Supporting information

First evidence of 1,3-bis-indolylallenes: generation by a sequential double nucleophilic process from ynones

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1. General methods

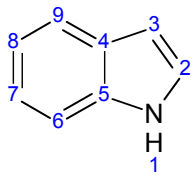
THF was dried and distilled over sodium/benzophenone. All other reagents were used as commercially available. In particular, commercial solutions of n-BuLi were 2.5 M in hexane. All reactions were carried out under argon atmosphere using Schlenk and vacuum line techniques. Column chromatography was carried out on silica gel (60 P, 70-200 mm). Silica gel thin–layer chromatography plates (60F254, 0.25 mm) were revealed by treatment with an ethanolic solution of phosphomolybdic acid (20 %). The following analytical instruments were used. 1H and 13C NMR: Bruker DPX 300, Avance 300, Avance 400, Avance 400WB or Avance 500 spectrometers. Mass spectrometry: Quadrupolar Nermag R10-10H spectrometer. Most of the NMR spectra were recorded in CDCl3 solutions. NMR chemical shifts δ are given in ppm, with positive values to high frequency relative to the tetramethylsilane reference; coupling constants J are in Hz. IR: 0.1 mm CaF2 cell, Perkin- Elmer GX FTIR. 3-bromo-5-ethyl-1,2-dimethyl-1H-indole **1a**,[1] 1-phenyl-3-(trimethylsilyl)prop-2-yn-1-one **2**[2] and trimethyl[3-oxo-5-(trimethylsilyl)penta-1,4-diyn-1-yl]silane **3**[3] were prepared following described procedures.

2. General procedure

To a solution of 3-bromo-5-methoxy-1,2-dimethyl-1*H*-indole **1a** (200 mg, 0.8 mmol) in THF (5 mL) under stirring at -78 °C was added *n*-BuLi (290 µl, 0.73 mmol). The reaction mixture was stirred over 1 hour at -78 °C before addition of a solution of the ketone substrate (0.66 mmol) in THF (3 mL). The temperature was allowed to raise slowly 0 °C over 3 h. The mixture was then quenched with saturated aqueous NH_4Cl. The aqueous layer was extracted with diethylether and the combined organic layers were washed with brine, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by silica gel chromatography (pentane/EtOAc).

3. Characterizations

The following numbering is used in the assignment of NMR spectra of indolyl derivatives:



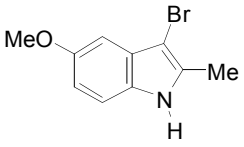
5-methoxy-3-[3-(5-methoxy-1,2-dimethyl-1H-indol-3-yl)-3-phenyl-1-(trimethylsilyl) propa-1,2-dien-1-yl]-1,2-dimethyl-1H-indole **(4).** Prepared from 1-phenyl-3-(trimethylsilyl)prop-2-yn-1-one **2** (133 mg, 0.66 mmol). Isolated as a light brown solid (30 mg, 15 %). Mp 149 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.27 (s, 9H, TMS); 2.18 (s, 3H, CH_3-C); 2.32 (s, 3H, CH_3-C); 3.38 (s, 3H, CH_3-N); 3.58 (s, 3H, CH_3-N); 3.66 (s, 3H, CH_3-O); 3.70 (s, 3H, CH_3-O); 6.64 (s, 1H, *H*9-Ind); 6.80 (d, *J* 10.4 Hz, 2H, *H*7-Ind); 6.88 (s, 1H, *H*9-Ind); 7.13 (d, *J* 8.7 Hz, 1H, *H*7-Ind); 7.16-7.22 (m, 2H, *H*6-Ind, *p*-Ph); 7.31 (t, *J* 7,5 Hz, 2H, *m*-Ph); 7.46 (d, *J* 7.8 Hz, 2H, *o*-Ph); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: -0.2 (TMS), 11.3, 11.8 (*C*H_3-C), 29.8 (2C, *C*H_3-N), 55.3, 55.5 (*C*H_3-O), 94.0, 94.6, 107.0, 108.0 (*C*=C=*C*, *C*3-Ind), 101.5, (2C, *C*9-Ind), 108.9, 109.0, 110.6, 110.7 (*C*6-, *C*7-Ind), 125.8 (*p*-Ph), 126.6, 128.2 (*m-,o*-Ph), 128.1, 132.0, 133.0, 135.3 (*C*2-, *C*4-, *C*5-Ind), 137.9 (*i*-Ph), 153.7, 153.7 (2C, *C*-OMe), 209.9 (C=*C*=C). IR: $\nu_{C=C=C}$ 1910 cm^{-1}; HRMS (DCI/CH_4) *m/z* calcd for $C_{34}H_{38}N_2O_2Si$: 534.2703, found: 534.2722.

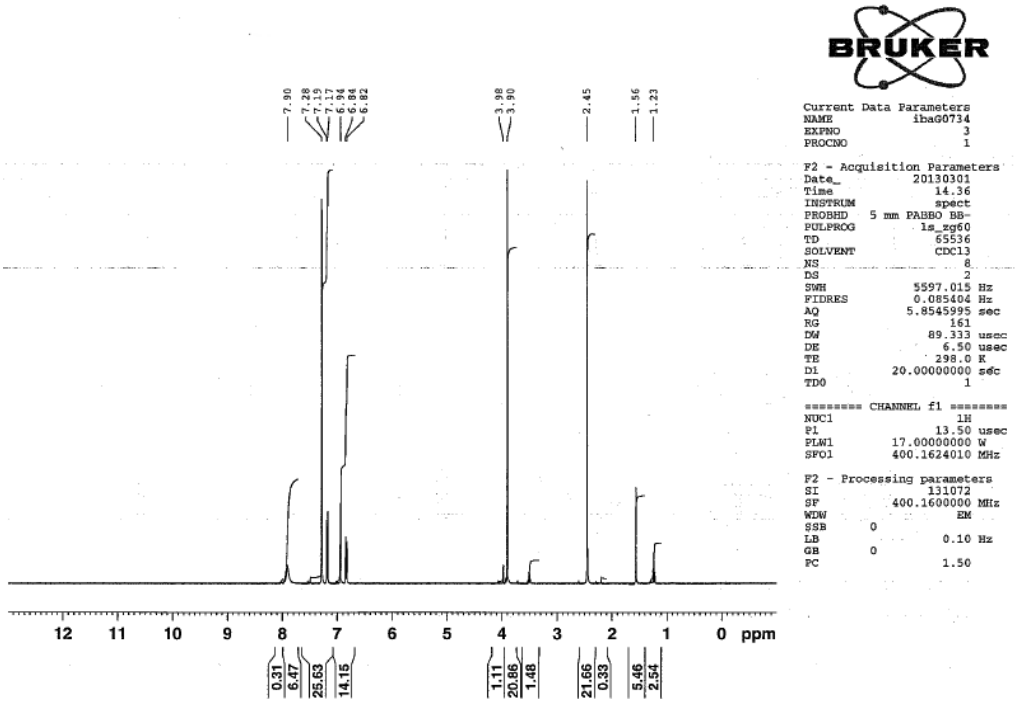
5-methoxy-3-[3-(5-methoxy-1,2-dimethyl-1H-indol-3-yl)-1,5-bis(trimethylsilyl)penta-1,2-dien-4-yn-1-yl]-1,2-dimethyl-1H-indole **(5).** Prepared from trimethyl[3-oxo-5-(trimethylsilyl) penta-1,4-diyn-1-yl]silane **3** (147 mg, 0.66 mmol). Isolated as a light brown oil (35 mg, 18 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.25 (s, 9H, TMS); 0.26 (s, 9H, TMS); 2.44 (s, 3H, CH_3-C); 2.46 (s, 3H, CH_3-C); 3.48 (s, 3H, CH_3-N); 3.62 (s, 3H, CH_3-N); 3.69 (s, 3H, CH_3-O); 3.82 (s, 3H, CH_3-O); 6.76 (d, *J* 8.9 Hz, 1H, *H*7-Ind); 6.82 (d, *J* 8.9 Hz, 1H, *H*7-Ind); 7.06-7.21 (m, 4H, *H*6-, *H*9-Ind); ^{13}C{^{1}H} NMR (101 MHz, Toluene-*d*8) δ: -0.08, -0.50 (TMS), 10.9, 11.0 (*C*H_3-C), 28.3, 28.7 (*C*H_3-N), 54.8, 55.0 (*C*H_3-O), 81.3, 94.9, 96.5, 102.9, 104.0, 107.0 (*C*≡*C*, *C*=C=*C*, *C*3-Ind), 101.6, 101.7 (*C*9-Ind), 108.9, 109.1, 111.0, 111.2 (*C*6-, *C*7-Ind), 127.1, 132.0, 132.3, 133.3, 133.6 (*C*2-,*C*4-,*C*5-Ind), 154.5 (*C*8-Ind), 216.7 (C=*C*=C); IR: $\nu_{C=C=C}$ 1901 cm^{-1}, $\nu_{C\equiv C}$ 2141cm^{-1}; HRMS (DCI/CH_4) *m/z* calcd for $C_{33}H_{42}N_2O_2Si_2$: 554.2785, found: 554.2803.

(5-methoxy-1,2-dimethyl-1H-indol-3-yl)(diphenyl)methanol **(6).** Prepared from benzophenone (120 mg, 0.66 mmol). Isolated as yellow solid (120 mg, 51 %). Mp 104 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 2.03 (s, 3H, C-CH_3); 2.92 (s, 1H, O*H*); 3.45 (s, 3H, N-CH_3); 3.63 (s, 3H, O-CH_3); 5.74 (s, 1H, *H*9-Ind); 6.74 (d, *J* 10.4 Hz, 1H, *H*7-Ind); 7.14 (d, *J* 8.8 Hz, 1H, *H*6-Ind); 7.29-7.39 (m, 6H, *m-, p*-Ph); 7.46 (d, *J* 7.9 Hz, 4H, *o*-Ph); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 12.2 (C-*C*H_3), 29.5 (N-*C*H_3), 55.4 (O-*C*H_3), 79.5 (*C*-OH), 102.5 (*C*9-Ind), 109.1 (*C*7-Ind), 110.5 (*C*6-Ind), 116.8 (*C*3-Ind), 127.0 (*p*-Ph), 127.7, 128.0 (*o-, m*-Ph), 127.7 (*C*4-Ind), 131.7, 136.4 (*C*2-, *C*5-Ind), 147.5 (*i*-Ph), 153.4 (*C*8-Ind); IR: ν_{O-H} 3499 cm^{-1}; HRMS (DCI/CH_4) *m/z* calcd for $C_{24}H_{23}NO_2$: 357.1729, found: 357.1713.

