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Scandium complexes with the tetraphenylethylene and anthracene dianions

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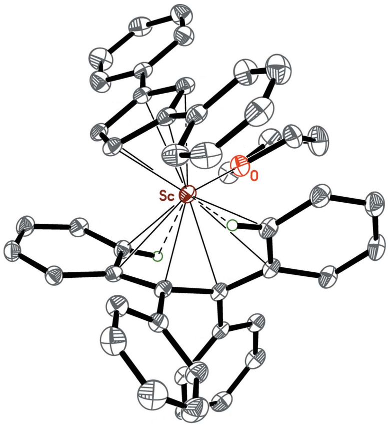
The structural study of Sc complexes containing dianions of anthracene and tetraphenylethylene should shed some light on the nature of rare-earth metal–carbon bonding. The crystal structures of (18-crown-6)bis(tetrahydrofuran-κO)sodium bis(η^6-1,1,2,2-tetraphenylethenediyl)scandium(III) tetrahydrofuran disolvate, $[Na(C_4H_8O)_2(C_{12}H_{24}O_6)][Sc(C_{26}H_{20})_2]\cdot 2C_4H_8O$ or $[Na(18\text{-crown-}6)(THF)_2][Sc(\eta^6\text{-}C_2Ph_4)_2]\cdot 2(THF)$, (**1b**), ($\eta^5$-1,3-diphenylcyclopentadienyl)(tetrahydrofuran-κO)(η^6-1,1,2,2-tetraphenylethenediyl)scandium(III) toluene hemisolvate, $[Sc(C_{17}H_{13})(C_{26}H_{20})(C_4H_8O)]\cdot 0.5C_7H_8$ or $[(\eta^5\text{-}1,3\text{-}Ph_2C_5H_3)Sc(\eta^6\text{-}C_2Ph_4)(THF)]\cdot 0.5(toluene)$, (**5b**), poly[[($\mu_2$-$\eta^3$:$\eta^3$-anthracenediyl)bis($\eta^6$-anthracenediyl)bis($\eta^5$-1,3-diphenylcyclopentadienyl)tetrakis(tetrahydrofuran)dipotassiumdiscandium(III)] tetrahydrofuran monosolvate], $\{[K_2Sc_2(C_{14}H_{10})_3(C_{17}H_{13})_2(C_4H_8O)_4]\cdot C_4H_8O\}_n$ or $[K(THF)_2]_2[(1,3\text{-}Ph_2C_5H_3)_2Sc_2(C_{14}H_{10})_3]\cdot THF$, (**6**), and 1,4-diphenylcyclopenta-1,3-diene, $C_{17}H_{14}$, (**3a**), have been established. The $[Sc(\eta^6\text{-}C_2Ph_4)_2]^-$ complex anion in (**1b**) contains the tetraphenylethylene dianion in a symmetrical bis-η^3-allyl coordination mode. The complex homoleptic $[Sc(\eta^6\text{-}C_2Ph_4)_2]^-$ anion retains its structure in THF solution, displaying hindered rotation of the coordinated phenyl rings. The 1D ^1H and $^{13}C\{^1H\}$, and 2D COSY ^1H–^1H and ^{13}C–^1H NMR data are presented for $M[Sc(Ph_4C_2)_2]\cdot x$THF [M = Na and x = 4 for (**1a**); M = K and x = 3.5 for (**2a**)] in THF-d_8 media. Complex (**5b**) exhibits an unsymmetrical bis-η^3-allyl coordination mode of the dianion, but this changes to a η^4 coordination mode for $(1,3\text{-}Ph_2C_5H_3)Sc(Ph_4C_2)(THF)_2$, (**5a**), in THF-$d_8$ solution. A ^{45}Sc NMR study of (**2a**) and UV–Vis studies of (**1a**), (**2a**) and (**5a**) indicate a significant covalent contribution to the Sc—Ph_4C_2 bond character. The unique Sc ate complex, (**6**), contains three anthracenide dianions demonstrating both a η^6-coordination mode for two bent ligands and a μ_2-η^3:η^3-bridging mode of a flat ligand. Each $[(1,3\text{-}Ph_2C_5H_3)_2Sc_2(C_{14}H_{10})_3]^{2-}$ dianionic unit is connected to four neighbouring units *via* short contacts with $[K(THF)_2]^+$ cations, forming a two-dimensional coordination polymer framework parallel to (001).



1. Introduction

Rare-earth complexes with dianions of aromatic hydrocarbons are currently of mainly fundamental interest. Generally, the Ln—C bond is considered to be ionic in character in many organometallic compounds (Raymond & Eigenbrot, 1980). However, a substantial covalent component is sometimes present in Ln—C bonding in rare-earth complexes with some dianionic ligands, which has been demonstrated, for example, for anthracenide complexes (Roitershtein *et al.*, 1993). The purpose of this work is the structural study of Sc complexes containing dianions of anthracene and tetraphenylethylene in order to shed some light on the nature of the rare-earth metal–carbon bonding.

