0.3818	0.3545	0.6085	0.038*
C10	0.4997 (2)	0.48144 (16)	0.58481 (8)	0.0290 (4)
H10	0.5140	0.5091	0.6220	0.035*
C11	0.55982 (18)	0.53479 (14)	0.53781 (8)	0.0230 (3)
O12	0.63745 (13)	0.62745 (10)	0.54449 (5)	0.0252 (3)
C13	0.62611 (19)	0.68788 (15)	0.59679 (7)	0.0246 (4)
H13A	0.6779	0.6506	0.6272	0.030*
H13B	0.5303	0.6930	0.6086	0.030*
C14	0.68259 (19)	0.80064 (15)	0.58584 (8)	0.0260 (4)
H14A	0.6806	0.8452	0.6210	0.031*
H14B	0.7775	0.7951	0.5728	0.031*
O15	0.60137 (12)	0.85070 (10)	0.54346 (5)	0.0254 (3)
C16	0.6598 (2)	0.94846 (15)	0.52162 (8)	0.0283 (4)
H16A	0.7546	0.9347	0.5102	0.034*
H16B	0.6590	1.0072	0.5508	0.034*
C17	0.5776 (2)	0.98335 (14)	0.47124 (8)	0.0265 (4)
H17A	0.4824	0.9962	0.4823	0.032*
H17B	0.6144	1.0523	0.4548	0.032*
O18	0.58638 (13)	0.89481 (10)	0.43112 (5)	0.0247 (3)
C19	0.51129 (18)	0.90076 (14)	0.38257 (7)	0.0222 (3)

C20	0.44003 (19)	0.99768 (14)	0.36737 (8)	0.0252 (4)
H20	0.4399	1.0597	0.3920	0.030*
C21	0.37152 (19)	1.00180 (14)	0.31726 (8)	0.0259 (4)
H21	0.3233	1.0668	0.3074	0.031*
C22	0.37114 (17)	0.91088 (14)	0.27984 (7)	0.0221 (3)
C23	0.30499 (18)	0.91791 (15)	0.22673 (8)	0.0266 (4)
H23	0.2596	0.9841	0.2166	0.032*
C24	0.30534 (18)	0.83130 (16)	0.18987 (8)	0.0278 (4)
H24	0.2616	0.8375	0.1542	0.033*
C25	0.37128 (19)	0.73227 (15)	0.20525 (8)	0.0267 (4)
H25	0.3711	0.6717	0.1797	0.032*
C26	0.43532 (18)	0.72237 (14)	0.25636 (7)	0.0233 (3)
H26	0.4780	0.6545	0.2658	0.028*
C27	0.43968 (16)	0.81129 (13)	0.29590 (7)	0.0205 (3)
C28	0.51017 (17)	0.80725 (13)	0.34881 (7)	0.0197 (3)
C29	0.58743 (17)	0.70475 (13)	0.36738 (7)	0.0188 (3)
H29	0.5672	0.6463	0.3387	0.023*
C30	0.74453 (17)	0.72452 (13)	0.36506 (7)	0.0198 (3)
H30	0.7688	0.7864	0.3912	0.024*
C31	0.81810 (17)	0.61987 (14)	0.38386 (7)	0.0210 (3)
O31	0.91288 (13)	0.58264 (11)	0.35728 (5)	0.0272 (3)
C32	0.76443 (17)	0.56617 (14)	0.43690 (7)	0.0224 (3)
H32A	0.7888	0.6114	0.4703	0.027*
H32B	0.8058	0.4923	0.4415	0.027*
N33	0.54213 (14)	0.66133 (11)	0.42210 (6)	0.0188 (3)
H33	0.561 (2)	0.7073 (18)	0.4510 (9)	0.023*
C33	0.78134 (16)	0.75633 (13)	0.30482 (7)	0.0207 (3)
O33	0.75215 (13)	0.69920 (10)	0.26467 (5)	0.0257 (3)
O34	0.84082 (12)	0.85530 (10)	0.30232 (5)	0.0235 (3)
C34	0.86182 (19)	0.91002 (15)	0.24635 (7)	0.0267 (4)
C35	0.7237 (2)	0.93573 (16)	0.22137 (8)	0.0305 (4)
H35A	0.6766	0.8666	0.2126	0.046*
H35B	0.7349	0.9789	0.1866	0.046*
H35C	0.6706	0.9783	0.2488	0.046*
C36	0.9493 (2)	0.83929 (18)	0.20796 (8)	0.0337 (4)
H36A	1.0319	0.8180	0.2282	0.051*

