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CARBON-13 NMR PARAMETERS AND MOLECULAR STRUCTURE OF {2-*exo*-BICYCLO[2.2.1]HEPT-*exo*-3-ACETATO-5-ENYL}-5,10,15,20-TETRAPHENYLPORPHINATOTHALLIUM(III)

F. BRADY, K. HENRICK and R.W. MATTHEWS *

Department of Chemistry, The Polytechnic of North London, Holloway, London N7 8DB (Great Britain)

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Summary

The compound {2-*exo*-bicyclo[2.2.1]hept-*exo*-3-acetato-5-enyl}-5,10,15,20-tetraphenylporphinatothallium(III) has been synthesized and its molecular structure determined. The compound crystallizes in the monoclinic space group $P2_1$, with a 13.112, b 29.193, c 11.249 Å, β 102.17°, $Z = 4$. The organothallium(III) group is situated above the plane of the porphyrin ring with the thallium atom displaced 0.9 Å from the N_4 plane. The thallium atom and the acetato group are attached *cis-exo* to the bicyclo[2.2.1]unit. Carbon-13 spectra show shielding effects arising from the porphyrin ring current.

Introduction

Monoalkylthallium(III) complexes with multidentate ligands (e.g. porphyrins) have been synthesized [1,2] in an effort to restrict coordination of solvent molecules at thallium and thus facilitate NMR studies of solvent interactions with organothallium(III) compounds. However, the low solubility of the CH_3-Tl^{2+} derivative of tetraphenylporphyrin ($TPPH_2$) (i.e. MeTlTPP) and other thallium-porphyrin derivatives [1] presented an obstacle in searching for ^{205}Tl NMR signals from this type of complex. Anticipating that the solubility problem could be overcome by increasing the size and complexity of the organo group attached to thallium, the complex {2-*exo*-bicyclo[2.2.1]hept-*exo*-3-acetato-5-enyl}-5,10,15,20-tetraphenylporphinatothallium(III), NORTlTPP, was prepared. Characterization of the complex by ^{13}C NMR involved consideration of the effect of the porphyrin ring currents [3a] on NMR parameters of the norbornenyl group and the relationship these effects might have to the relative positions of this group and the porphyrin ring. For this reason, and to determine the effect of a bulky organic group on the position of Tl relative to the

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porphyrin ring, the molecular structure was determined by single crystal X-ray analysis.

Experimental

Preparation of {2-exo-bicyclo[2.2.1]hept-exo-3-acetato-5-enyl}diacetatothallium(III) (NORTl(OAc)$_2$).

This compound was prepared as previously described [4]. Anal. Found: C, 33.0; H, 3.8. Calcd. for $C_{13}H_{12}O_6Tl$: C, 32.9; H, 4.0%. The ^{13}C NMR spectrum in dimethylsulphoxide-d_6 solution (below) is similar to that reported for this compound in $CDCl_3$ solution [5,6]. ^{13}C NMR: δ 44.6 ppm (C(1), ^{2}J(Tl—C) = 221 Hz); 67.6 ppm (C(2), ^{1}J(^{205}Tl—C) = 6464 Hz); 74.3 ppm (C(3), ^{2}J(Tl—C) = 419 Hz); 47.9 ppm (C(4), ^{3}J(Tl—C) $<$ 5 Hz); 134.2 ppm (C(5), ^{4}J(Tl—C) = 110 Hz); 141.5 ppm (C(6), ^{3}J(Tl—C) = 1020 Hz); 46.6 ppm (C(7), ^{3}J(Tl—C) = 18 Hz); 174.9 ppm ($CH_3\mathit{C}O_2$); 22.9 ppm ($\mathit{C}H_3CO_2$). (The norbornenyl group is labelled as shown in Fig. 1.)

Preparation of NORTlTPP

$TPPH_2$ (305 mg, 0.5 mmol) was dissolved with heating in chloroform (50 cm^3). After cooling, NORTl(OAc)$_2$ (240 mg, 0.5 mmol) in chloroform (25 cm^3) was added and the solution was set aside for 1 h. After concentration to ca. 10 cm^3 and addition of n-pentane (20 cm^3), the green solution was left to stand overnight at —5° C. Crystals of NORTlTPP were obtained as blue rods and collected by filtration; m.p. >300°C (370 mg, 76%). Anal. Found: C, 67.1; H, 4.6; N, 5.6. Calcd. for $C_{55}H_{39}N_4O_2Tl$: C, 67.0; H, 4.9; N, 5.3%.