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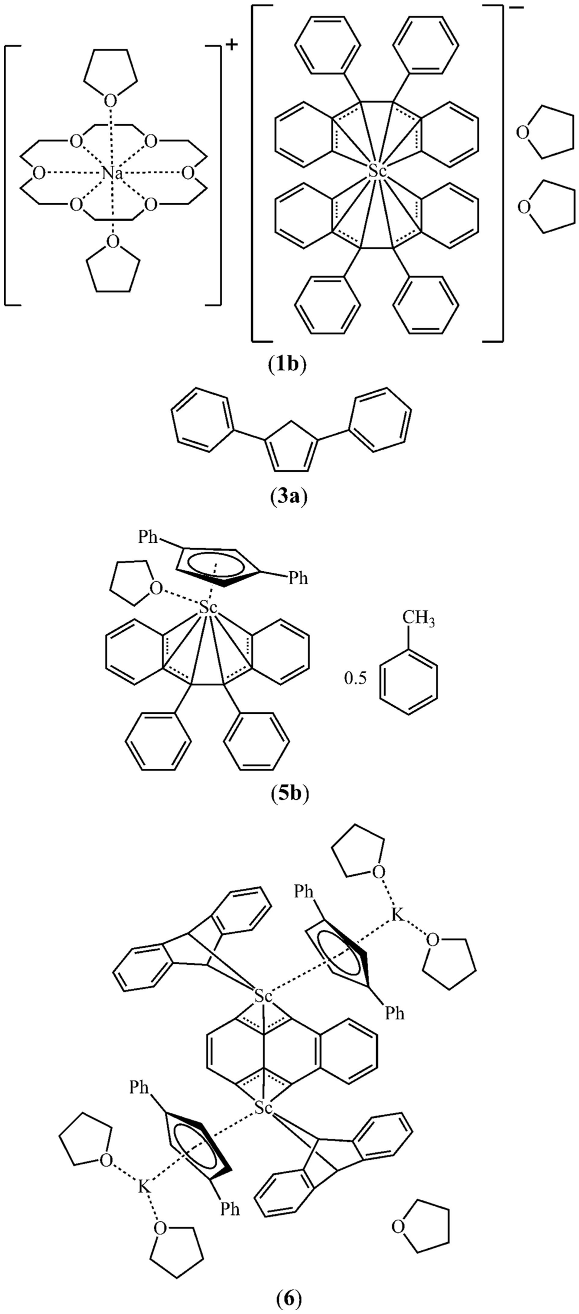
Tetraphenylethylene (for its crystal structure, see, for example, Hua *et al.*, 2007; Jin *et al.*, 2016) is easily reduced to the dianion by alkali metals in σ-donating solvent media (Et$_2$O, THF, diglyme, *etc.*) (Matsui *et al.*, 1994; Szwarc, 1968). Although detailed preparation and reactivity studies of the disodium tetraphenylethylene salt were reported 90 years ago (Schlenk & Bergmann, 1928), the crystal structures of alkali-metal adducts with the tetraphenylethylene dianion were established much later (Bock *et al.*, 1989, 1996; Minyaev *et al.*, 2007; Minyaev & Ellis, 2014). These alkali-metal salts of tetraphenylethylene are synthetic precursors to rare-earth tetraphenylethenide complexes, of which the number with established crystal structures is limited [Ln = Y, Yb^{II} and Lu; Roitershtein *et al.*, 1998, 2004; Roitershtein, Minyaev *et al.*, 2007; Minyaev *et al.*, 2007; see *Results and discussion* (§3) below], in some part due to their extremely high reactivity to traces of moisture and oxygen.



(**1b**)

(**3a**)

(**5b**)

(**6**)

Scheme 1

Anthracene π-complexes of *d*-metals have been explored extensively in recent decades [57 crystal structures according to the Cambridge Structural Database (CSD; Version 5.39, with latest updates to February 2018; Groom *et al.*, 2016)], but rare-earth anthracenide complexes have been poorly explored to date, including their crystal structures (see *The CSD survey*, §3.6.3) and possible practical applications.

We report herein the synthesis and structures of two Sc–tetraphenylethenide complexes, namely [Na(18-crown-6)(THF)$_2$][Sc(η^6-C$_2$Ph$_4$)$_2$]·2(THF), (**1b**), and [(η^5-1,3-Ph$_2$C$_5$H$_3$)Sc(η^6-C$_2$Ph$_4$)(THF)]·0.5(toluene), (**5b**), and the Sc–anthracenide complex [K(THF)$_2$]$_2$[(1,3-Ph$_2$C$_5$H$_3$)$_2$Sc$_2$(η^6-C$_{14}$H$_{10}$)$_2$(μ_2-η^3:η^3-C$_{14}$H$_{10}$)]·THF, (**6**) (Scheme 1).

Complexes Na[Sc(C$_2$Ph$_4$)$_2$]·4THF, (**1a**), and K[Sc(C$_2$Ph$_4$)$_2$]·3.5THF, (**2a**), were obtained in reactions between M_2[C$_2$Ph$_4$] and ScCl$_3$(THF)$_3$ (or anhydrous ScI$_3$) in a 2:1 molar ratio in THF (Fig. 1). Numerous attempts to obtain suitable single crystals of the THF solvates (**1a**) and (**2a**) for X-ray diffraction studies were unsuccessful, presumably due to the easy loss of THF at low temperature and upon vacuum drying. This problem was noted earlier for the analogous complexes M[Lu(C$_2$Ph$_4$)$_2$]·5THF (M = Na and K; Roitershtein *et al.*, 2004). Slow diffusion of Et$_2$O into a THF solution of (**1a**), containing 18-crown-6, allowed us to isolate single crystals of (**1b**) (Fig. 1).

Metallation of a mixture of isomeric 1,4- and 1,3-diphenylcyclopentadienes, (**3a**)/(**3b**), with KCH$_2$Ph led to *in situ*-generated K(1,3-Ph$_2$C$_5$H$_3$), which was then reacted with ScCl$_3$(THF)$_3$, leading to Sc(1,3-Ph$_2$C$_5$H$_3$)Cl$_2$(THF)$_3$, (**4**) (Fig. 2). We were not able to obtain single crystals of (**4**) suitable for X-ray studies. One attempt to crystallize (**4**) from a THF/pentane mixture resulted in the formation of 1,4-diphenylcyclopenta-1,3-diene, (**3a**), likely due to unintended partial hydrolysis of (**4**). The reaction of *in situ*-generated (**4**) with disodium tetraphenylethylene provided microcrystals of [Sc(η^5-1,3-Ph$_2$C$_5$H$_3$)(η^6-C$_2$Ph$_4$)(THF)]·THF, (**5a**) (Fig. 2). Recrystallization of the latter from a toluene/hexane mixture resulted in the formation of (**5b**), the structure of which was established by X-ray diffraction analysis.

Poorly soluble complex (**6**) was first synthesized by the reaction of *in situ*-generated (**4**) and 2 equiv. of potassium anthracenide, K$^+$·C$_{14}$H$_{10}^{\bullet-}$ (Fig. 3, upper synthesis), targeting an unknown (1,3-Ph$_2$C$_5$H$_3$)Sc(C$_{14}$H$_{10}$) complex. A higher yield of (**6**) was then obtained by reaction of ScCl$_3$(THF)$_3$ with 1 equiv. of K$^+$C$_{14}$H$_{10}^{\bullet-}$, and then with 1 equiv. of *in situ*-generated K(1,3-Ph$_2$C$_5$H$_3$) (Fig. 3, middle synthesis). Analogous reactions to prepare Y and Lu derivatives failed, leading to mixtures of solids containing considerable amounts of paramagnetic impurities. Crystallization of (**6**) was performed from a THF/hexane mixture in both cases. An attempt to synthesize (**6**) from potassium–graphite, anthracene and *in situ*-generated (**4**) failed (Fig. 3, lower synthesis), providing a black solid, which was presumably an unknown Sc anthracenide compound, and K(1,3-Ph$_2$C$_5$H$_3$) (^{1}H NMR data).