H36B	0.9733	0.8814	0.1739	0.051*
H36C	0.8992	0.7728	0.1970	0.051*
C37	0.9342 (2)	1.01587 (17)	0.26266 (8)	0.0342 (4)
H37A	0.8792	1.0567	0.2901	0.051*
H37B	0.9480	1.0613	0.2288	0.051*
H37C	1.0220	0.9979	0.2796	0.051*

Atomic displacement parameters ($\text{\AA}^2$) for 7

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0192 (7)	0.0192 (7)	0.0207 (7)	0.0015 (6)	0.0007 (6)	-0.0004 (6)
C2	0.0194 (8)	0.0182 (7)	0.0228 (8)	0.0036 (6)	0.0017 (6)	0.0020 (6)
C3	0.0217 (8)	0.0177 (7)	0.0248 (8)	0.0038 (6)	0.0023 (7)	0.0032 (6)
C4	0.0259 (8)	0.0201 (7)	0.0240 (8)	0.0006 (7)	0.0027 (7)	0.0021 (6)
C5	0.0320 (10)	0.0226 (8)	0.0294 (9)	-0.0020 (7)	0.0002 (8)	-0.0011 (7)
C6	0.0358 (10)	0.0225 (8)	0.0415 (11)	-0.0084 (8)	0.0070 (9)	-0.0010 (8)
C7	0.0404 (11)	0.0210 (8)	0.0344 (10)	-0.0042 (8)	0.0119 (8)	0.0031 (8)
C8	0.0289 (9)	0.0207 (8)	0.0282 (9)	0.0001 (7)	0.0053 (7)	0.0035 (7)
C9	0.0409 (10)	0.0279 (9)	0.0251 (9)	-0.0017 (8)	0.0093 (8)	0.0056 (7)
C10	0.0388 (10)	0.0278 (9)	0.0206 (8)	-0.0009 (8)	0.0035 (8)	0.0002 (7)
C11	0.0233 (8)	0.0217 (8)	0.0242 (8)	0.0011 (6)	-0.0001 (7)	0.0017 (7)
O12	0.0273 (6)	0.0259 (6)	0.0223 (6)	-0.0043 (5)	0.0037 (5)	-0.0039 (5)
C13	0.0246 (8)	0.0289 (9)	0.0204 (8)	0.0032 (7)	-0.0027 (7)	-0.0031 (7)
C14	0.0230 (8)	0.0309 (9)	0.0241 (8)	0.0002 (7)	-0.0035 (7)	-0.0045 (7)
O15	0.0236 (6)	0.0267 (6)	0.0260 (6)	-0.0023 (5)	-0.0018 (5)	0.0010 (5)
C16	0.0331 (9)	0.0233 (8)	0.0286 (9)	-0.0060 (7)	-0.0010 (8)	-0.0045 (7)
C17	0.0317 (9)	0.0200 (8)	0.0277 (9)	-0.0019 (7)	0.0024 (8)	-0.0042 (7)
O18	0.0296 (6)	0.0214 (6)	0.0232 (6)	0.0019 (5)	-0.0012 (5)	-0.0037 (5)
C19	0.0231 (8)	0.0213 (8)	0.0222 (8)	-0.0028 (7)	0.0024 (7)	0.0027 (7)
C20	0.0296 (9)	0.0183 (7)	0.0276 (9)	0.0009 (7)	0.0044 (7)	0.0001 (7)
C21	0.0278 (9)	0.0199 (7)	0.0301 (9)	0.0029 (7)	0.0037 (7)	0.0052 (7)
C22	0.0201 (8)	0.0213 (7)	0.0250 (8)	-0.0008 (6)	0.0034 (6)	0.0054 (7)
C23	0.0237 (8)	0.0265 (8)	0.0295 (9)	0.0015 (7)	0.0006 (7)	0.0089 (7)
C24	0.0238 (8)	0.0336 (9)	0.0261 (9)	-0.0004 (7)	-0.0026 (7)	0.0055 (7)
C25	0.0267 (9)	0.0268 (8)	0.0265 (8)	-0.0015 (7)	-0.0014 (7)	-0.0011 (7)
C26	0.0227 (8)	0.0210 (8)	0.0263 (8)	-0.0014 (7)	0.0002 (7)	0.0015 (7)
C27	0.0175 (7)	0.0209 (7)	0.0231 (8)	-0.0018 (6)	0.0044 (6)	0.0038 (6)