^{13}C NMR spectra

^{13}C NMR spectra were obtained at 22.63 and 45.28 MHz on Bruker HX 90E and WH 180 spectrometers, respectively.

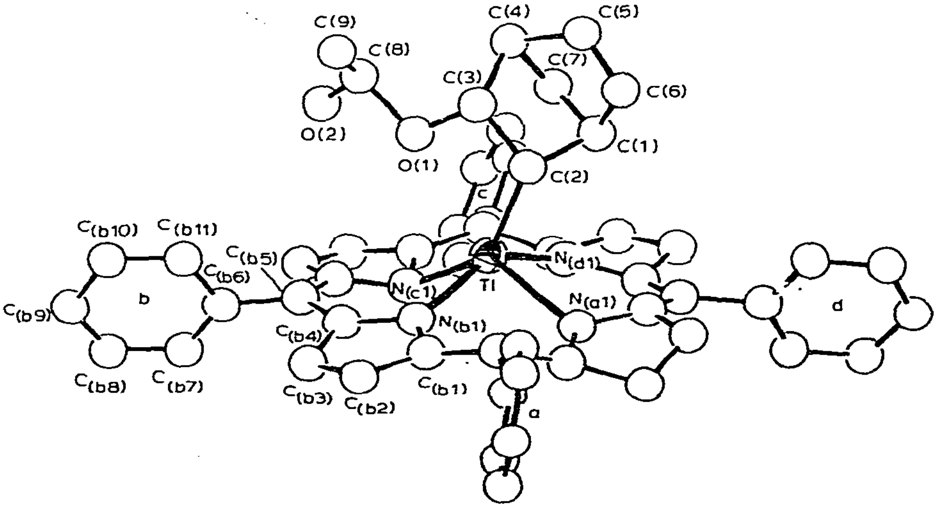

Fig. 1. The structure of NORTlTPP (molecule 1).

Crystal data

$C_{53}H_{39}N_4O_2Tl$, M 967.3, monoclinic, $P2_1$, $a = 13.112(2)$, $b = 29.193(3)$, $c = 11.249(4)$ Å, $\beta = 102.17(3)°$, $V = 4209$ Å^3, $Z = 4$, $D_c = 1.526$ g cm^{-3}, $\mu(\text{Mo-}K_\alpha) = 37.13$ cm^{-1}, $F(000)$ 2096, Mo-K_α radiation ($\lambda = 0.71069$ Å). Intensities were collected for a crystal of dimensions 0.16 × 0.10 × 0.20 mm on a Philips PW1100 4-circle diffractometer using a constant scan speed of 0.05° s^{-1} and scan width of 0.7° in the range $\theta = 3$—25°. Lorentz polarization corrections were applied and equivalents were averaged to give 3269 unique observed data [$F > 6\,\sigma(F)$]. Absorption corrections were not applied.

Structure solution and refinement

The two thallium atoms were located from a Patterson synthesis with SHELX76 [7] and the other non-hydrogen atom positions were obtained from a series of difference Fourier syntheses. The two independent molecules (I and II) are approximately related by a pseudo *c*-glide and for refinement the two molecules were kept in separate blocks to avoid correlation. The refined parameters included anisotropic thermal parameters for Tl, and the phenyl rings were refined as rigid groups with the constraints: C—C 1.395 Å, C—C—C 120.0°. The isotropic thermal parameters for analogous atoms in molecules I and II were set equal throughout the refinement. Complex neutral-atom scattering factors and weights $w = 1/[\sigma^2(F)]$, were used. Hydrogen atoms were not included. As a result of the pseudo symmetry, bond lengths and angles from the refinement for the two molecules are only poorly defined.

The refinement, in $P2_1$, converged to $R = 0.095$ and $R' = \Sigma w^{1/2}\,\Delta/\Sigma w^{1/2}|F_0| = 0.097$. The final atomic coordinates are listed in Table 1, and the labelling scheme is shown in Figs. 1 and 2 (ORTEP2 [8]). Tables of thermal parame-