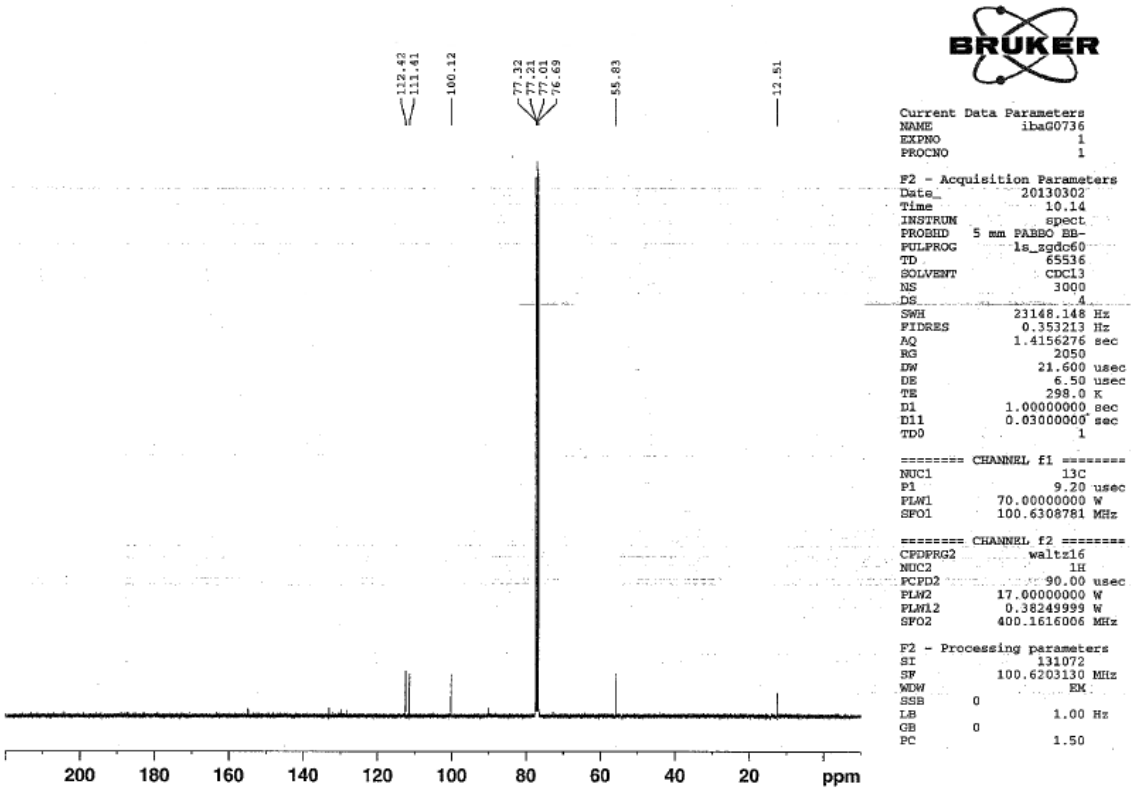
4. Spectra

• 3-bromo-5-methoxy-2-methyl-1*H*-indole (secondary indole precursor of **1a**):

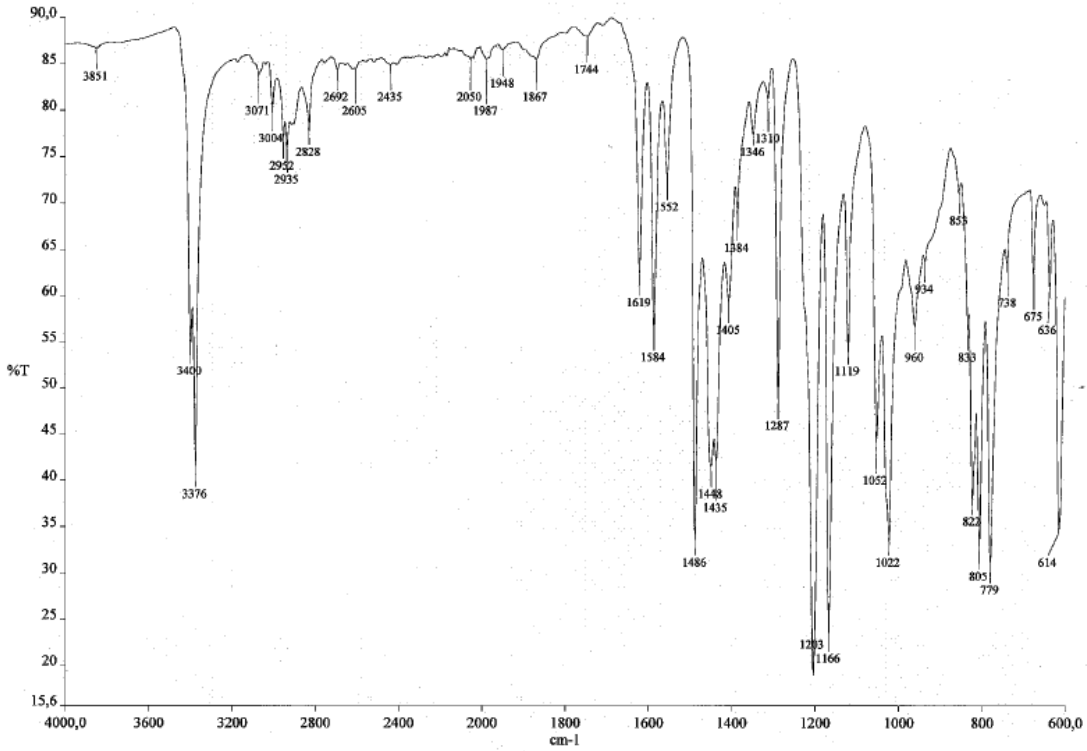




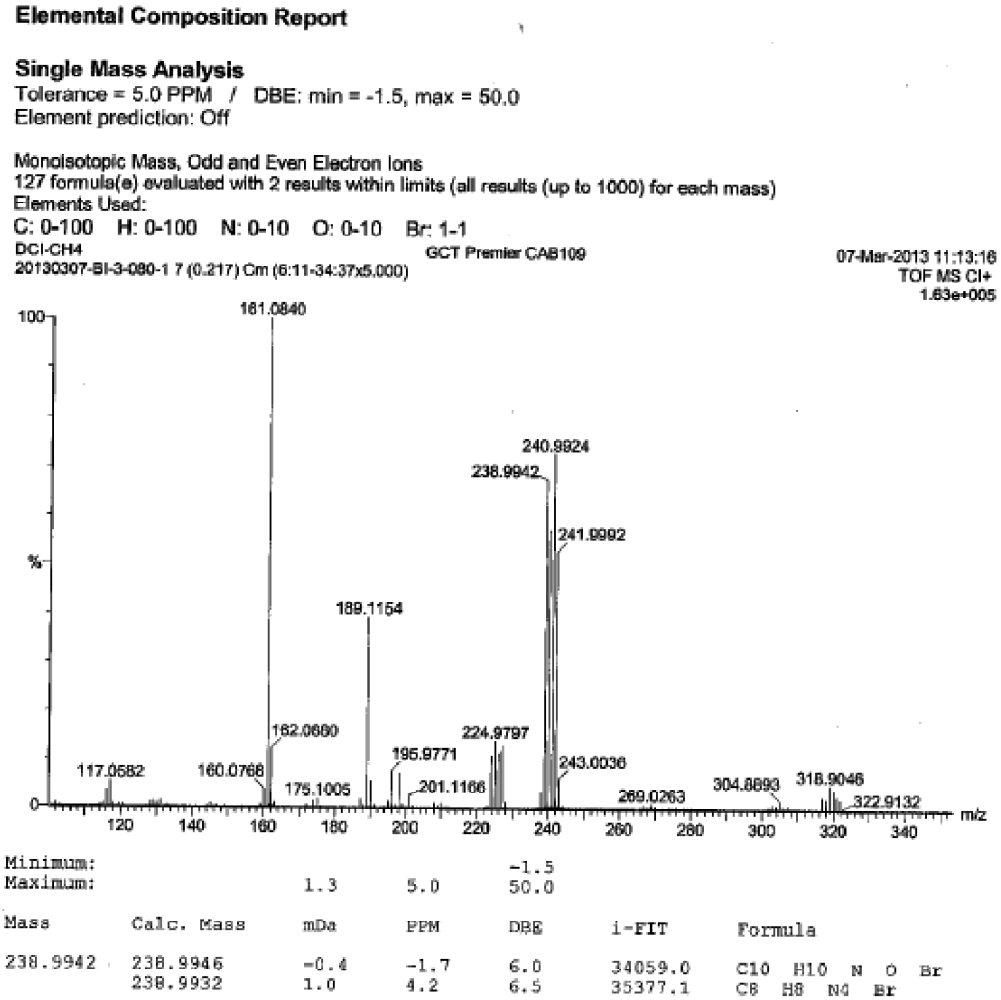
^{1}H NMR spectrum of the secondary indole precursor of **1a** ($CDCl_3$, 400 MHz).



^{13}C{^{1}H} NMR spectrum of the secondary indole precursor of **1a** ($CDCl_3$, 100 MHz).