C28	0.0191 (8)	0.0180 (7)	0.0219 (8)	-0.0013 (6)	0.0039 (6)	0.0035 (6)
C29	0.0176 (7)	0.0180 (7)	0.0206 (7)	-0.0005 (6)	0.0004 (6)	-0.0001 (6)
C30	0.0195 (8)	0.0193 (7)	0.0204 (8)	-0.0021 (6)	0.0020 (6)	-0.0012 (6)
C31	0.0162 (7)	0.0232 (8)	0.0234 (8)	-0.0014 (6)	-0.0016 (6)	-0.0045 (7)
O31	0.0209 (6)	0.0330 (7)	0.0278 (6)	0.0033 (5)	0.0036 (5)	-0.0050 (5)
C32	0.0189 (8)	0.0237 (8)	0.0245 (8)	0.0037 (6)	0.0003 (7)	0.0010 (6)
N33	0.0179 (6)	0.0181 (6)	0.0204 (6)	0.0010 (5)	0.0020 (5)	0.0011 (5)
C33	0.0178 (7)	0.0209 (7)	0.0235 (8)	-0.0009 (6)	0.0031 (6)	-0.0007 (6)
O33	0.0272 (6)	0.0267 (6)	0.0231 (6)	-0.0044 (5)	0.0016 (5)	-0.0024 (5)
O34	0.0251 (6)	0.0245 (6)	0.0211 (6)	-0.0070 (5)	0.0027 (5)	0.0010 (5)
C34	0.0296 (9)	0.0281 (8)	0.0223 (8)	-0.0068 (8)	0.0036 (7)	0.0031 (7)
C35	0.0348 (10)	0.0300 (9)	0.0266 (9)	0.0004 (8)	0.0008 (8)	0.0049 (7)
C36	0.0318 (10)	0.0409 (10)	0.0285 (9)	-0.0068 (8)	0.0099 (8)	-0.0004 (8)
C37	0.0443 (11)	0.0331 (10)	0.0252 (9)	-0.0148 (9)	0.0055 (9)	0.0031 (8)

Geometric parameters (Å, °) for 7

C1—N33	1.471 (2)	C21—C22	1.412 (3)
C1—C2	1.516 (2)	C21—H21	0.9500
C1—C32	1.554 (2)	C22—C23	1.416 (3)
C1—H1	1.0000	C22—C27	1.435 (2)
C2—C11	1.386 (2)	C23—C24	1.363 (3)
C2—C3	1.434 (2)	C23—H23	0.9500
C3—C4	1.426 (2)	C24—C25	1.413 (3)
C3—C8	1.428 (2)	C24—H24	0.9500
C4—C5	1.372 (3)	C25—C26	1.367 (3)
C4—H4	0.9500	C25—H25	0.9500
C5—C6	1.406 (3)	C26—C27	1.426 (2)
C5—H5	0.9500	C26—H26	0.9500
C6—C7	1.363 (3)	C27—C28	1.431 (2)
C6—H6	0.9500	C28—C29	1.523 (2)
C7—C8	1.419 (3)	C29—N33	1.464 (2)
C7—H7	0.9500	C29—C30	1.573 (2)
C8—C9	1.414 (3)	C29—H29	1.0000
C9—C10	1.365 (3)	C30—C33	1.517 (2)
C9—H9	0.9500	C30—C31	1.528 (2)
C10—C11	1.415 (3)	C30—H30	1.0000
C10—H10	0.9500	C31—O31	1.215 (2)