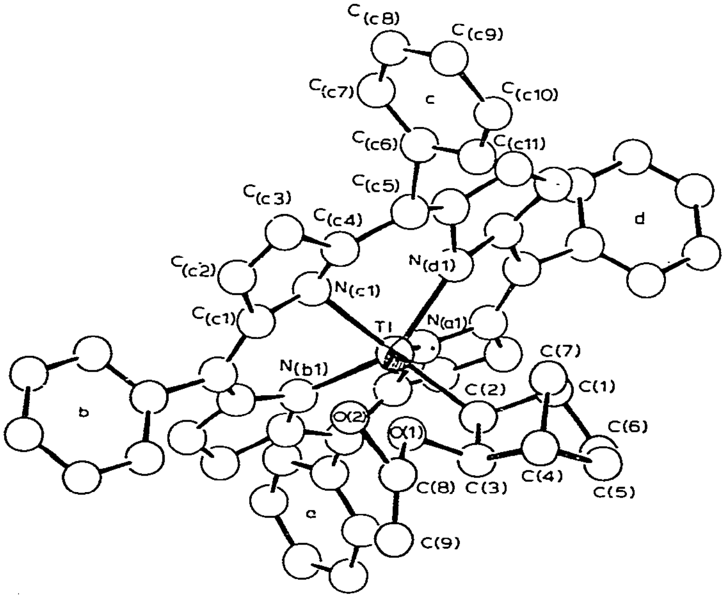

Fig. 2. The structure of NORTlTPP (molecule I), showing *cis-exo* substitution.

TABLE 1

ATOMIC COORDINATES FOR NORTITPP [a]