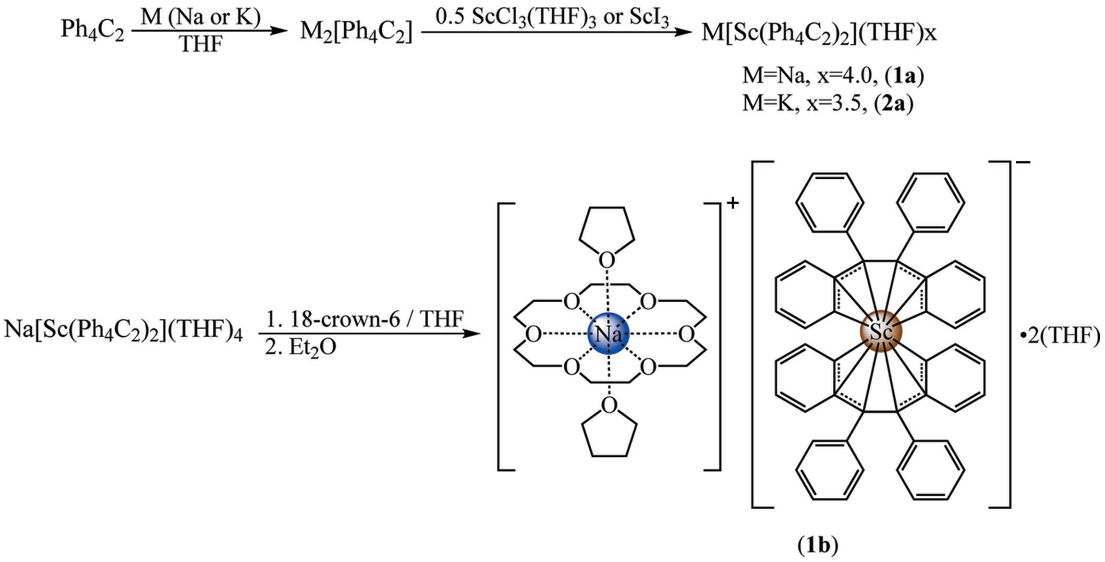


Figure 1
Synthesis of (**1a**) and (**2a**), and crystallization of (**1b**).

2. Experimental

2.1. Synthesis and crystallization

2.1.1. General experimental remarks. The studied dianionic Sc complexes are extremely sensitive to oxygen and moisture. All described synthetic manipulations were performed under an argon atmosphere or under vacuum, using an argon-filled glove-box, standard Schlenk glassware or evacuated sealed Schlenk-type vessels equipped with Teflon stopcocks, vacuum-line techniques and absolute solvents. Tetrahydrofuran (THF) and diethyl ether were predried over NaOH and distilled from sodium/benzophenone ketyl. Pentane, hexane and THF-d_8 were distilled from Na/K alloy. Toluene was distilled from sodium/benzophenone ketyl. 18-Crown-6 was distilled from sodium. Tetraphenylethylene was purified by recrystallization from anhydrous toluene and dried under vacuum. Anthracene was vacuum sublimed. Aliphatic hydrocarbon oil was vacuum distilled from Na prior to the preparation of the specimens for X-ray diffraction studies. Benzyl potassium was prepared according to the published procedure of Schlosser & Hartmann (1973). $ScCl_3(THF)_3$ was synthesized according to the literature procedure of Herrmann (1997). The scandium content in $ScCl_3(THF)_3$ and in all the studied Sc complexes was determined by direct complexometric titration with the disodium salt of EDTA (ethylenediaminetetraacetic acid), using xylenol orange indicator. Due to the highly pyrophoric nature of the dianionic complexes, their elemental C/H analyses were not performed. Diphenylcyclopenta-1,3-diene was obtained according to the literature as a mixture of 1,3- and 1,4-isomers (Roitershtein, Minyaev *et al.*, 2007), and was purified by vacuum sublimation.

Electronic absorption spectra were recorded on a Varian Cary 300 Bio instrument (software Version 3.0, double beam method, baseline correction, 200–800 nm range). The C/H

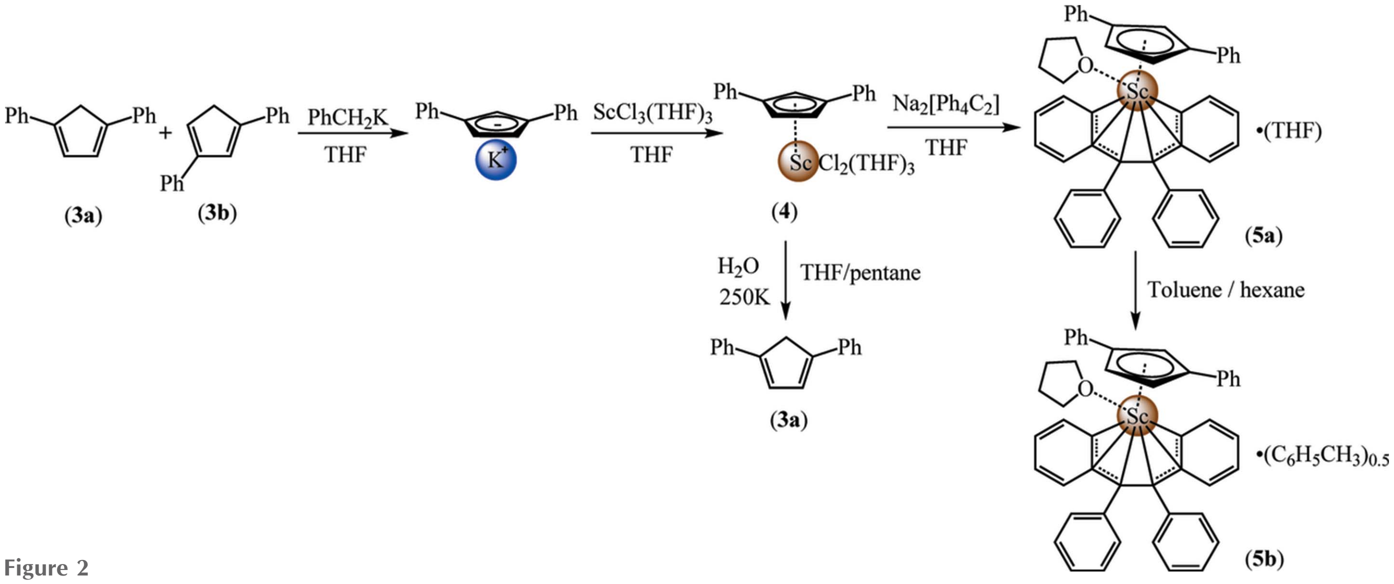
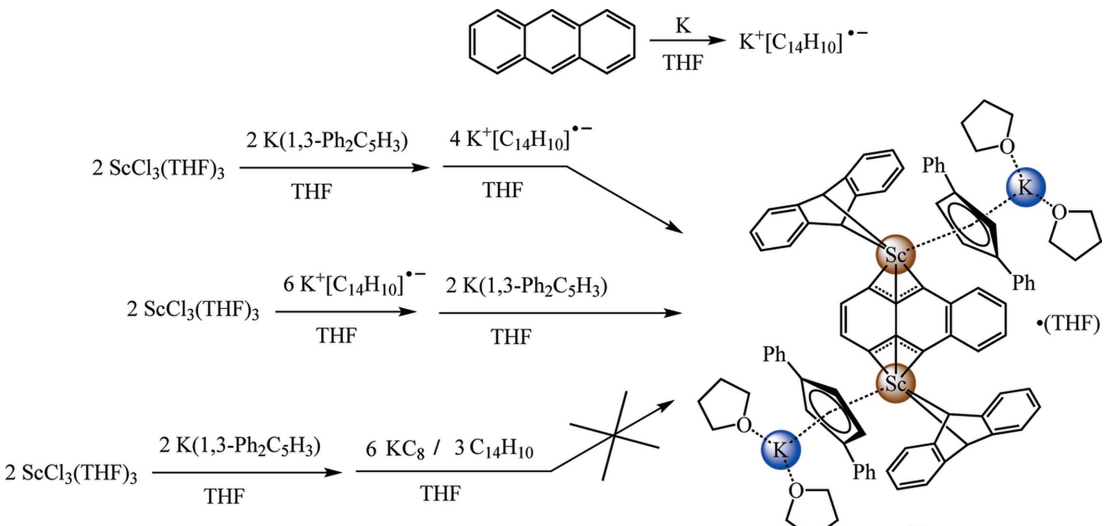