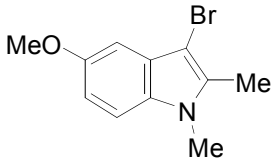


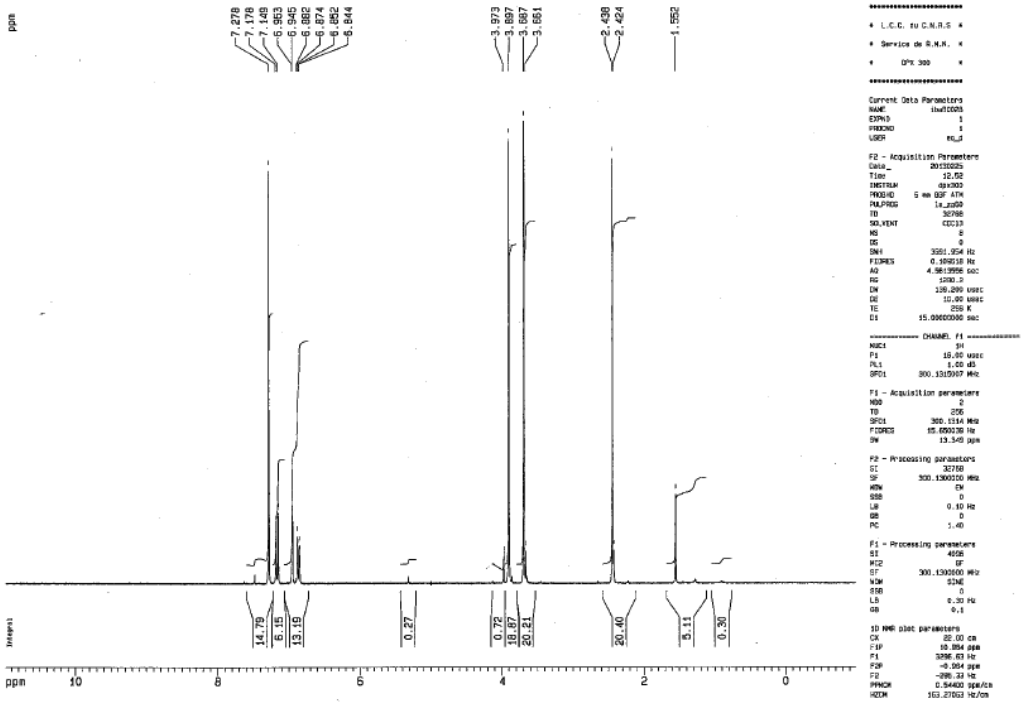
FT-IR spectrum of the secondary indole precursor of **1a**.



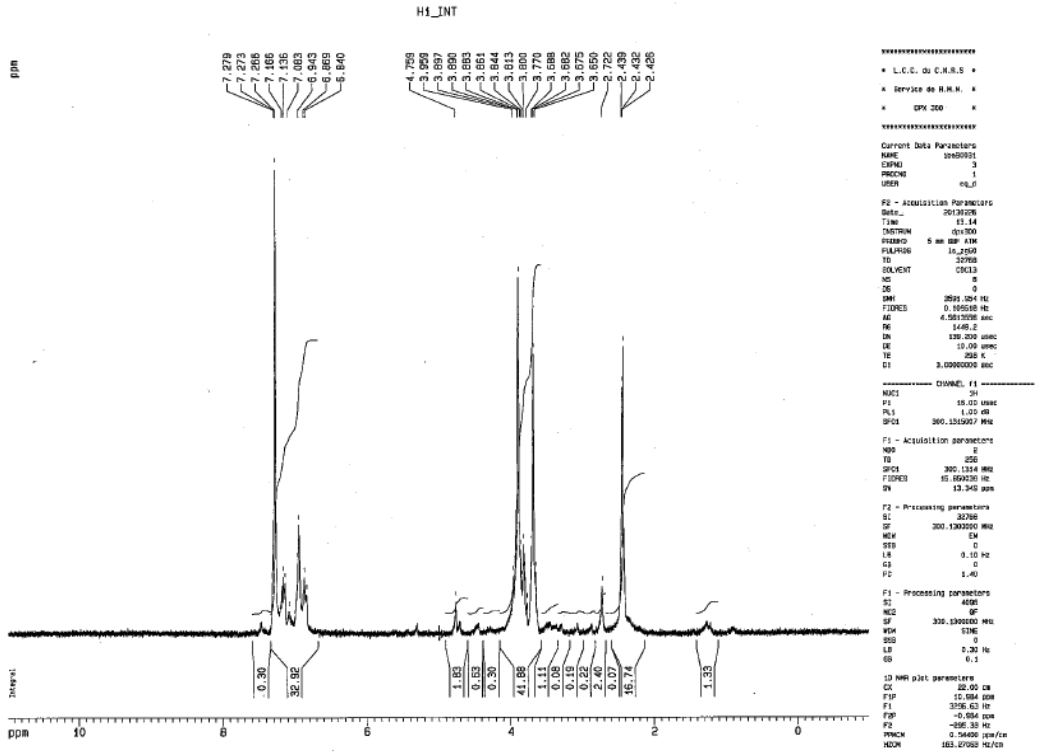
HRMS (DCI/NH_4) of the secondary indole precursor of **1a**.

• 3-bromo-5-methoxy-1,2-dimethyl-1*H*-indole (**1a**):



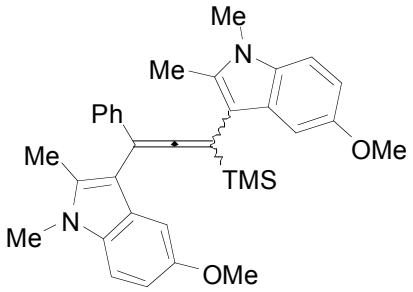


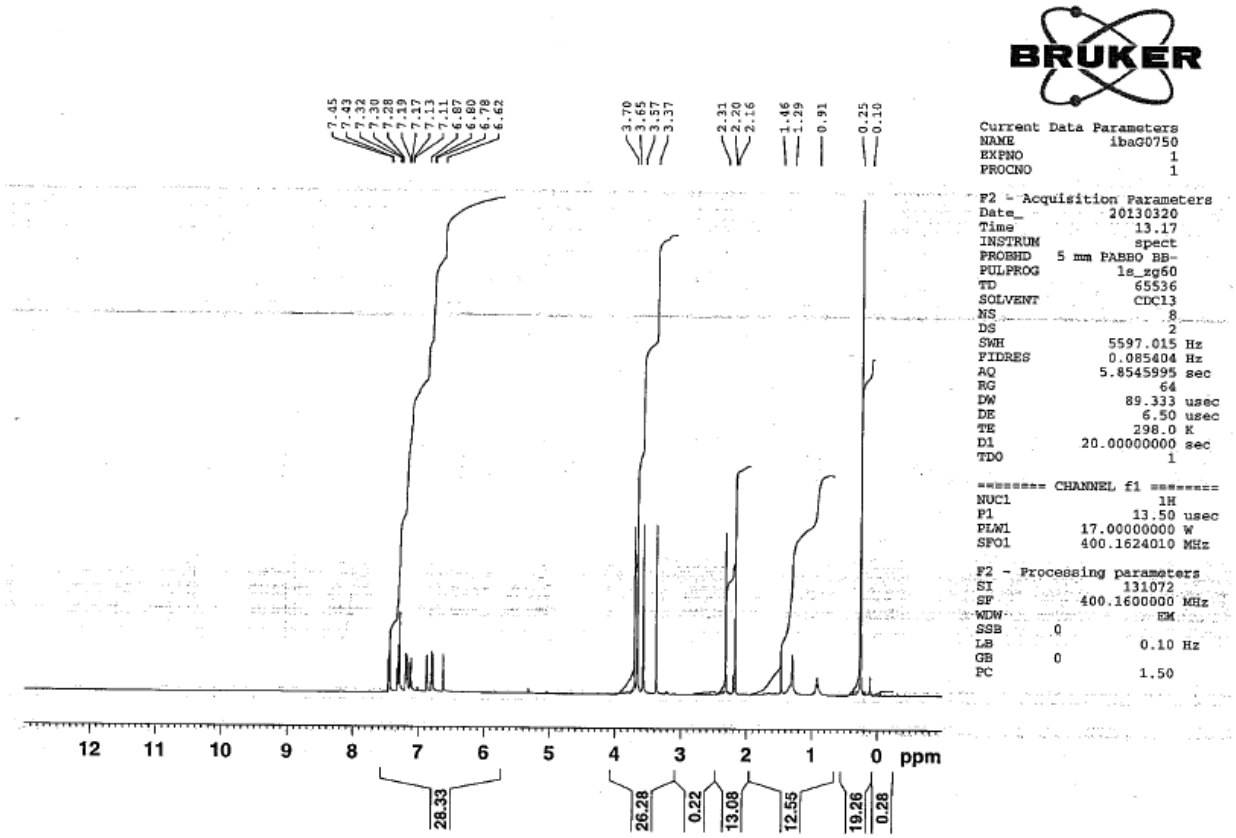
Initial ^{1}H NMR spectrum of **1a** ($CDCl_3$, 300 MHz).



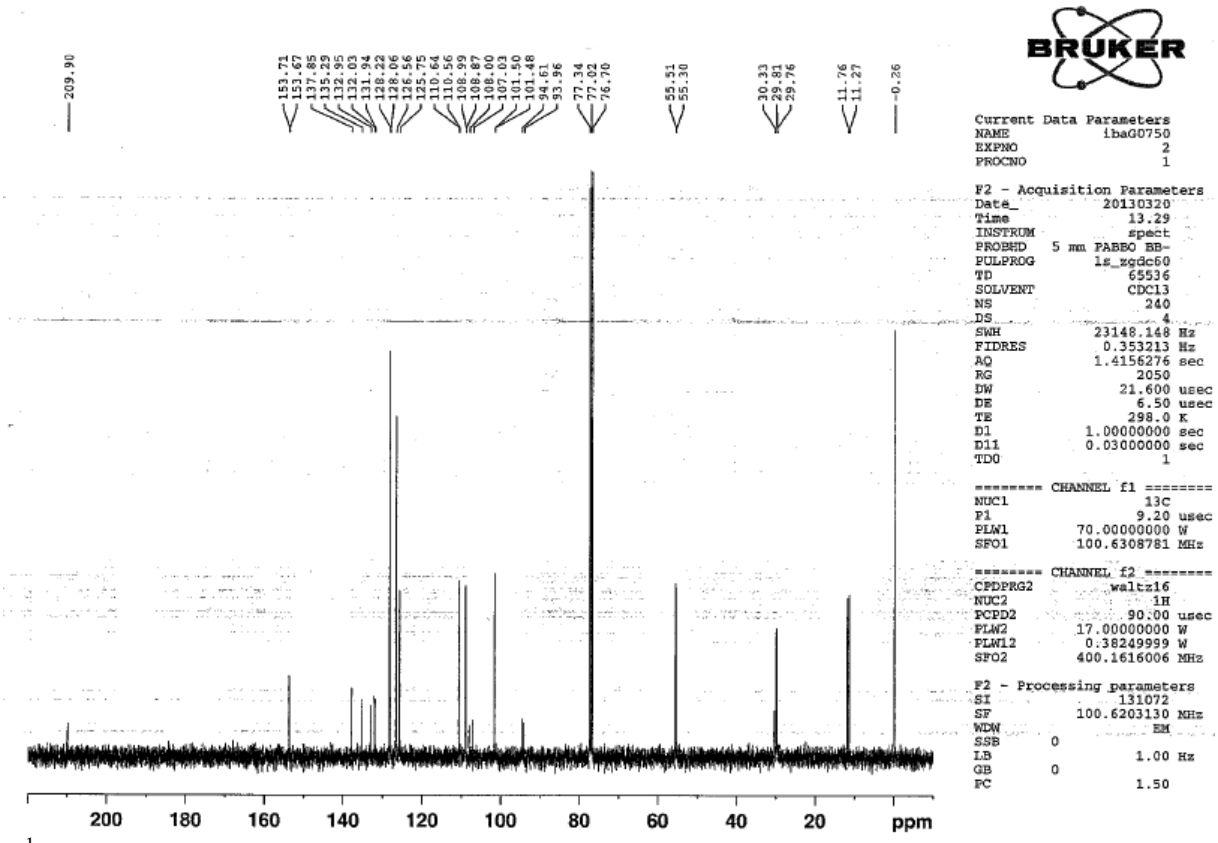
^{1}H NMR spectrum a few hours after purification of **1a**, evidencing a poor stability, which prevented recording of $^{13}C\{^{1}H\}$ NMR and MS spectra.