Atom [b]	x		y		z	
Tl	0.1984(1)	[0.7129(1)]	0	[0.2325(1)]	0.0646(1)	[0.0717(1)]
C(1)	0.1852(11)	[0.6667(12)]	0.1037(9)	[0.1340(11)]	0.1352(8)	[0.1245(9)]
C(2)	0.1675(8)	[0.6762(10)]	0.0694(10)	[0.1651(10)]	0.0284(10)	[0.0164(8)]
C(3)	0.2197(10)	[0.7295(12)[	0.0962(8)	[0.1401(10)]	−0.0610(9)	[−0.0750(12)
C(4)	0.2837(11)	[0.7698(12)]	0.1377(10)	[0.1018(12)]	−0.0025(13)	[0.0170(16)
C(5)	0.1977(14)	[0.6822(13)]	0.1652(12)	[0.0663(16)]	0.0400(10)	[−0.0206(13)
C(6)	0.1146(13)	[0.6289(14)]	0.1433(10)	[0.0894(12)]	0.0762(11)	[0.0561(13)
C(7)	0.2973(12)	[0.7813(14)]	0.1151(11)	[0.1181(12)]	0.1234(10)	[0.1494(12)
C(8)	0.3451(10)	[0.8539(12)]	0.0797(12)	[0.1731(13)]	−0.1780(9)	[−0.2067(16)
C(9)	0.4394(8)	[0.9595(16)]	0.0454(8)	[0.2015(14)]	−0.1371(11)	[−0.1919(8)]
O(1)	0.2887(16)	[0.8226(21)]	0.0582(30)	[0.1683(18)]	−0.0863(9)	[−0.0842(12)
O(2)	0.2912(11)	[0.8420(11)]	0.0964(11)	[0.1275(12)]	−0.2848(11)	[−0.2240(13)
N_{a1}	0.0454(30)	[0.5634(30)]	−0.0147(18)	[0.2459(17)]	0.1367(30)	[0.1422(31)
N_{b1}	0.1306(28)	[0.6427(30)]	−0.0497(16)	[0.2976(18)]	−0.0697(30)	[−0.0438(29)
N_{c1}	0.3405(29)	[0.8502(31)]	−0.507(18)	[0.2721(15)]	0.0788(30)	[0.0731(34)
N_{d1}	0.2618(30)	[0.7723(30)]	0.0063(17)	[0.2441(19)]	0.2577(21)	[0.2959(33)
C_{a1}	0.0357(29)	[0.5325(92)]	0.0109(18)	[0.2458(17)]	0.2352(26)	[0.2517(33)
C_{a2}	−0.0803(26)	[0.4176(24)]	0.0128(18)	[0.2364(16)]	0.2199(28)	[0.2403(34)
C_{a3}	−0.1263(30)	[0.3772(29)]	−0.0230(18)	[0.2455(18)]	0.1426(34)	[0.1159(33)
C_{a4}	−0.0517(29)	[0.4665(30)]	−0.0309(18)	[0.2589(18)]	0.0599(33)	[0.0607(34)
C_{a5}	−0.0511(30)	[0.4497(27)]	−0.0368(17)	[0.2898(18)]	−0.0671(30)	[−0.0409(30)
C_{a6}	−0.1643(24)	[0.3434(26)]	−0.0501(16)	[0.2980(17)]	−0.1185(33)	[−0.1241(29)
C_{a7}	−0.2157	[0.2804]	−0.0137	[0.2621]	−0.1862	[−0.1782]
C_{a8}	−0.3162	[0.1791]	−0.0197	[0.2710]	−0.2557	[−0.2433]
C_{a9}	−0.3654	[0.1408]	−0.0622	[0.3157]	−0.2575	[−0.2523]
C_{a10}	−0.3140	[0.2038]	−0.0986	[0.3516]	−0.1898	[−0.1972]
C_{a11}	−0.2135	[0.3051]	−0.0925	[0.3427]	−0.1203	[−0.1331]
C_{b1}	0.0266(30)	[0.5368(30)]	−0.0601(18)	[0.3046(17)]	−0.1184(30)	[−0.0913(31)]
C_{b2}	0.0114(26)	[0.5232(30)[	−0.0862(16)	[0.3328(18)]	−0.2278(33)	[−0.1971(29)]
C_{b3}	0.1129(27)	[0.6173(15)]	−0.0977(17)	[0.3432(17)]	−0.2333(29)	[−0.2292(21)]
C_{b4}	0.1903(30)	[0.6901(28)]	−0.0719(18)	[0.3190(16)]	−0.1421(34)	[−0.1295(21)]
C_{b5}	0.2983(24)	[0.7987(29)]	−0.0737(16)	[0.3232(18)]	−0.1379(33)	[−0.1184(28)]
C_{b6}	0.3297(27)	[0.8435(26)]	−0.0971(18)	[0.3370(19)]	−0.2384(31)	[−0.2204(34)]
C_{b7}	0.3239	[0.8183]	−0.0736	[0.3125]	−0.3475	[−0.3291]
C_{b8}	0.3652	[0.8511]	−0.0930	[0.3283]	−0.4403	[−0.4319]
C_{b9}	0.4124	[0.9090]	−0.1361	[0.3686]	−0.4239	[−0.4261]
C_{b10}	0.4183	[0.9343]	−0.1596	[0.3931]	−0.3148	[−0.3174]
C_{b11}	0.3769	[0.9015]	−0.1401	[0.3773]	−0.2220	[−0.2145]
C_{c1}	0.3693(30)	[0.8646(27)]	−0.0624(18)	[0.3009(16)]	−0.0312(34)	[−0.0225(27)]
C_{c2}	0.4851(27)	[0.9806(28)]	−0.0706(16)	[0.3008(15)]	0.0041(31)	[−0.0168(27)]
C_{c3}	0.5244(31)	[1.0270(26)]	−0.0571(18)	[0.2788(14)]	0.1116(33)	[0.0772(29)]
C_{c4}	0.4343(27)	[0.9476(28)]	−0.0347(17)	[0.2715(13)]	0.1482(28)	[0.1513(27)]
C_{c5}	0.4537(28)	[0.9521(31)]	−0.0076(17)	[0.2587(16)]	0.2660(33)	[0.2831(34)]
C_{c6}	0.5564(26)	[1.0678(30)]	−0.0126(18)	[0.2448(16)]	0.3633(31)	[0.3305(26)]
C_{c7}	0.5939	[1.1110]	0.0321	[0.2879]	0.3798	[0.3630]
C_{c8}	0.6962	[1.2120]	0.0402	[0.2915]	0.4417	[0.4333]
C_{c9}	0.7611	[1.2697]	0.0036	[0.2520]	0.4869	[0.4710]
C_{c10}	0.7236	[1.2266]	−0.0412	[0.2089]	0.4704	[0.4384]
C_{c11}	0.6213	[1.1256]	−0.4930	[0.2054]	0.4085	[0.3682]
C_{d1}	0.3672(30)	[0.8808(24)]	−0.0028(16)	[0.2272(14)]	0.3289(31)	[0.3273(27)]
C_{d2}	0.3827(31)	[0.8902(28)]	0.0202(14)	[0.2196(12)]	0.4437(24)	[0.4550(31)]
C_{d3}	0.2917(32)	[0.7892(26)]	0.0332(18)	[0.2138(19)]	0.4736(28)	[0.4632(32)]
C_{d4}	0.2210(30)	[0.7109(29)]	0.0113(14)	[0.2112(19)]	0.3698(34)	[0.3486(27)]
C_{d5}	0.1036(26)	[0.5953(29)]	0.0187(14)	[0.2185(14)]	0.3435(30)	[0.3359(26)]
C_{d6}	0.0559(27)	[0.5770(31)]	0.0356(17)	[0.2017(17)]	0.4503(31)	[0.4602(31)]
C_{d7}	0.0700	[0.5810]	0.0073	[0.2284]	0.5524	[0.5640]
C_{d8}	0.0347	[0.5440]	0.0213	[0.2107]	0.6553	[0.6623]
C_{d9}	−0.0147	[0.5031]	0.0636	[0.1665]	0.6560	[0.6566]
C_{d10}	−0.0288	[0.4991]	0.0919	[0.1399]	0.5539	[0.5527]
C_{d11}	0.0065	[0.5361]	0.0779	[0.1575]	0.4510	[0.4545]