Figure 2
Synthesis of (**4**) and (**5a**), and crystallization of (**3a**) and (**5b**).



(6)

Figure 3
The synthesis of (**6**).

elemental analysis of (**4**) was performed with a PerkinElmer 2400 Series II elemental analyzer. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra were registered with Varian VI-300, Bruker AM-300 (300 MHz for ^{1}H and 75.5 MHz for ^{13}C) and Bruker AV-600 (600 MHz for ^{1}H and 150.9 MHz for ^{13}C) spectrometers in THF-d_8 for the Sc complexes and in $CDCl_3$ for (**3a**).

2.1.2. Synthesis of (1a) (see Fig. 1). A solution of $Na_2[C_2Ph_4]$ was prepared from a sodium mirror (0.615 g, 26.7 mmol) and tetraphenylethylene (1.509 g, 4.54 mmol) in THF (95 ml) over a period of 2 d. The $Na_2[C_2Ph_4]$ solution was added to a suspension of $ScCl_3(THF)_3$ (0.825 g, 2.24 mmol) in THF (50 ml). The reaction mixture was stirred for 2 d. The product was extracted from one ampoule to another by multiple operations of decanting the solution from the precipitate, condensing THF back at low temperatures and stirring for 15 min. The shiny dark-red crystals were washed with THF (3 × 5 ml) and dried under vacuum for 3 h to a constant mass. The yield of obtained microcrystalline powder was 1.805 g (1.77 mmol, 79%). Analysis found (calculated for $C_{68}H_{72}NaO_4Sc$): Sc 4.35% (4.40%).

^{1}H NMR (243 K, 300 MHz, 128 scans, THF-d_8): δ 4.65 (*dd*, 4H, H_5), 6.29 (*t*, 4H, H_3), 6.58 (*t*, 4H, H_{para}), 6.87 (*br t*, 12H, $H_2 + H_{meta}$), 7.07 (*t*, 4H, H_4), 7.14 (*d*, 8H, H_{ortho}), 7.65 (*dd*, 4H, H_1). $^{13}C\{^{1}H\}$ NMR (253 K, 150.9 MHz, THF-d_8): δ 86.1

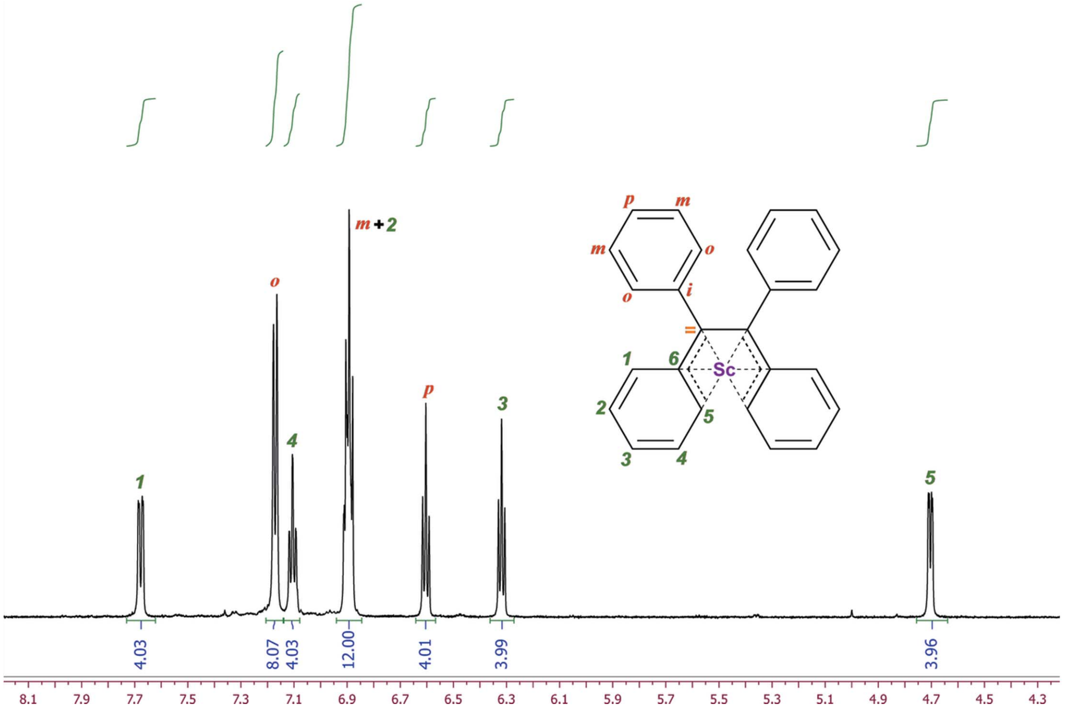


Figure 4
^{1}H NMR spectrum of $K[Sc(Ph_4C_2)_2]$, (**2a**), at 253 K and the atom-numbering scheme for the ^{1}H and ^{13}C signals.