- 5-methoxy-3-[3-(5-methoxy-1,2-dimethyl-1*H*-indol-3-yl)-1-phenyl-3-(trimethylsilyl) propa-1,2-dien-1- yl]-1,2-dimethyl-1*H*-indole (**4**):

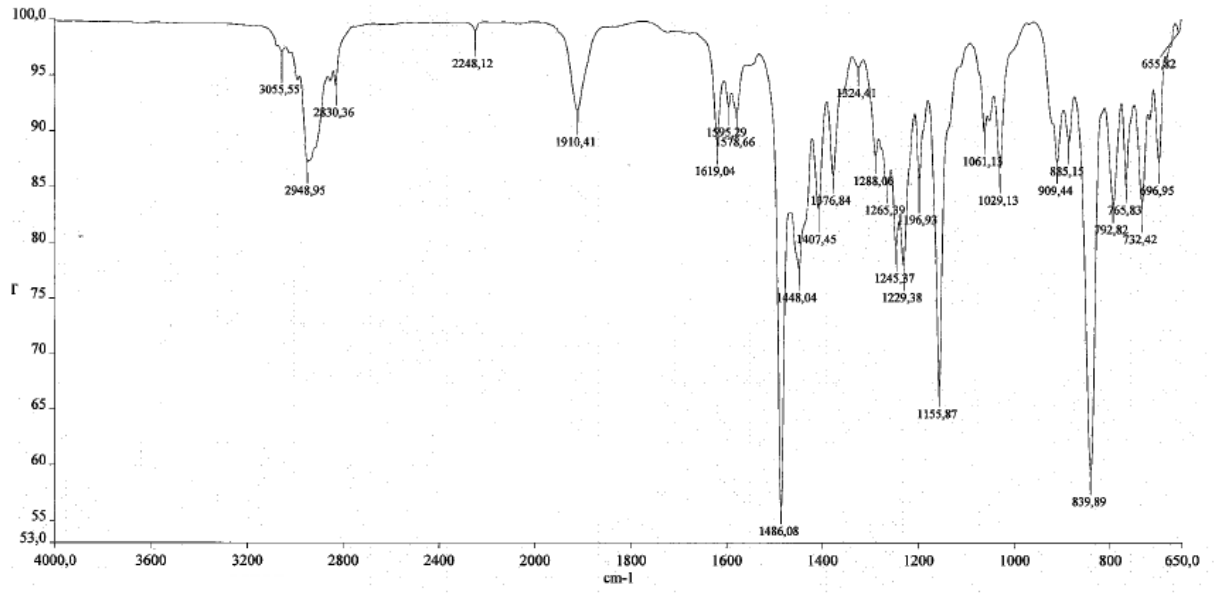




^{1}H NMR spectrum of **4** ($CDCl_3$, 400 MHz).



$^{13}C\{^{1}H\}$ NMR spectrum of **4** ($CDCl_3$, 100 MHz).



FT-IR spectrum of **4.**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
691 formula(e) evaluated with 7 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-10 O: 0-10 Si: 1-1

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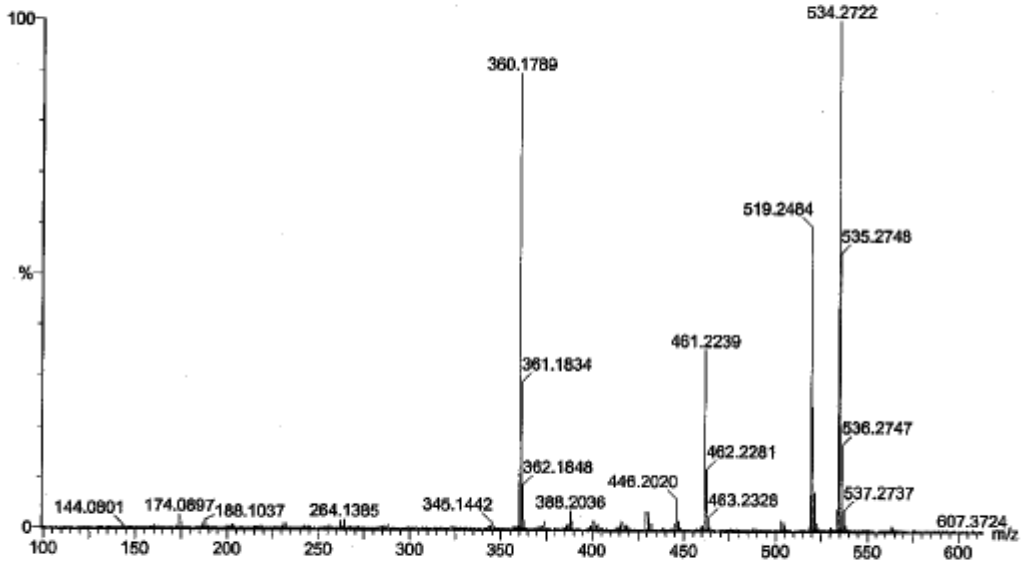

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Maximum:		1.3	5.0	50.0						
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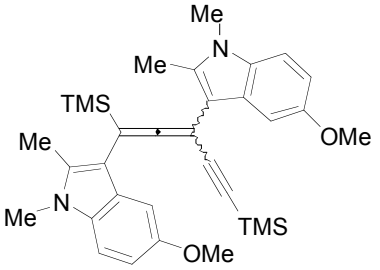
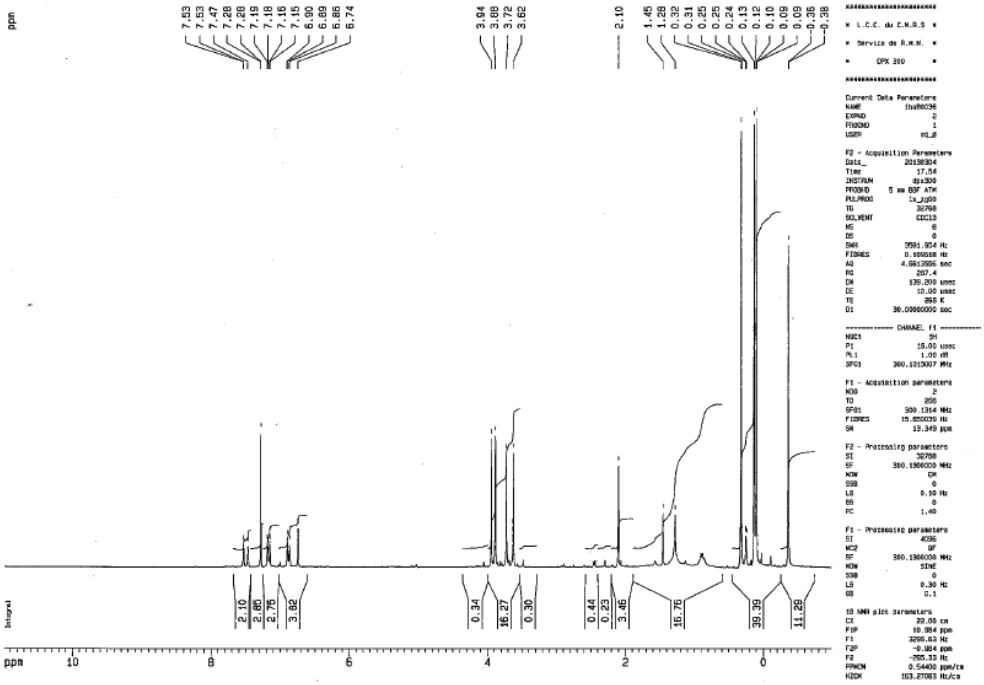
	534.2703	1.9	3.6	18.0	46.1	C34	H38	N2	O2	Si
	534.2748	-2.6	-4.9	9.5	175.8	C25	H40	N5	O6	Si
	534.2734	-1.2	-2.2	10.0	205.5	C23	H38	N8	O5	Si
	534.2735	-1.3	-2.4	4.5	215.5	C24	H44	N	O10	Si
	534.2721	0.1	0.2	5.0	248.2	C22	H42	N4	O9	Si
	534.2708	1.4	2.6	5.5	284.0	C20	H40	N7	O8	Si