[a] Values for molecule I are given first, followed by the values for molecule II in square brackets. Least-squares e.s.d. values are given in parentheses. [b] Each symbol for an atom of the tetraphenylporphyrin carries a literal subscript to identify its particular subunit (a, b, c or d) and a numerical subscript that follows to distinguish atoms of the same element within the same subunit (Fig. 2).

ters, bond lengths and angles, least squares planes and the observed and calculated structure factors are available from the authors.

Results and discussion

Reaction of stoichiometric amounts of NORTl$(OAc)_2$ and $TPPH_2$ in chloroform solution gave blue crystals of NORTlTPP suitable for single crystal X-ray analysis. The structural results, however, are of low accuracy because of refinement difficulties arising from pseudo-symmetry relating the two independent molecules in the unit cell. Thus Figs. 1 and 2 represent only the gross structural features of the molecule, and the selected bond lengths and angles listed in Table 2 are averaged between two two independent molecules.

Overall, the structure consists of a thallium atom placed above the plane defined by the porphyrin ring system with the 3-acetylbicyclo[2.2.1]hept-5-ene unit attached to thallium on the opposite side to the porphyrin ring. The structure therefore resembles that found for MeTlTPP [1] and like the latter compound is characterized by a large displacement (0.9 Å) of the thallium atom from the N_4 plane of the TPP ring (maximum deviation from N_4 least squares plane, ±0.2 Å). Dihedral angles between the plane of the porphinato core and that of each phenyl group lie in the range 90—60° normally observed [3b] for metallo-TPP derivatives. The averaged Tl—C(2) distance (2.09 Å) is

TABLE 2

SELECTED BOND LENGTHS AND ANGLES FOR NORTlTPP AVERAGED BETWEEN THE TWO INDEPENDENT MOLECULES

(a) Distances (Å)

Tl—C(2)	2.09	Tl—N_{c1}	2.24
Tl—N_{a1}	2.32	Tl—N_{d1}	2.33
Tl—N_{b1}	2.26	Tl...O(1)	2.97
C(1)—C(2)	1.54	C(1)—C(6)	1.54
C(1)—C(7)	1.53	C(2)—C(3)	1.55
C(3)—C(4)	1.50	C(3)—O(1)	1.49
C(4)—C(5)	1.54	C(4)—C(7)	1.57
C(5)—C(6)	1.39	O(1)—C(8)	1.52
C(8)—C(9)	1.59	C(8)—O(2)	1.35

(b) Mean distances for chemically equivalent bonds within the porphyrin skeleton (Å)

N_1—C_1	1.41	N_1—C_4	1.43
C_1—C_2	1.46	C_2—C_3	1.35
C_3—C_4	1.48	C_4—C_5	1.47
C_5—C_6	1.52	C_1—C_5	1.42

(c) Angles (°)

N_{a1}—Tl—N_{b1}	80	C(2)—Tl—N_{a1}	96
N_{a1}—Tl—N_{c1}	129	C(2)—Tl—N_{b1}	121
N_{a1}—Tl—N_{d1}	78	C(2)—Tl—N_{c1}	134
N_{b1}—Tl—N_{c1}	79	C(2)—Tl—N_{d1}	106
N_{b1}—Tl—N_{d1}	129		
N_{c1}—Tl—N_{d1}	81		

TABLE 3

^{13}C CHEMICAL SHIFTS AND ^{205}Tl—^{13}C COUPLING CONSTANTS FOR NORBORNENE DERIVATIVES OF THALLIUM(III) [a]

Compound	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)	Other
NORTlTPP [b]	41.9 (246)	53.1 (6218) [c]	73.8 (347)	46.6 (<5)	131.5 [d] (73)	139.6 (797)	44.4 (16)	176.7	20.2 (39)	C(α), 150.4 (32); C(β), 131.9 (24); C(meso), 121.8 (37); Phenyl carbons: C1, 142.8; C2, 134.6; C3, 126.4; C4, 127.5
NORTl(OAc)$_2$ [e]	45.7 (244)	72.9 (5471) [c]	75.2 (420)	48.2 (<3)	135.3 (120)	140.3 (1057)	47.4 (17)	[f]	21.2 (49)	(OAc)$_2$Tl-: CH_3, 22.8; CO, 179.4
Δδ(^{13}C) [g]	−3.8	−19.8	−1.4	−1.6	−3.8	−0.7	−3.0		−1.0	