Table 1
Experimental details.

Experiments were carried out with Mo $K\alpha$ radiation.

	(1b)	**(3a)**	**(5b)**	**(6)**
Crystal data				
Chemical formula	$[Na(C_4H_8O)_2(C_{12}H_{24}O_6)][Sc(C_{26}H_{20})_2]\cdot 2C_4H_8O$	$C_{17}H_{14}$	$[Sc(C_{17}H_{13})(C_{26}H_{20})(C_4H_8O)]\cdot 0.5C_7H_8$	$[K_2Sc_2(C_{14}H_{10})_3(C_{17}H_{13})_2(C_4H_8O)_4]\cdot C_4H_8O$
M_r	1285.51	218.28	712.82	1497.84
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Monoclinic, $C2/c$	Orthorhombic, $Ibam$
Temperature (K)	173	123	173	150
a, b, c (Å)	12.6071 (18), 14.764 (2), 18.585 (3)	7.3278 (5), 5.9440 (4), 26.6551 (18)	36.060 (5), 9.5557 (14), 23.662 (3)	18.1056 (12), 19.9164 (13), 21.8633 (14)
α, β, γ (°)	84.507 (2), 88.508 (2), 84.033 (2)	90, 94.352 (1), 90	90, 113.840 (2), 90	90, 90, 90
V (Å^3)	3424.2 (9)	1157.65 (14)	7457.9 (19)	7883.9 (9)
Z	2	4	8	4
μ (mm^{-1})	0.17	0.07	0.24	0.33
Crystal size (mm)	0.40 × 0.25 × 0.25	0.60 × 0.20 × 0.20	0.50 × 0.40 × 0.25	0.25 × 0.08 × 0.07
Data collection				
Diffractometer	Bruker SMART Platform CCD area detector	Bruker SMART Platform CCD area detector	Bruker SMART Platform CCD area detector	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2003)	Multi-scan (*SADABS*; Bruker, 2003)	Multi-scan (*SADABS*; Bruker, 2003)	Multi-scan *SADABS* (Bruker, 2008)
T_{min}, T_{max}	0.869, 0.967	0.829, 0.985	0.869, 0.967	0.922, 0.977
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	32161, 11986, 6407	13442, 2788, 2406	42147, 8560, 6319	28915, 3990, 2831
R_{int}	0.073	0.083	0.044	0.072
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.595	0.667	0.650	0.617
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.074, 0.183, 1.03	0.044, 0.128, 1.03	0.058, 0.159, 1.04	0.055, 0.150, 1.03
No. of reflections	11986	2788	8560	3990
No. of parameters	860	210	467	307
No. of restraints	46	0	0	128
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	All H-atom parameters refined	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.48	0.32, −0.25	1.17, −0.45	0.77, −0.47

Computer programs: *SMART* (Bruker, 2003), *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2003), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SIR2002* (Burla *et al.*, 2003), *SHELXL2017* (Sheldrick, 2015) and *SHELXTL* (Sheldrick, 2008).

(Ph_2CCPh_2 or $C_=$), 100.5 (*br*, C_5), 112.0 (C_3), 118.6 (*br*, C_1), 119.4 (C_{para}), 125.5 (C_{meta}), 128.1 (C_{ortho}), 129.7 (*br*, C_2), 137.4 (*br*, C_4), 137.8 (C_6), 144.3 (C_{ipso}). Please refer to Fig. 4 for the NMR numbering scheme. Low-temperature ^{1}H NMR spectra are presented in the supporting information in Fig. S6 (the temperature range is from 203 to 283 K, with a step of 20 K). UV–Vis (THF) λ_{max}: 312, 392, 555 (*sh*) nm.

2.1.3. Crystallization of (1b). $Na[Sc(Ph_4C_2)_2]\cdot 4.5(THF)$ (170 mg, 0.17 mmol) and 18-crown-6 (70 mg, 0.26 mmol) were dissolved in THF (15 ml). Diethyl ether (35 ml) was layered on top of the formed solution. Crystals of (**1b**) formed over a period of three weeks.

2.1.4. Synthesis of (2a). The product (**2a**) was obtained in an analogous manner to (**1a**), starting from potassium (1.191 g, 30.5 mmol), tetraphenylethylene (2.001 g, 6.02 mmol) and $ScCl_3(THF)_3$ (1.092 g, 2.97 mmol). The yield of a dark-red microcrystalline powder was 1.904 g (1.90 mmol, 64%). Analysis found (calculated for $C_{66}H_{68}KO_{3.5}Sc$): Sc 4.47% (4.49%).

^{1}H NMR (253 K, 600 MHz, THF-d_8): δ 4.70 (*dd*, 4H, H_5), 6.32 (*t*, 4H, H_3), 6.60 (*t*, 4H, H_{para}), 6.90 (*br t*, 12H, $H_2 + H_{meta}$), 7.10 (*br t*, 4H, H_4), 7.18 (*d*, 8H, H_{ortho}), 7.68 (*dd*, 4H, H_1). ^{13}C{^{1}H} NMR (253 K, 150.9 MHz, THF-d_8): δ 86.2 ($C_=$), 100.5 (*br*, C_5, $^1J_{CH}$ = 147.3 Hz), 112.1 (C_3, $^1J_{CH}$ = 160.6 Hz), 118.6 (*br*, C_1, $^1J_{CH}$ = 154.6 Hz), 119.5 (C_{para}, $^1J_{CH}$ = 156.5 Hz), 125.5 (C_{meta}, $^1J_{CH}$ = 154.2 Hz), 128.1 (C_{ortho}, $^1J_{CH}$ = 156.1 Hz), 129.8 (*br*, C_2, $^1J_{CH}$ = 154.2 Hz), 137.4 (*br*, C_4, $^1J_{CH}$ = 148.6 Hz), 144.3 (C_{ipso}), 137.8 (C_6). Please refer to Fig. 4 for the NMR numbering scheme. ^{45}Sc NMR (295 K, 72.94 MHz, THF-d_8): δ 64.0 [reference: 203.3 ppm for $ScCl_3(THF)_3$; see Figs. S10 and S11 in the supporting information]. UV–Vis (THF) λ_{max}: 312, 391, 520 (*sh*) nm.