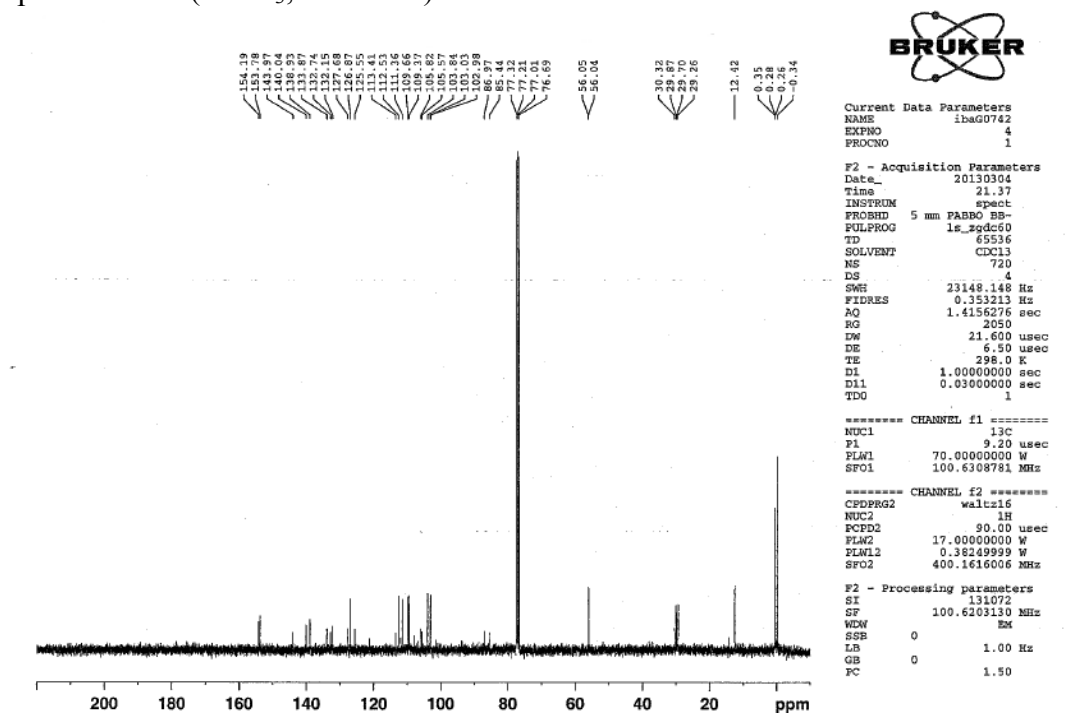
HRMS (DCI/CH_4) spectrum of **4.**

• 5-methoxy-3-[1-(5-methoxy-1,2-dimethyl-1*H*-indol-3-yl)-1,5-bis(trimethylsilyl) penta-1,2-dien-4-yn-3-yl]-1,2-dimethyl-1*H*-indole (**5**):



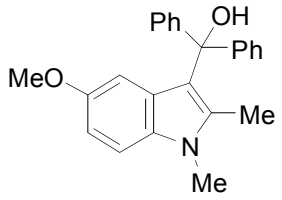


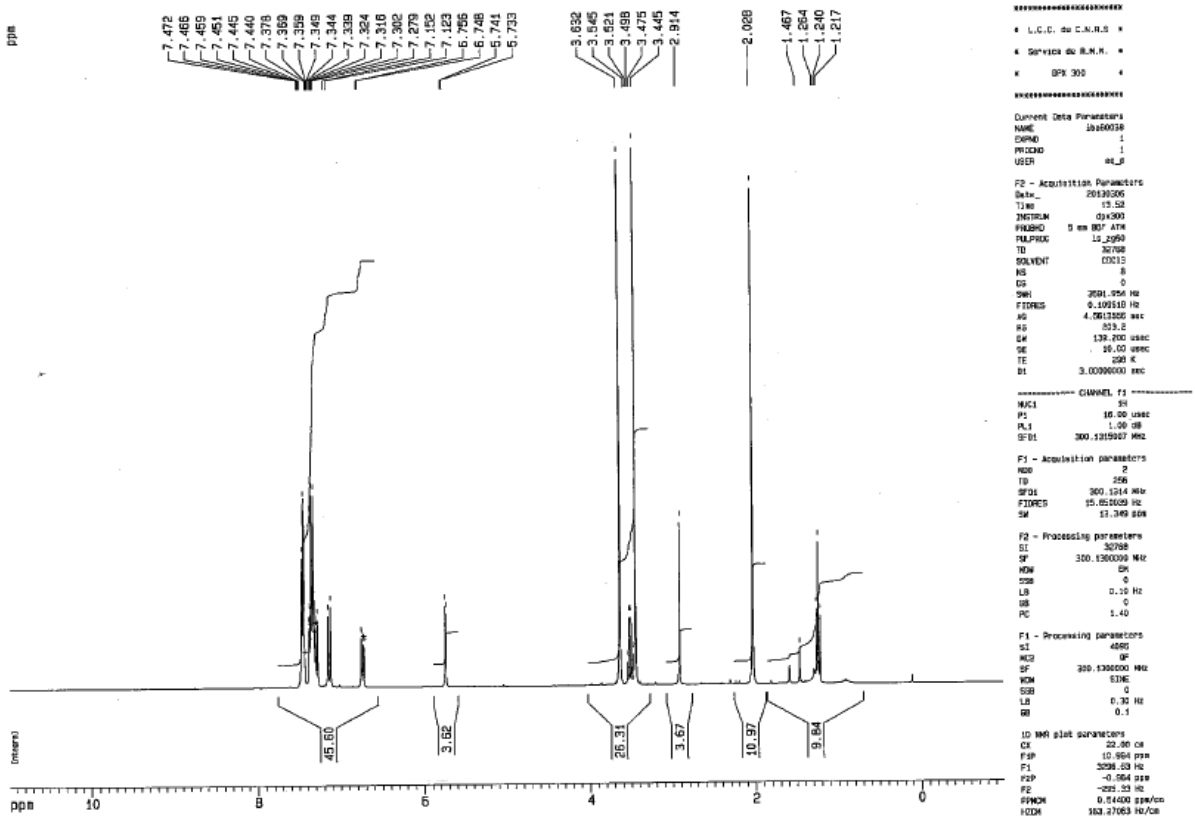
^{1}H NMR spectrum of **5** ($CDCl_3$, 300 MHz)



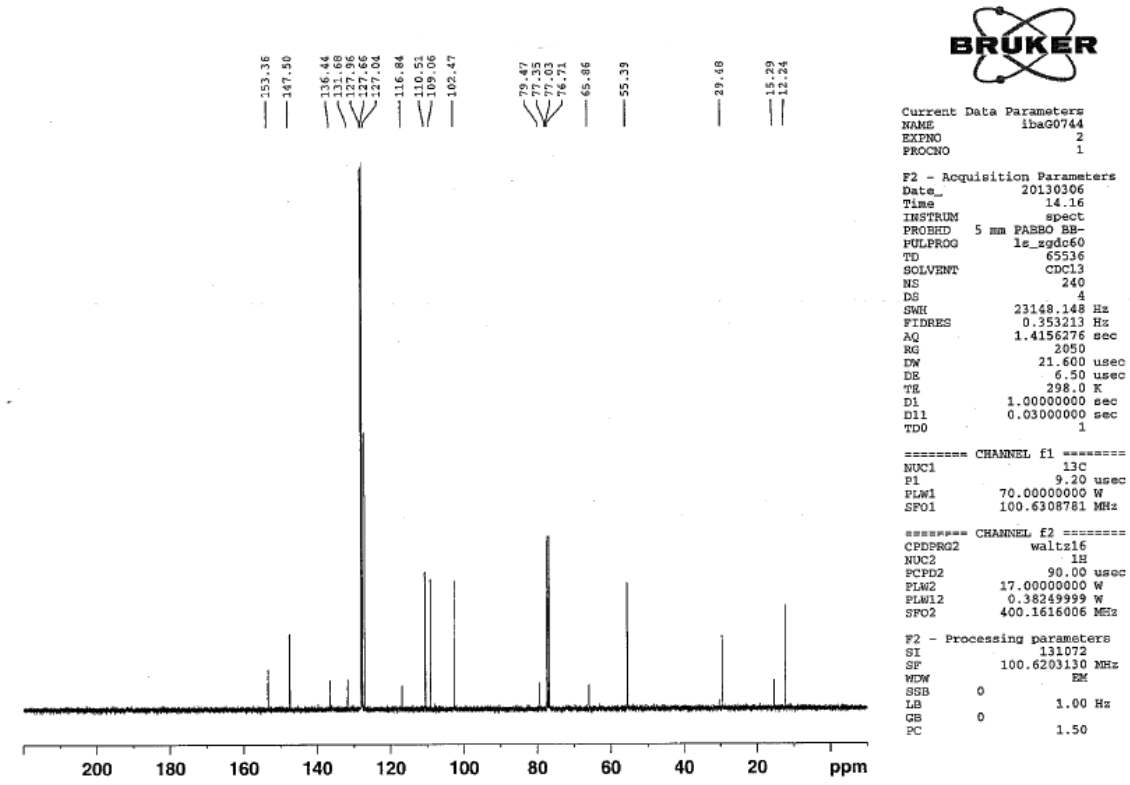
^{13}C{^{1}H} NMR spectrum of **5** ($CDCl_3$, 100 MHz).

• (5-methoxy-1,2-dimethyl-1*H*-indol-3- yl)diphenylmethanol (**6**) :





^{1}H NMR spectrum of **6** ($CDCl_3$, 300 MHz).



BRUKER

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PROCNO                       1

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SFO1             100.6308781 MHz