C11—O12	1.369 (2)	C31—C32	1.507 (2)
O12—C13	1.439 (2)	C32—H32A	0.9900
C13—C14	1.498 (3)	C32—H32B	0.9900
C13—H13A	0.9900	N33—H33	0.90 (2)
C13—H13B	0.9900	C33—O33	1.208 (2)
C14—O15	1.419 (2)	C33—O34	1.337 (2)
C14—H14A	0.9900	O34—C34	1.492 (2)
C14—H14B	0.9900	C34—C36	1.518 (3)
O15—C16	1.415 (2)	C34—C37	1.518 (3)
C16—C17	1.501 (3)	C34—C35	1.520 (3)
C16—H16A	0.9900	C35—H35A	0.9800
C16—H16B	0.9900	C35—H35B	0.9800
C17—O18	1.433 (2)	C35—H35C	0.9800
C17—H17A	0.9900	C36—H36A	0.9800
C17—H17B	0.9900	C36—H36B	0.9800
O18—C19	1.367 (2)	C36—H36C	0.9800
C19—C28	1.385 (2)	C37—H37A	0.9800
C19—C20	1.416 (2)	C37—H37B	0.9800
C20—C21	1.364 (3)	C37—H37C	0.9800
C20—H20	0.9500		
N33—C1—C2	111.69 (13)	C21—C22—C27	119.33 (16)
N33—C1—C32	111.89 (14)	C23—C22—C27	120.15 (16)
C2—C1—C32	114.43 (14)	C24—C23—C22	121.16 (17)
N33—C1—H1	106.0	C24—C23—H23	119.4
C2—C1—H1	106.0	C22—C23—H23	119.4
C32—C1—H1	106.0	C23—C24—C25	119.40 (17)
C11—C2—C3	118.31 (15)	C23—C24—H24	120.3
C11—C2—C1	120.89 (15)	C25—C24—H24	120.3
C3—C2—C1	120.71 (15)	C26—C25—C24	120.99 (17)
C4—C3—C8	116.57 (15)	C26—C25—H25	119.5
C4—C3—C2	123.53 (15)	C24—C25—H25	119.5
C8—C3—C2	119.83 (16)	C25—C26—C27	121.65 (16)
C5—C4—C3	121.59 (16)	C25—C26—H26	119.2
C5—C4—H4	119.2	C27—C26—H26	119.2
C3—C4—H4	119.2	C26—C27—C28	124.04 (15)
C4—C5—C6	121.19 (18)	C26—C27—C22	116.64 (15)
C4—C5—H5	119.4	C28—C27—C22	119.30 (15)
C6—C5—H5	119.4	C19—C28—C27	118.55 (15)