2.1.5. Synthesis of (4) (see Fig. 2). A cold (203 K) solution of KCH_2Ph (2.103 g, 16.2 mmol) in THF (55 ml) was added dropwise to a stirred cold (203 K) solution of diphenylcyclopenta-1,3-diene [a mixture of (**3a**) and (**3b**); 3.420 g, 15.7 mmol] in THF (100 ml). The mixture was stirred for 30 min, heated to 303 K and stirred for another 30 min. The resulting solution was added dropwise to a stirred suspension of $ScCl_3(THF)_3$ (5.650 g, 15.4 mmol) in THF (10 ml) at 203 K. The mixture was stirred for 30 min at 203 K and then for 1 h at 303 K. Almost all the THF was evaporated under reduced pressure. The complex was extracted from the residue with portions of toluene (270 ml total) at room temperature. The resulting solution was filtered through a fine frit. Almost all the toluene was evaporated. The resulting product was dissolved in THF (160 ml) and the mixture was filtered. All but *ca* 60 ml of solvent was then evaporated from the solution. Pentane (150 ml) was layered on top. The next day the two-layer mixture was stirred and left for 5 h without stirring. The precipitated product was filtered off and dried under vacuum. The yield of pale-yellow microcrystals of (**4**) was 3.749 g (6.82 mmol, 44%). Analysis found (calculated for $C_{29}H_{37}Cl_2O_3Sc$): C 63.28 (63.39), H 6.61 (6.79), Sc 8.23% (8.18%). ^{1}H NMR (300 MHz, THF-d_8): δ 6.01 (*br s*, 1H), 6.46 (*br s*, 1H), 6.96–7.00 (*m*, 1H), 7.11–7.19 (*m*, 2H), 7.25–7.37 (*m*, 4H), 7.58–7.64 (*m*, 4H).

2.1.6. Crystallization of (3a). Pentane (40 ml) was layered on top of a solution of (**4**) (160 mg) in THF (18 ml). Crystals had not formed within two weeks after the layers were mixed at room temperature. A Schlenk vessel containing this solution was placed in a freezer (*ca* 250 K). A few crystals of (**3a**) formed within a week. ^{1}H NMR (295 K, 300 MHz, CDCl$_3$): δ 3.77 (*s*, 2H, $=$CPh$-$*CH$_2$*$-$CPh$=$), 6.93 (*s*, 2H, –CPh$=$*CH*$-$*CH*$=$CPh–), 7.19 (*t*, 2H, H_{para}), 7.33 (*t*, 4H, H_{meta}), 7.54 (*d*, 4H, H_{ortho}).

2.1.7. Synthesis of (5a) (see Fig. 2). A solution of K(1,3-$Ph_2C_5H_3$), which was obtained by adding a solution of KCH_2Ph (425 mg, 3.26 mmol) in THF (40 ml) to a stirred solution of (**3a**)/(**3b**) (693 mg, 3.18 mmol) in THF (40 ml), followed by stirring the resulting mixture for 1 h at room temperature, was added dropwise to a stirred suspension of $ScCl_3(THF)_3$ (1.132 g, 3.08 mmol) in THF (30 ml). The formed reaction mixture was stirred overnight at room temperature. A solution of $Na_2[C_2Ph_4]$, prepared from C_2Ph_4 (1.035 g, 3.11 mmol) in THF (70 ml) over a potassium mirror (975 mg, 25 mmol) for a period of 2 d, was added dropwise to the stirred reaction mixture over a period of 30 min at room temperature. The resulting mixture was then stirred for 2 d. All the THF was evaporated under vacuum. The product was extracted from the residue with toluene (3 × 50 ml). The resulting black–green solution was decanted from the precipitate. All the toluene was evaporated under vacuum. The residue was extracted with THF (3 × 30 ml). Almost all the THF was evaporated (to *ca* 3 ml) and the THF was then decanted. The resulting green microcrystals were washed with THF (3 × 1 ml), hexane (2 × 10 ml) and dried under vacuum. The yield was 933 mg (1.26 mmol, 41%).

^{1}H NMR (243 K, 300 MHz, THF-d_8): δ 4.54 (*br s*, 1H), 5.57 (*t*, 1H), 5.72 (*br s*, 1H), 5.99 (*t*, 1H), 6.20 (*dd*, 1H), 6.69–6.93 (*m*, 7H), 6.98 (*t*, 2H), 7.02–7.23 (*m*, 9H), 7.27 (*t*, 2H), 7.37 (*dt*, 1H), 7.45–7.51 (*m*, 4H), 7.59–7.64 (*m*, 2H), 8.03 (*dd*, 1H). UV–Vis (THF) λ_{max}: 210, 313, 382, 570 (*br sh*) nm. Analysis found (calculated for $C_{51}H_{49}O_2Sc$): Sc 5.89% (6.08%).

2.1.8. Crystallization of (5b). Hexane (27 ml) was layered on top of a solution of (**5a**) (82 mg) in toluene (23 ml). Crystals of (**5b**) suitable for X-ray diffraction analysis had formed in 5 d.

2.1.9. Synthesis and crystallization of (6) (see Fig. 3, middle reaction). Potassium (120 mg, 3.06 mmol) and a solution of anthracene (556 mg, 3.12 mmol) in THF (100 ml) were reacted for 15 h until all the potassium pieces were dissolved. The resulting blue solution of $KC_{14}H_{10}$ was added to a stirred suspension of $ScCl_3(THF)_3$ (368 mg, 1.00 mmol) in THF (10 ml). The colour of the reaction mixture turned deep violet. The mixture was stirred for 2 h. A solution of K(1,3-$Ph_2C_5H_3$), prepared from KCH_2Ph (135 mg, 1.04 mmol) and (**3a**)/(**3b**) (224 mg, 1.03 mmol) in THF (20 ml), was dropwise added to the reaction mixture, which was then stirred for 2 h. The mixture was centrifuged. All but the THF (*ca* 25 ml) was evaporated from the resulting solution under reduced pressure. Hexane (60 ml) was layered on top of the THF solution. After two weeks, the solution was decanted from the resulting black needle-shaped crystals. Some of these were taken for X-ray studies. The remaining crystals were washed with hexane (2 × 5 ml) and dried briefly under vacuum. The yield of crystals was 535 mg (0.36 mmol, 71%). Analysis found (calculated for $C_{96}H_{96}K_2O_5Sc_2$): Sc 6.12% (6.00%). The complex is poorly soluble in THF.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Non-H atoms were refined with individual anisotropic displacement parameters, with the exception of a toluene molecule in (**5b**) and a noncoordinating THF molecule in (**6**), which were refined with individual [toluene C atoms in (**5b**)] or equal [C and O atoms of the THF molecule in (**6**)] isotropic displacement parameters. The positions of the H atoms in all the Sc complexes [with the exception of the disordered molecules and fragments in (**1b**), (**5b**) and (**6**)] were found from difference maps. The positions of two H atoms per tetraphenylethylene dianion [atoms H4, H16, H30 and H42 in (**1b**), and atoms H4 and H16 in (**5b**)], interacting with the Sc^{3+} cation, were refined since they deviated from geometrically calculated positions, but the other H atoms in (**1b**) and (**5b**), and all the H atoms in (**6**) were positioned geometrically [$C-H = 0.95$ Å for aromatic, 0.98 Å for methyl, 0.99 Å for methylene and 1.00 Å for Cp (cyclopentadienyl) H atoms] and refined as riding atoms with relative isotropic displacement parameters [$U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ otherwise]. A rotating-group model was applied for the methyl groups. The positions and isotropic displacement parameters of all the H atoms in (**3a**) were refined due to the sufficiently good collected data. Only a major twinning component (over 95% according to *CELL_NOW*; Sheldrick, 2003) of (**3a**) was used in the refinement. For (**6**), reflection 110 was affected by the beam stop and was therefore omitted from the refinement.