[a] In $CDCl_3$. Chemical shifts in ppm relative to internal $SiMe_4$. Numbers in parentheses are thallium—carbon coupling constants in Hz. Separate coupling to ^{203}Tl and ^{205}Tl was not resolved unless otherwise noted. [b] Errors: chemical shifts, ±0.2 ppm; coupling constants, ±3 Hz. [c] $^1J(^{205}Tl$—$^{13}C)$. [d] Assignment tentative because of overlapping signals in this region. [e] Data from ref. 5. [f] Not observed. [g] $\Delta\delta(^{13}C) = \delta(^{13}C, \text{NORTlTPP}) - \delta(^{13}C, \text{NORTl(OAc)}_2)$.

typical of Tl—C bond lengths in mono-organothallium(III) compounds (2.073—2.160 Å) [1,2,9].

The stereochemistry of the substituted bicyclo[2.2.1]hept-5-ene is shown in Fig. 2 and clearly confirms that acetoxythallation of norbornene proceeds to *cis-exo* products. This stereochemistry has been assumed for NORTl(OAc)$_2$ and related compounds [4—6,10—13] and was originally deduced on the basis of ^{1}H NMR spectra [14].

The relative orientations of the TPP ring and the bicyclo[2.2.1]hept-5-ene unit are such that normals to the C(2)—C(3)—C(5)—C(6) plane (maximum deviation from least squares plane, ±0.17 Å) and the N$_4$ plane intersect at ca. 90°. An interesting feature of the structure is that the Tl—C bond is bent away (average angle 20°) from the normal to the N$_4$ plane (Fig. 1). This is in contrast to CH$_3$TlTPP [1] where the Tl—CH$_3$ bond is tipped only 1° from the normal to the mean skeletal plane of the porphyrin ring. The Tl...O(1) distance is essentially fixed (2.97 Å) by the *cis-exo* configuration of the substituted norbornenyl unit. Thus the origin of the tilt may arise in non-bonding interactions between the porphyrin ring system and O(1) (e.g. the average value of O(1)...N$_{c1}$ is 3.7 Å). In support of this, the Tl, C(2), C(3), O(1) plane (maximum deviation from least squares plane, ±0.08 Å) contains the normal to the N$_4$ plane (the normals intersect at an average angle of 92°).

The ^{13}C NMR spectrum of NORTlTPP was determined in chloroform and the NMR parameters are compared with those of NORTl(OAc)$_2$ [5] in the same solvent in Table 3. Barron et al. [6] have also reported ^{13}C NMR parameters for NORTl(OAc)$_2$ which are in generally close agreement with those of Inubushi et al. [5]. (Significant differences are noted for ^1J(Tl—C) and δC(6)). Assignments for NORTlTPP were made using proton broad-band decoupled and SFORD (single frequency off resonance decoupled) spectra obtained at 22.63 and 45.28 MHz. δ(^{13}C) values for the TPP ring are very close to those found for other thallium derivatives of this ligand [1,15], and coupling between Tl and the TPP ring carbons is characterized by values more closely resembling those found for MeTlTPP [1] than for ClTlTPP [15].

Thallium coordination to the TPP ring results in increased shielding for the carbon atoms of the bicyclo[2.2.1]unit (Table 3). An increase in shielding is expected for atoms positioned above and within the porphyrin ring current loop [3a]. The carbon atoms of the bicyclo[2.2.1]unit are so positioned and, with the exception of C(2), show increased shielding (0.7—3.8 ppm) of the predicted order [3a]. By comparison, the increase in shielding for C(2) between NORTl(OAc)$_2$ and NORTlTPP is rather large and presumably reflects the additional effect of replacing the O-donor atoms of OAc with the N-donors of TPP. The more distant carbon atoms are less likely to be affected by this substitution. An almost identical shift (—19.6 ppm) is observed between MeTl(OAc)$_2$ (δ(*C*H$_3$), 18.7 ppm [16]) and methyl-2,3,7,8,12,13,17,18-octaethylporphinatothallium(III) (δ(*C*H$_3$), —0.9 ppm).

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