======== CHANNEL f2 ========
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LB                         1.00 Hz
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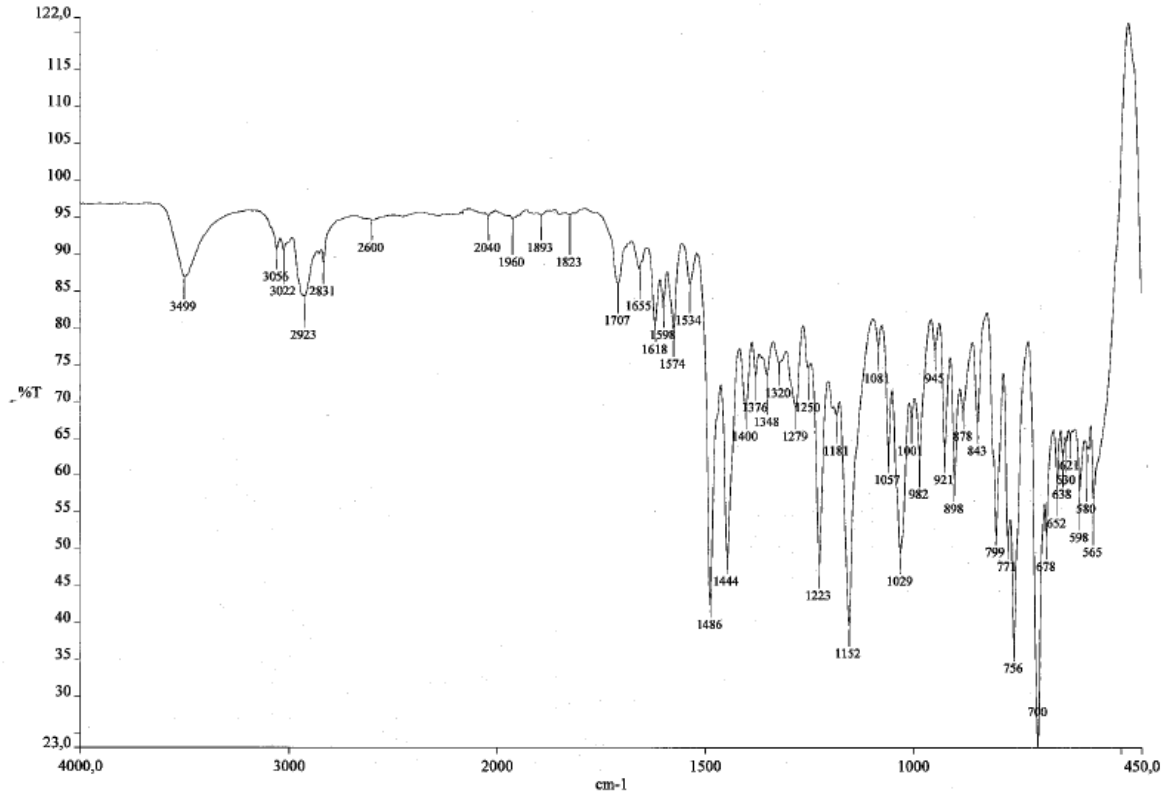
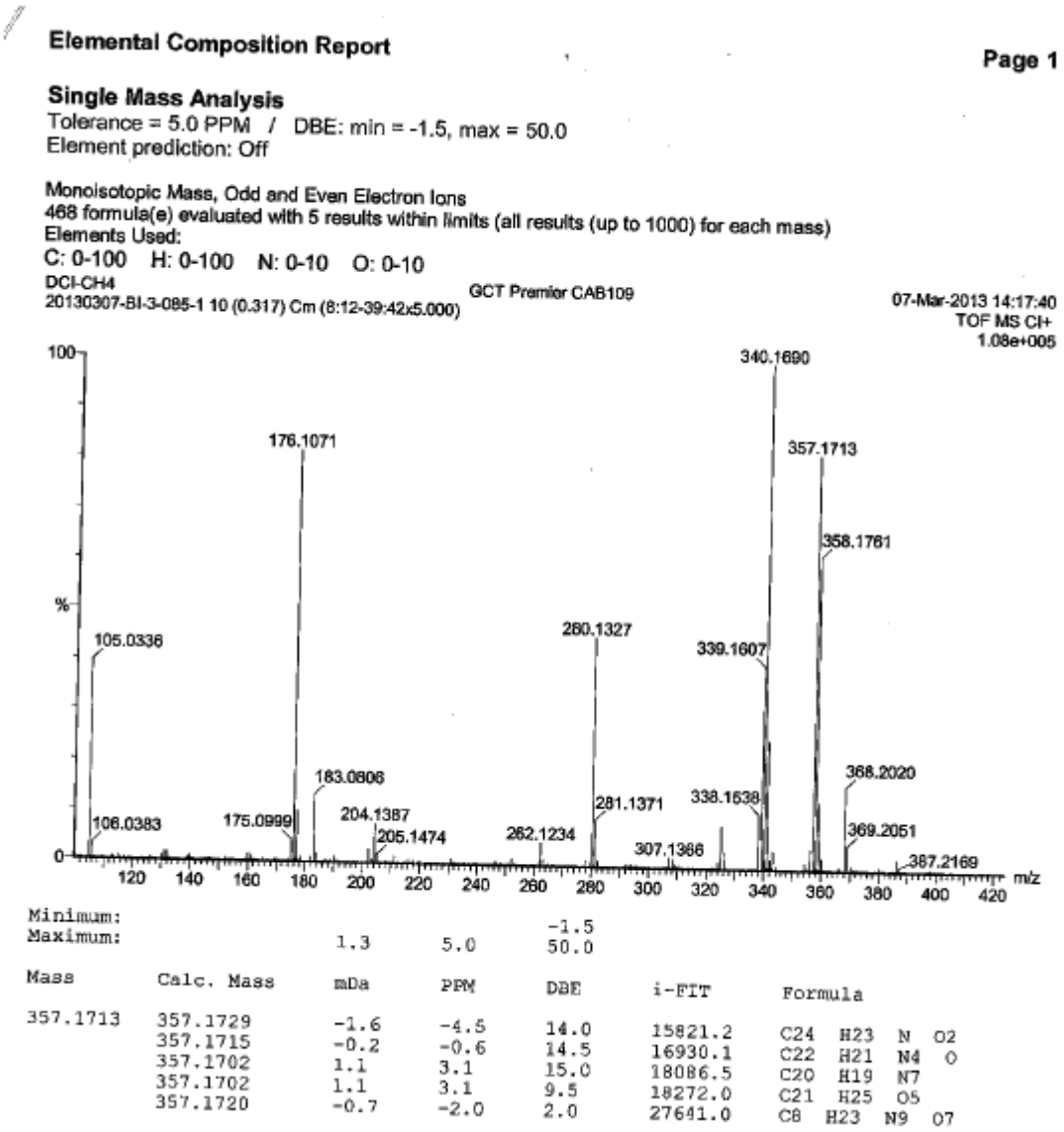
$^{13}C\{^{1}H\}$ NMR spectrum of **6** ($CDCl_3$, 100 MHz).



FT-IR spectrum of **6.**



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Maximum:		1.3	5.0	50.0		
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	357.1715	-0.2	-0.6	14.5	16930.1	C22 H21 N4 O
	357.1702	1.1	3.1	15.0	18086.5	C20 H19 N7
	357.1702	1.1	3.1	9.5	18272.0	C21 H25 O5
	357.1720	-0.7	-2.0	2.0	27641.0	C8 H23 N9 O7

HRMS (DCI/CH_4) spectrum of **6.**

5. Crystallographic data for **4**

Intensity data were collected at 100 K on a Gemini Agilent diffractometer using Cu Kα radiation source and equipped with an Oxford Cryosystems Cryostream Cooler Device. The structure was solved by SUPERFLIP,[4] and refined by full-matrix least-squares procedures on F, using the programs of the PC version of CRYSTALS.[5] Atomic scattering factors were taken from the International Tables for X-Ray Crystallography.[6] All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using a riding model. Absorption corrections were introduced using the MULTISCAN program.[7]

Crystal data for **4**: $C_{34}H_{38}N_2O_2Si$, $2(CH_2Cl_2)$, M = 704.64 g.mol^{-1}, Triclinic, a = 11.7650(4), b = 12.2770(4), c = 13.2538(7) Å, α = 84.895(3), β = 71.579(4), γ = 80.329(3)°, V = 1789.15(14) Å^3, T = 100 K, space group P-1, Z = 2, μ(Mo-Kα) = 3.594 mm^{-1}, 22420 reflections measured, 5593 unique (Rint = 0.022), 5048 reflections used in the calculations [I > 3σ(I)], 406 parameters, R1 = 0.0452, wR2 = 0.0581.

Crystal Data

a = 11.7650(4) Å	α = 84.895(3)°		
b = 12.2770(4) Å	β = 71.579(4)°		
c = 13.2538(7) Å	γ = 80.329(3)°		
Volume	1789.15(14) Å³	Crystal Class	triclinic
Space group	P -1	Z =	2
Formula	C_{36} H_{42} Cl_4 N_2 O_2 Si_1	M_r	704.64
Cell determined from	12806 reflections	Cell θ range =	4 - 62°
Temperature	100K		
Shape	block		
Colour	colorless	Size	0.10 × 0.20 × 0.20 mm
D_x	1.31	F000	740.000
μ	3.594 mm^{-1}		
Absorption correction	multi-scan		
T_{min}	0.49	T_{max}	0.70

Data Collection

Diffractometer	multi-scan
Scan type	φ and ω scans
Reflections measured	22420
Independent reflections	5593
Rint	0.0219
θ_{max}	61.9856
h =	-13 → 13
k =	-14 → 13
l =	-15 → 15

Refinement

$\Delta\rho_{min}$ =	-1.06 e Å^{-3}
$\Delta\rho_{max}$ =	1.15 e Å^{-3}
Reflections used	5048
Cutoff: I >	0.00σ(I)
Parameters refined	406
S =	1.09
R-factor	0.045
weighted R-factor	0.058
Δ/σ_{max}	0.0004
Refinement on	F
w =	$w' \times [1 - (\Delta F_{obs} / 6 \times \Delta F_{est})^2]^2$
w′ =	$[P_0T_0'(x) + P_1T_1'(x) + ...P_{n-1}T_{n-1}'(x)]^{-1}$, where P_i are the coefficients of a Chebychev series in $t_i(x)$, and $x = F_{calc}/F_{calcmax}$.
P_0 - P_{n-1} =	8.87 6.22 6.55 1.89

Parameters

Label	*x*	*y*	*z*	$U_{iso/equiv}$	Occupancy
N1	0.60050(18)	0.40486(16)	0.74768(16)	0.0262	1.0000
C2	0.4835(2)	0.38568(19)	0.80105(18)	0.0235	1.0000
C3	0.4560(2)	0.30404(17)	0.75204(17)	0.0193	1.0000
C4	0.56102(19)	0.27008(18)	0.66430(17)	0.0203	1.0000
C5	0.6490(2)	0.33500(19)	0.66338(18)	0.0243	1.0000
C6	0.7627(2)	0.3236(2)	0.5862(2)	0.0291	1.0000
C7	0.7863(2)	0.2465(2)	0.51044(19)	0.0297	1.0000
C8	0.6992(2)	0.18077(19)	0.50987(18)	0.0259	1.0000
C9	0.5863(2)	0.19131(18)	0.58606(17)	0.0205	1.0000
C10	0.6663(3)	0.4821(2)	0.7750(2)	0.0365	1.0000
C11	0.4051(2)	0.4523(2)	0.89332(19)	0.0303	1.0000
C12	0.33849(19)	0.25991(17)	0.78147(16)	0.0180	1.0000