The crystal structure of (**1b**) has a disordered CH_2-CH_2 fragment in a coordinated THF molecule with a disorder ratio of 0.639 (12):0.361 (12) for the C71*A*/C72*A* and C71*B*/C72*B* atoms. Displacement ellipsoids for some non-H atoms in the disordered fragment and in two noncoordinating THF molecules were restrained to approximately simulate isotropic behaviour. The toluene molecule in (**5b**) lies on a twofold rotation axis, and its C atoms are disordered equally over two overlapping sets of positions (see Fig. S1 in the supporting

Table 2
Selected bond lengths (Å) for (**3a**).

C1—C2	1.3615 (15)	C1—C5	1.5051 (14)
C2—C3	1.4548 (16)	C1—C6	1.4641 (14)
C3—C4	1.3598 (15)	C4—C12	1.4651 (14)
C4—C5	1.5077 (14)		

information). The noncoordinating THF molecule in (**6**) is disordered over three twofold rotation axes (25% site occupancies; see Fig. S3 in the supporting information). Atoms C22–C25 of the coordinated THF molecule in (**6**) are disordered over two positions (C22*A*–C25*A* and C22*B*–C25*B*), with a corresponding disorder ratio of 0.728 (10):0.272 (10). Similar C—O and C—C bond distances were restrained to be equal within 0.01 Å in this molecule. The disordered C atoms (C22*A*–C25*A* and C22*B*–C25*B*) still exhibited rather high atomic displacement parameters, but further refinement intended to resolve this problem only complicated the crystallographic model. Four atoms of the phenyl group in (**6**) are disordered equally [0.501 (5):0.499 (5)] over two positions (C5*A*–C8*A*/C5*B*–C8*B*). Similarity constraints were applied to the anisotropic displacement parameters of these atoms. In order to further improve the model, constraints were applied to the anisotropic displacement parameters of nine C atoms exhibiting high atomic displacement parameters to approximately simulate isotropic behaviour. This did not improve the crystallographic model significantly, but considerably changed the parameter/restraint ratio from 307/128 to 307/188. Therefore, the latter modelling was not used in the final model of (**6**).

3. Results and discussion

3.1. 1,4-Diphenylcyclopenta-1,3-diene, (3a)

Partial hydrolysis of (**4**) at low temperature resulted in the formation of a few crystals of thermodynamically favourable hydrocarbon 1,4-$\mathrm{Ph_2C_5H_4}$, (**3a**) (Figs. 2 and 5). The C—C bond lengths were as expected (Table 2). The five-membered C1–C5 ring is flat, with a maximum deviation of 0.0156 (6) Å for atom C5. The C_{ipso}(Ph) atoms are out of the plane by 0.1063 (18) Å for atom C6 and 0.1126 (18) Å for C12. The Cp–Ph (cyclopentadienyl–phenyl) dihedral angle is 10.50 (5)° for the C6–C11 phenyl ring and 11.66 (5)° for the C12–C17 phenyl ring. These data may be considered as reference parameters for 1,3-diphenylcyclopentadienyl complexes.

3.2. Tetraphenylethenide complexes with the homoleptic $[\mathrm{Sc(C_2Ph_4)_2}]^-$ complex anion

The reference points for the crystal structures of tetraphenylethenide rare-earth complexes are structures of alkali-metal adducts with tetraphenylethylene, *viz.* $[\mathrm{Na(Et_2O)_2}]^+_2[\mathrm{C_2Ph_4}]^{2-}$ (CSD refcode VEDXOR; Bock *et al.*, 1989), $[\mathrm{Na(diglyme)_2}]^+_2[\mathrm{C_2Ph_4}]^{2-}$ (FOGFUE; Minyaev & Ellis, 2014), $[\mathrm{K(DME)_2}]^+_2[\mathrm{C_2Ph_4}]^{2-}$ (XIDHIC; Minyaev *et al.*, 2007) and $[\mathrm{Cs(diglyme)}]^+_2[\mathrm{C_2Ph_4}]^{2-}$ (ZUXXIZ; Bock *et al.*, 1996). The C—C bond-length redistribution inside the dianion could be rationalized by localization of the negative charge predominantly at two atoms of the former double bond ($C_=$) and also at all eight C_{ortho} atoms [see Minyaev & Ellis (2014) for details]. However, the situation changes upon coordination of the easily polarizable dianionic ligand with the strongly polarizing $\mathrm{Sc^{3+}}$ cation.

Table 3
Selected bond lengths (Å) for (**1b**).