C7—C6—C5	119.12 (18)	C19—C28—C29	119.84 (15)
C7—C6—H6	120.4	C27—C28—C29	121.58 (14)
C5—C6—H6	120.4	N33—C29—C28	113.16 (13)
C6—C7—C8	121.35 (17)	N33—C29—C30	112.81 (13)
C6—C7—H7	119.3	C28—C29—C30	111.18 (13)
C8—C7—H7	119.3	N33—C29—H29	106.4
C9—C8—C7	120.86 (17)	C28—C29—H29	106.4
C9—C8—C3	118.93 (16)	C30—C29—H29	106.4
C7—C8—C3	120.17 (17)	C33—C30—C31	111.64 (13)
C10—C9—C8	121.16 (17)	C33—C30—C29	107.97 (13)
C10—C9—H9	119.4	C31—C30—C29	109.50 (13)
C8—C9—H9	119.4	C33—C30—H30	109.2
C9—C10—C11	119.93 (17)	C31—C30—H30	109.2
C9—C10—H10	120.0	C29—C30—H30	109.2
C11—C10—H10	120.0	O31—C31—C32	122.70 (16)
O12—C11—C2	116.87 (15)	O31—C31—C30	121.71 (15)
O12—C11—C10	121.34 (16)	C32—C31—C30	115.58 (14)
C2—C11—C10	121.79 (16)	C31—C32—C1	110.03 (14)
C11—O12—C13	118.17 (13)	C31—C32—H32A	109.7
O12—C13—C14	106.67 (14)	C1—C32—H32A	109.7
O12—C13—H13A	110.4	C31—C32—H32B	109.7
C14—C13—H13A	110.4	C1—C32—H32B	109.7
O12—C13—H13B	110.4	H32A—C32—H32B	108.2
C14—C13—H13B	110.4	C29—N33—C1	109.69 (13)
H13A—C13—H13B	108.6	C29—N33—H33	112.4 (13)
O15—C14—C13	107.48 (14)	C1—N33—H33	108.3 (13)
O15—C14—H14A	110.2	O33—C33—O34	125.75 (16)
C13—C14—H14A	110.2	O33—C33—C30	122.12 (15)
O15—C14—H14B	110.2	O34—C33—C30	112.02 (14)
C13—C14—H14B	110.2	C33—O34—C34	119.92 (13)
H14A—C14—H14B	108.5	O34—C34—C36	110.89 (15)
C16—O15—C14	112.55 (14)	O34—C34—C37	102.50 (14)
O15—C16—C17	107.66 (15)	C36—C34—C37	111.08 (16)
O15—C16—H16A	110.2	O34—C34—C35	108.01 (14)
C17—C16—H16A	110.2	C36—C34—C35	113.37 (16)
O15—C16—H16B	110.2	C37—C34—C35	110.41 (16)
C17—C16—H16B	110.2	C34—C35—H35A	109.5
H16A—C16—H16B	108.5	C34—C35—H35B	109.5
O18—C17—C16	106.25 (14)	H35A—C35—H35B	109.5

O18—C17—H17A	110.5	C34—C35—H35C	109.5
C16—C17—H17A	110.5	H35A—C35—H35C	109.5
O18—C17—H17B	110.5	H35B—C35—H35C	109.5
C16—C17—H17B	110.5	C34—C36—H36A	109.5
H17A—C17—H17B	108.7	C34—C36—H36B	109.5
C19—O18—C17	118.76 (14)	H36A—C36—H36B	109.5
O18—C19—C28	116.31 (15)	C34—C36—H36C	109.5
O18—C19—C20	121.77 (16)	H36A—C36—H36C	109.5
C28—C19—C20	121.91 (16)	H36B—C36—H36C	109.5
C21—C20—C19	119.83 (16)	C34—C37—H37A	109.5
C21—C20—H20	120.1	C34—C37—H37B	109.5
C19—C20—H20	120.1	H37A—C37—H37B	109.5
C20—C21—C22	121.00 (16)	C34—C37—H37C	109.5
C20—C21—H21	119.5	H37A—C37—H37C	109.5
C22—C21—H21	119.5	H37B—C37—H37C	109.5
C21—C22—C23	120.51 (16)		
N33—C1—C2—C11	-69.7 (2)	C23—C24—C25—C26	-0.4 (3)
C32—C1—C2—C11	58.7 (2)	C24—C25—C26—C27	-0.7 (3)
N33—C1—C2—C3	106.87 (17)	C25—C26—C27—C28	-177.11 (16)
C32—C1—C2—C3	-124.70 (16)	C25—C26—C27—C22	1.2 (2)
C11—C2—C3—C4	175.48 (16)	C21—C22—C27—C26	179.84 (16)
C1—C2—C3—C4	-1.2 (2)	C23—C22—C27—C26	-0.8 (2)
C11—C2—C3—C8	-1.4 (2)	C21—C22—C27—C28	-1.7 (2)
C1—C2—C3—C8	-178.05 (15)	C23—C22—C27—C28	177.68 (15)
C8—C3—C4—C5	0.7 (2)	O18—C19—C28—C27	-176.37 (14)
C2—C3—C4—C5	-176.30 (17)	C20—C19—C28—C27	2.9 (2)
C3—C4—C5—C6	-0.5 (3)	O18—C19—C28—C29	1.4 (2)
C4—C5—C6—C7	-0.3 (3)	C20—C19—C28—C29	-179.40 (15)
C5—C6—C7—C8	0.9 (3)	C26—C27—C28—C19	177.46 (16)
C6—C7—C8—C9	176.9 (2)	C22—C27—C28—C19	-0.8 (2)
C6—C7—C8—C3	-0.7 (3)	C26—C27—C28—C29	-0.2 (2)
C4—C3—C8—C9	-177.76 (17)	C22—C27—C28—C29	-178.53 (15)
C2—C3—C8—C9	-0.7 (3)	C19—C28—C29—N33	59.3 (2)
C4—C3—C8—C7	-0.1 (2)	C27—C28—C29—N33	-123.00 (16)
C2—C3—C8—C7	177.03 (17)	C19—C28—C29—C30	-68.82 (19)
C7—C8—C9—C10	-176.03 (19)	C27—C28—C29—C30	108.83 (17)
C3—C8—C9—C10	1.7 (3)	N33—C29—C30—C33	173.49 (13)
C8—C9—C10—C11	-0.5 (3)	C28—C29—C30—C33	-58.15 (17)