Si13	0.30402(5)	0.14833(5)	0.88958(4)	0.0198	1.0000
C14	0.4351(2)	0.0350(2)	0.8564(2)	0.0350	1.0000
O15	0.73649(15)	0.10768(14)	0.42851(13)	0.0325	1.0000
C16	0.6518(2)	0.0400(2)	0.4228(2)	0.0320	1.0000
C17	0.26286(19)	0.29248(17)	0.72647(16)	0.0184	1.0000
C18	0.18767(18)	0.32298(17)	0.67043(16)	0.0177	1.0000
C19	0.20774(19)	0.26620(17)	0.56991(16)	0.0178	1.0000
C20	0.13813(19)	0.19337(17)	0.55450(17)	0.0197	1.0000
N21	0.18616(17)	0.15737(15)	0.45146(15)	0.0217	1.0000
C22	0.2891(2)	0.20467(17)	0.40123(17)	0.0207	1.0000
C23	0.30548(19)	0.27385(17)	0.47340(17)	0.0181	1.0000
C24	0.4063(2)	0.33000(17)	0.44404(17)	0.0192	1.0000
C25	0.4888(2)	0.31334(18)	0.34429(18)	0.0229	1.0000
C26	0.4718(2)	0.24376(19)	0.27283(17)	0.0248	1.0000
C27	0.3724(2)	0.18996(18)	0.29969(17)	0.0236	1.0000
C28	0.1605(2)	0.1009(2)	0.8928(2)	0.0335	1.0000
C29	0.2891(2)	0.2053(2)	1.01955(18)	0.0301	1.0000
C30	0.08814(19)	0.41836(17)	0.70155(17)	0.0186	1.0000
C31	0.0508(2)	0.48605(18)	0.62428(18)	0.0223	1.0000
C32	-0.0381(2)	0.57757(19)	0.65229(19)	0.0246	1.0000
C33	-0.0933(2)	0.60179(19)	0.7586(2)	0.0274	1.0000
C34	-0.0583(2)	0.5340(2)	0.83655(19)	0.0288	1.0000
C35	0.0323(2)	0.44336(19)	0.80821(18)	0.0247	1.0000
C36	0.0339(2)	0.14862(19)	0.63308(19)	0.0255	1.0000
C37	0.1422(2)	0.0774(2)	0.4045(2)	0.0304	1.0000
O38	0.59325(15)	0.36061(13)	0.30607(13)	0.0290	1.0000
C39	0.6109(2)	0.43751(19)	0.37229(19)	0.0275	1.0000
C40	0.8649(2)	0.2580(3)	0.2015(2)	0.0434	1.0000
Cl41	0.97410(6)	0.13819(6)	0.19157(5)	0.0412	1.0000
Cl42	0.87314(7)	0.32114(7)	0.07480(6)	0.0505	1.0000
C43	0.7643(4)	0.2041(3)	-0.1249(3)	0.0789	1.0000

Cl44	0.78371(10)	0.07113(7)	0.15267(10)	0.0897	1.0000
Cl45	0.63500(6)	0.25025(6)	0.02114(5)	0.0420	1.0000
H61	0.8221(2)	0.3683(2)	0.5867(2)	0.0367	1.0000
H71	0.8624(2)	0.2364(2)	0.45556(19)	0.0379	1.0000
H91	0.5259(2)	0.14705(18)	0.58625(17)	0.0271	1.0000
H102	0.6994(3)	0.5283(2)	0.7132(2)	0.0584	1.0000
H101	0.7309(3)	0.4430(2)	0.7999(2)	0.0586	1.0000
H103	0.6108(3)	0.5284(2)	0.8308(2)	0.0587	1.0000
H113	0.3247(2)	0.4301(2)	0.91499(19)	0.0471	1.0000
H111	0.4388(2)	0.4397(2)	0.95211(19)	0.0471	1.0000
H112	0.3991(2)	0.5308(2)	0.87305(19)	0.0471	1.0000
H141	0.4239(2)	-0.0212(2)	0.9134(2)	0.0540	1.0000
H142	0.5093(2)	0.0657(2)	0.8480(2)	0.0541	1.0000
H143	0.4401(2)	0.0036(2)	0.7903(2)	0.0546	1.0000
H162	0.6892(2)	-0.0065(2)	0.3612(2)	0.0486	1.0000
H163	0.6286(2)	-0.0074(2)	0.4874(2)	0.0486	1.0000
H161	0.5787(2)	0.0858(2)	0.4128(2)	0.0485	1.0000
H241	0.4184(2)	0.37628(17)	0.49217(17)	0.0242	1.0000
H261	0.5309(2)	0.23434(19)	0.20382(17)	0.0311	1.0000
H271	0.3602(2)	0.14421(18)	0.25106(17)	0.0301	1.0000
H282	0.1443(2)	0.0433(2)	0.9469(2)	0.0523	1.0000
H281	0.0948(2)	0.1622(2)	0.9098(2)	0.0525	1.0000
H283	0.1692(2)	0.0737(2)	0.8235(2)	0.0526	1.0000
H291	0.2761(2)	0.1474(2)	1.07442(18)	0.0470	1.0000
H293	0.3619(2)	0.2344(2)	1.01597(18)	0.0470	1.0000
H292	0.2192(2)	0.2629(2)	1.03602(18)	0.0473	1.0000
H311	0.0880(2)	0.46898(18)	0.55035(18)	0.0282	1.0000
H321	-0.0620(2)	0.62351(19)	0.59807(19)	0.0317	1.0000
H331	-0.1547(2)	0.66453(19)	0.7777(2)	0.0343	1.0000
H341	-0.0965(2)	0.5495(2)	0.91049(19)	0.0373	1.0000
H351	0.0574(2)	0.39752(19)	0.86239(18)	0.0312	1.0000

H362	-0.0358(2)	0.16056(19)	0.60760(19)	0.0409	1.0000
H361	0.0548(2)	0.06966(19)	0.64440(19)	0.0410	1.0000
H363	0.0139(2)	0.18518(19)	0.69995(19)	0.0410	1.0000
H373	0.1329(2)	0.1061(2)	0.3376(2)	0.0488	1.0000
H371	0.1980(2)	0.0106(2)	0.3921(2)	0.0493	1.0000
H372	0.0648(2)	0.0611(2)	0.4488(2)	0.0493	1.0000
H391	0.6872(2)	0.46501(19)	0.33450(19)	0.0440	1.0000
H392	0.6170(2)	0.40154(19)	0.43941(19)	0.0436	1.0000
H393	0.5445(2)	0.49918(19)	0.38561(19)	0.0437	1.0000
H401	0.8819(2)	0.3129(3)	0.2451(2)	0.0545	1.0000
H402	0.7836(2)	0.2374(3)	0.2341(2)	0.0544	1.0000
H431	0.8353(4)	0.2166(3)	-0.1073(3)	0.1041	1.0000
H432	0.7621(4)	0.2482(3)	-0.1896(3)	0.1030	1.0000

Thermal Parameters

Label	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	0.0285(10)	0.0249(10)	0.0299(11)	0.0008(8)	-0.0129(9)	-0.0101(8)
C2	0.0287(12)	0.0216(11)	0.0228(11)	0.0009(9)	-0.0121(10)	-0.0033(9)
C3	0.0219(11)	0.0180(10)	0.0180(11)	0.0010(8)	-0.0068(9)	-0.0019(8)
C4	0.0196(11)	0.0199(11)	0.0207(11)	0.0035(9)	-0.0074(9)	-0.0011(9)
C5	0.0236(11)	0.0248(12)	0.0264(12)	0.0043(9)	-0.0113(10)	-0.0047(9)
C6	0.0220(12)	0.0326(13)	0.0339(13)	0.0124(11)	-0.0109(10)	-0.0095(10)
C7	0.0184(11)	0.0357(13)	0.0284(13)	0.0089(11)	-0.0023(10)	-0.0008(10)
C8	0.0253(12)	0.0253(12)	0.0216(12)	0.0027(9)	-0.0042(9)	0.0037(9)
C9	0.0217(11)	0.0192(11)	0.0189(11)	0.0041(9)	-0.0063(9)	-0.0012(9)
C10	0.0415(15)	0.0320(14)	0.0468(16)	0.0024(12)	-0.0254(13)	-0.0139(11)
C11	0.0419(14)	0.0234(12)	0.0275(13)	-0.0051(10)	-0.0128(11)	-0.0041(10)
C12	0.0190(10)	0.0192(10)	0.0144(10)	-0.0032(8)	-0.0040(8)	-0.0002(8)
Si13	0.0226(3)	0.0192(3)	0.0157(3)	-0.0015(2)	-0.0034(2)	-0.0022(2)
C14	0.0389(14)	0.0257(13)	0.0307(13)	0.0023(10)	-0.0036(11)	0.0060(11)
O15	0.0303(9)	0.0344(9)	0.0240(9)	-0.0036(7)	0.0013(7)	0.0015(7)

C16	0.0355(14)	0.0309(13)	0.0256(13)	-0.0059(10)	-0.0071(11)	0.0040(11)
C17	0.0192(11)	0.0167(10)	0.0159(10)	-0.0037(8)	0.0011(9)	-0.0044(8)
C18	0.0168(10)	0.0192(10)	0.0166(10)	0.0004(8)	-0.0031(8)	-0.0055(8)
C19	0.0181(10)	0.0172(10)	0.0175(11)	-0.0006(8)	-0.0059(8)	0.0000(8)
C20	0.0208(11)	0.0188(11)	0.0200(11)	0.0008(8)	-0.0091(9)	0.0006(9)
N21	0.0257(10)	0.0201(9)	0.0226(10)	-0.0042(7)	-0.0118(8)	-0.0023(8)
C22	0.0246(11)	0.0159(10)	0.0214(11)	-0.0006(8)	-0.0096(9)	0.0027(9)
C23	0.0218(11)	0.0151(10)	0.0172(10)	-0.0001(8)	-0.0080(9)	0.0018(8)
C24	0.0235(11)	0.0167(10)	0.0168(10)	-0.0004(8)	-0.0067(9)	-0.0006(8)
C25	0.0235(11)	0.0174(11)	0.0231(11)	0.0025(9)	-0.0034(9)	0.0010(9)
C26	0.0303(12)	0.0226(11)	0.0153(11)	0.0001(9)	-0.0023(9)	0.0037(9)
C27	0.0335(13)	0.0192(11)	0.0177(11)	-0.0046(9)	-0.0111(10)	0.0054(9)
C28	0.0363(14)	0.0398(14)	0.0260(13)	0.0032(11)	-0.0071(11)	-0.0168(11)
C29	0.0402(14)	0.0317(13)	0.0180(11)	0.0000(10)	-0.0072(10)	-0.0078(11)
C30	0.0171(10)	0.0185(11)	0.0213(11)	-0.0031(9)	-0.0057(9)	-0.0054(8)
C31	0.0235(11)	0.0229(11)	0.0208(11)	-0.0011(9)	-0.0069(9)	-0.0042(9)
C32	0.0259(12)	0.0209(11)	0.0295(13)	-0.0006(9)	-0.0128(10)	-0.0021(9)
C33	0.0227(11)	0.0249(12)	0.0358(14)	-0.0097(10)	-0.0111(10)	0.0020(9)
C34	0.0280(12)	0.0337(13)	0.0220(12)	-0.0097(10)	-0.0051(10)	0.0024(10)
C35	0.0255(11)	0.0285(12)	0.0199(11)	-0.0020(9)	-0.0080(9)	-0.0012(9)
C36	0.0235(12)	0.0260(12)	0.0294(12)	-0.0022(10)	-0.0095(10)	-0.0068(9)
C37	0.0366(13)	0.0272(12)	0.0331(13)	-0.0078(10)	-0.0166(11)	-0.0057(10)
O38	0.0271(9)	0.0268(9)	0.0254(9)	-0.0008(7)	0.0031(7)	-0.0052(7)
C39	0.0263(12)	0.0254(12)	0.0273(12)	0.0024(10)	-0.0037(10)	-0.0052(10)
C40	0.0292(14)	0.0555(18)	0.0384(15)	-0.0033(13)	-0.0037(12)	0.0011(12)
Cl41	0.0356(4)	0.0408(4)	0.0379(4)	0.0005(3)	-0.0009(3)	-0.0019(3)
Cl42	0.0524(4)	0.0587(5)	0.0443(4)	-0.0023(3)	-0.0253(3)	0.0018(3)
C43	0.096(3)	0.0443(19)	0.059(2)	-0.0116(17)	0.032(2)	-0.0118(19)
Cl44	0.0753(6)	0.0388(5)	0.1102(8)	-0.0216(5)	0.0418(6)	-0.0131(4)
Cl45	0.0442(4)	0.0456(4)	0.0287(3)	-0.0032(3)	-0.0048(3)	0.0030(3)