C3—C2—C11—O12	-178.06 (14)	N33—C29—C30—C31	51.75 (17)
C1—C2—C11—O12	-1.4 (2)	C28—C29—C30—C31	-179.89 (13)
C3—C2—C11—C10	2.6 (3)	C33—C30—C31—O31	13.6 (2)
C1—C2—C11—C10	179.23 (16)	C29—C30—C31—O31	133.11 (16)
C9—C10—C11—O12	179.01 (17)	C33—C30—C31—C32	-165.87 (14)
C9—C10—C11—C2	-1.7 (3)	C29—C30—C31—C32	-46.36 (19)
C2—C11—O12—C13	163.12 (15)	O31—C31—C32—C1	-131.15 (17)
C10—C11—O12—C13	-17.5 (2)	C30—C31—C32—C1	48.32 (19)
C11—O12—C13—C14	-161.60 (14)	N33—C1—C32—C31	-55.11 (18)
O12—C13—C14—O15	62.00 (17)	C2—C1—C32—C31	176.57 (14)
C13—C14—O15—C16	-168.44 (14)	C28—C29—N33—C1	172.31 (13)
C14—O15—C16—C17	170.88 (15)	C30—C29—N33—C1	-60.37 (17)
O15—C16—C17—O18	-60.80 (19)	C2—C1—N33—C29	-168.43 (13)
C16—C17—O18—C19	174.78 (14)	C32—C1—N33—C29	61.81 (17)
C17—O18—C19—C28	-172.10 (15)	C31—C30—C33—O33	65.2 (2)
C17—O18—C19—C20	8.7 (2)	C29—C30—C33—O33	-55.2 (2)
O18—C19—C20—C21	176.93 (16)	C31—C30—C33—O34	-118.36 (15)
C28—C19—C20—C21	-2.3 (3)	C29—C30—C33—O34	121.22 (14)
C19—C20—C21—C22	-0.4 (3)	O33—C33—O34—C34	7.4 (2)
C20—C21—C22—C23	-177.01 (17)	C30—C33—O34—C34	-168.90 (14)
C20—C21—C22—C27	2.4 (3)	C33—O34—C34—C36	-59.8 (2)
C21—C22—C23—C24	179.11 (17)	C33—O34—C34—C37	-178.43 (15)
C27—C22—C23—C24	-0.3 (3)	C33—O34—C34—C35	64.97 (19)
C22—C23—C24—C25	0.9 (3)		

Hydrogen-bond geometry (Å, °) for 7

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C21—H21 $\cdots$ O33 ⁱ	0.95	2.45	3.309 (2)	150
C30—H30 $\cdots$ O18	1.00	2.42	3.022 (2)	118
C32—H32A $\cdots$ O12	0.99	2.31	2.928 (2)	119
N33—H33 $\cdots$ O12	0.90 (2)	2.52 (2)	3.0653 (19)	119.2 (16)
N33—H33 $\cdots$ O18	0.90 (2)	2.33 (2)	2.8702 (19)	118.1 (16)
C37—H37B $\cdots$ O31 ⁱⁱ	0.98	2.47	3.309 (2)	144

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