Distances

N1	C2	1.384(3)Å		N1	C5	1.383(3)Å
N1	C10	1.454(3)Å		C2	C3	1.374(3)Å
C2	C11	1.493(3)Å		C3	C4	1.434(3)Å
C3	C12	1.494(3)Å		C4	C5	1.405(3)Å
C4	C9	1.409(3)Å		C5	C6	1.398(3)Å
C6	C7	1.374(4)Å		C6	H61	0.961Å
C7	C8	1.409(4)Å		C7	H71	0.955Å
C8	C9	1.385(3)Å		C8	O15	1.379(3)Å
C9	H91	0.964Å		C10	H102	0.967Å
C10	H101	0.961Å		C10	H103	0.970Å
C11	H113	0.976Å		C11	H111	0.970Å
C11	H112	0.974Å		C12	Si13	1.888(2)Å
C12	C17	1.312(3)Å		Si13	C14	1.862(2)Å
Si13	C28	1.864(3)Å		Si13	C29	1.862(2)Å
C14	H141	0.968Å		C14	H142	0.978Å
C14	H143	0.969Å		O15	C16	1.423(3)Å
C16	H162	0.979Å		C16	H163	0.981Å
C16	H161	0.985Å		C17	C18	1.315(3)Å
C18	C19	1.493(3)Å		C18	C30	1.494(3)Å
C19	C20	1.377(3)Å		C19	C23	1.433(3)Å
C20	N21	1.384(3)Å		C20	C36	1.486(3)Å
N21	C22	1.380(3)Å		N21	C37	1.450(3)Å
C22	C23	1.411(3)Å		C22	C27	1.398(3)Å
C23	C24	1.403(3)Å		C24	C25	1.379(3)Å
C24	H241	0.948Å		C25	C26	1.410(3)Å
C25	O38	1.377(3)Å		C26	C27	1.373(3)Å
C26	H261	0.961Å		C27	H271	0.950Å
C28	H282	0.958Å		C28	H281	0.969Å
C28	H283	0.974Å		C29	H291	0.965Å
C29	H293	0.969Å		C29	H292	0.971Å
C30	C31	1.394(3)Å		C30	C35	1.395(3)Å

C31	C32	1.388(3)Å		C31	H311	0.965Å
C32	C33	1.389(3)Å		C32	H321	0.957Å
C33	C34	1.389(4)Å		C33	H331	0.955Å
C34	C35	1.392(3)Å		C34	H341	0.961Å
C35	H351	0.962Å		C36	H362	0.966Å
C36	H361	0.970Å		C36	H363	0.973Å
C37	H373	0.957Å		C37	H371	0.951Å
C37	H372	0.956Å		O38	C39	1.426(3)Å
C39	H391	0.978Å		C39	H392	0.975Å
C39	H393	0.977Å		C40	Cl41	1.768(3)Å
C40	Cl42	1.767(3)Å		C40	H401	1.008Å
C40	H402	0.982Å		C43	Cl44	1.668(4)Å
C43	Cl45	1.747(3)Å		C43	H431	0.974Å
C43	H432	0.978Å				

Angles

C2	N1	C5	108.75(18)°		C2	N1	C10	127.1(2)°
C5	N1	C10	124.1(2)°		N1	C2	C3	109.3(2)°
N1	C2	C11	121.4(2)°		C3	C2	C11	129.2(2)°
C2	C3	C4	107.16(19)°		C2	C3	C12	127.1(2)°
C4	C3	C12	125.67(19)°		C3	C4	C5	106.91(19)°
C3	C4	C9	133.0(2)°		C5	C4	C9	120.0(2)°
N1	C5	C4	107.92(19)°		N1	C5	C6	130.5(2)°
C4	C5	C6	121.6(2)°		C5	C6	C7	117.8(2)°
C5	C6	H61	120.725°		C7	C6	H61	121.442°
C6	C7	C8	121.4(2)°		C6	C7	H71	120.858°
C8	C7	H71	117.704°		C7	C8	C9	121.2(2)°
C7	C8	O15	114.4(2)°		C9	C8	O15	124.4(2)°
C4	C9	C8	117.9(2)°		C4	C9	H91	120.170°
C8	C9	H91	121.907°		N1	C10	H102	109.602°
N1	C10	H101	110.570°		H102	C10	H101	109.467°
N1	C10	H103	108.932°		H102	C10	H103	109.059°

H101	C10	H103	109.184°		C2	C11	H113	108.189°
C2	C11	H111	110.382°		H113	C11	H111	109.888°
C2	C11	H112	110.202°		H113	C11	H112	109.594°
H111	C11	H112	108.580°		C3	C12	Si13	120.34(15)°
C3	C12	C17	119.84(19)°		Si13	C12	C17	119.60(16)°
C12	Si13	C14	106.89(10)°		C12	Si13	C28	108.61(11)°
C14	Si13	C28	111.95(13)°		C12	Si13	C29	109.15(10)°
C14	Si13	C29	109.46(12)°		C28	Si13	C29	110.67(12)°
Si13	C14	H141	109.317°		Si13	C14	H142	108.807°
H141	C14	H142	110.026°		Si13	C14	H143	108.945°
H141	C14	H143	110.127°		H142	C14	H143	109.591°
C8	O15	C16	117.09(18)°		O15	C16	H162	108.521°
O15	C16	H163	111.219°		H162	C16	H163	109.118°
O15	C16	H161	110.647°		H162	C16	H161	108.019°
H163	C16	H161	109.247°		C12	C17	C18	178.7(2)°
C17	C18	C19	119.18(19)°		C17	C18	C30	121.15(19)°
C19	C18	C30	119.54(18)°		C18	C19	C20	126.09(19)°
C18	C19	C23	126.37(19)°		C20	C19	C23	107.49(18)°
C19	C20	N21	109.01(19)°		C19	C20	C36	128.7(2)°
N21	C20	C36	122.1(2)°		C20	N21	C22	108.83(18)°
C20	N21	C37	126.5(2)°		C22	N21	C37	124.59(19)°
N21	C22	C23	108.13(19)°		N21	C22	C27	130.7(2)°
C23	C22	C27	121.2(2)°		C19	C23	C22	106.51(19)°
C19	C23	C24	133.2(2)°		C22	C23	C24	120.3(2)°
C23	C24	C25	118.0(2)°		C23	C24	H241	120.905°
C25	C24	H241	121.094°		C24	C25	C26	121.3(2)°
C24	C25	O38	124.2(2)°		C26	C25	O38	114.44(19)°
C25	C26	C27	121.3(2)°		C25	C26	H261	119.139°
C27	C26	H261	119.525°		C22	C27	C26	117.9(2)°
C22	C27	H271	120.760°		C26	C27	H271	121.331°
Si13	C28	H282	108.210°		Si13	C28	H281	109.362°
H282	C28	H281	109.526°		Si13	C28	H283	109.461°
H282	C28	H283	110.601°		H281	C28	H283	109.653°

Si13	C29	H291	109.469°		Si13	C29	H293	110.401°
H291	C29	H293	109.075°		Si13	C29	H292	108.060°
H291	C29	H292	109.050°		H293	C29	H292	110.761°
C18	C30	C31	120.63(19)°		C18	C30	C35	120.96(19)°
C31	C30	C35	118.4(2)°		C30	C31	C32	121.0(2)°
C30	C31	H311	119.102°		C32	C31	H311	119.927°
C31	C32	C33	120.2(2)°		C31	C32	H321	119.794°
C33	C32	H321	119.974°		C32	C33	C34	119.4(2)°
C32	C33	H331	120.059°		C34	C33	H331	120.536°
C33	C34	C35	120.3(2)°		C33	C34	H341	120.156°
C35	C34	H341	119.559°		C30	C35	C34	120.7(2)°
C30	C35	H351	119.296°		C34	C35	H351	120.008°
C20	C36	H362	110.798°		C20	C36	H361	109.914°
H362	C36	H361	107.918°		C20	C36	H363	109.043°
H362	C36	H363	109.575°		H361	C36	H363	109.576°
N21	C37	H373	109.985°		N21	C37	H371	110.820°
H373	C37	H371	107.799°		N21	C37	H372	111.671°
H373	C37	H372	107.304°		H371	C37	H372	109.123°
C25	O38	C39	116.76(17)°		O38	C39	H391	107.524°
O38	C39	H392	110.774°		H391	C39	H392	108.681°
O38	C39	H393	110.073°		H391	C39	H393	109.522°
H392	C39	H393	110.208°		Cl41	C40	Cl42	111.13(15)°
Cl41	C40	H401	108.704°		Cl42	C40	H401	107.655°
Cl41	C40	H402	109.141°		Cl42	C40	H402	108.637°
H401	C40	H402	111.583°		Cl44	C43	Cl45	116.1(2)°
Cl44	C43	H431	107.523°		Cl45	C43	H431	108.567°
Cl44	C43	H432	107.767°		Cl45	C43	H432	108.743°
H431	C43	H432	107.879°					

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