Sc1—C1	2.351 (4)	C16—C17	1.385 (6)
Sc1—C2	2.357 (4)	C17—C18	1.357 (6)
Sc1—C3	2.577 (4)	C18—C19	1.397 (6)
Sc1—C4	2.518 (4)	C19—C20	1.353 (5)
Sc1—C15	2.559 (4)	C15—C20	1.431 (5)
Sc1—C16	2.536 (4)	C2—C21	1.486 (5)
Sc1—C27	2.354 (4)	C27—C29	1.451 (5)
Sc1—C28	2.380 (4)	C29—C30	1.426 (5)
Sc1—C29	2.590 (4)	C30—C31	1.391 (6)
Sc1—C30	2.563 (4)	C31—C32	1.362 (6)
Sc1—C41	2.562 (4)	C32—C33	1.393 (6)
Sc1—C42	2.560 (4)	C33—C34	1.370 (6)
C1—C3	1.434 (5)	C29—C34	1.424 (5)
C3—C4	1.418 (6)	C27—C35	1.488 (5)
C4—C5	1.393 (5)	C27—C28	1.503 (5)
C5—C6	1.370 (6)	C28—C41	1.444 (5)
C6—C7	1.387 (6)	C41—C42	1.434 (5)
C7—C8	1.369 (6)	C42—C43	1.388 (5)
C3—C8	1.432 (5)	C43—C44	1.365 (6)
C1—C9	1.482 (6)	C44—C45	1.400 (6)
C1—C2	1.501 (5)	C45—C46	1.360 (5)
C2—C15	1.442 (5)	C41—C46	1.428 (5)
C15—C16	1.425 (6)	C28—C47	1.487 (5)

The ate complex (**1b**) contains the homoleptic $[\mathrm{Sc}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^-$ complex anion (Fig. 6*a*), the $[\mathrm{Na(18\text{-}crown\text{-}6)(THF)_2}]^+$ cation (Fig. 6*b*) and two noncoordinating THF molecules (not shown), which are well separated in the crystal structure. The Na—O(18-crown-6) bonds [2.663 (3) Å for Na1—O4 to 2.860 (3) Å for Na1—O2] are longer than the corresponding Na—O(THF) bonds [2.394 (3) Å for Na1—O7 and 2.355 (3) Å for Na1—O8] in the complex cation. In $[\mathrm{Sc}(\eta^6\text{-}\mathrm{Ph_4C_2})_2]^-$, the $\mathrm{Sc^{3+}}$ cation is nearly symmetrically coordinated by two η^6-$\mathrm{Ph_4C_2}^{2-}$ dianions, each of them exhibiting six short Sc—C contacts, *i.e.* to two atoms of the former double bond ($C_=$ is C1═C2 and C27═C28), two C_{ipso} atoms of two phenyl rings (C3/C15 and C29/C41) and two C_{ortho} atoms (C4/C16 and C30/C42) (Fig. 6*a* and Table 3). The determined positions of the H atoms at coordinated C_{ortho} atoms deviate noticeably from the corresponding phenyl-ring planes. Therefore, the geometrical positions of these H atoms (H4, H16, H30 and H42) were refined (see §2.2). Due to the reduction to the dianion, the length of a former double bond ($C_=$—$C_=$ = C1═C2 and C27═C28) is elongated to a single C—C bond

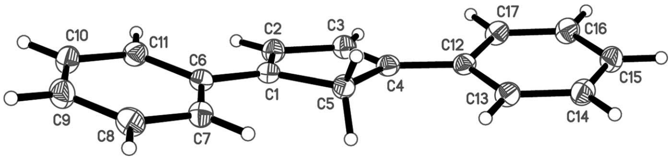
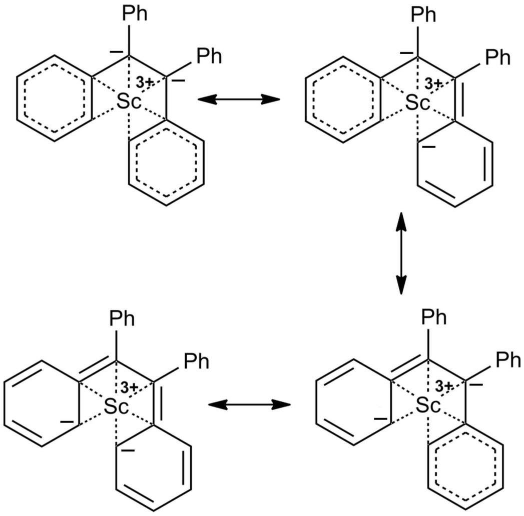


Figure 5
The molecular structure of 1,4-$\mathrm{Ph_2C_5H_4}$. Displacement ellipsoids are drawn at the 50% probability level.

(Table 3). The $\mathrm{Sc{-}C}_{=}$ bond lengths are considerably shorter than the $\mathrm{Sc{-}C}_{ipso}$ and $\mathrm{Sc{-}C}_{ortho}$ bond lengths. The $\mathrm{C}_{=}{-}\mathrm{C}_{ipso}$ distances for the noncoordinated phenyl groups are slightly longer than those for the coordinated phenyl groups. The C—C bond-distance redistribution within the coordinated phenyl rings and the $\mathrm{C}_{=}{-}\mathrm{C}_{ipso}$ and Sc—C bond lengths (Table 3) indicate that the tetraphenylethenide ligand displays a bis-η^3-allylic coordination mode. The Sc coordination number ($\mathrm{CN}_{\mathrm{Sc}}$) is therefore 8, since each allyl fragment occupies two coordination sites. The bis-η^3-allylic coordination type is consistent with the major resonance forms with predominant localization of an excessive negative charge at the $\mathrm{C}_{=}$ and coordinated C_{ortho} atoms (Scheme 2), unlike in alkali-metal $M_2[\mathrm{C_2Ph_4}]$ salts (see above). This relatively strong $\mathrm{Sc^{3+}}$–$\mathrm{C_2Ph_4}^{2-}$ interaction is also reflected in a considerable short wavelength shift of a LMCT (ligand-to-metal charge transfer) absorbance band as compared to $M_2[\mathrm{C_2Ph_4}]$ (M = Na and K) adducts (see below).



Scheme 2

The structures of similar homoleptic anions were determined previously for $[\mathrm{Na(THF)_6}]^+[\mathrm{Y}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^-\cdot 2(\mathrm{THF})$ (FIGQER; Roitershtein *et al.*, 1998), $[\mathrm{Na(diglyme)_2}]^+[\mathrm{Lu}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^-\cdot 0.5(\mathrm{THF})$ (KAMZII; Roitershtein *et al.*, 2004) and $[\mathrm{Na(THF)_3}]^+[\mathrm{Na(THF)_4}]^+[\mathrm{Yb}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^{2-}$ (XIDHEY; Minyaev *et al.*, 2007). Unlike the labile $[\mathrm{Yb}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^{2-}$ complex dianion of $\mathrm{Yb^{II}}$, the rather rigid $[\mathrm{Ln}(\eta^6\text{-}\mathrm{Ph_4C_2})_2]^-$ anions (Ln = Y and Lu) demonstrated retention of the bis-η^3-allylic ligand coordination mode in a THF-d_8 solution. They also displayed a temperature-dependent hindered rotation of the coordinated phenyl groups along the $\mathrm{C}_{=}{-}\mathrm{C}_{ipso}$ bonds, whereas the ^{1}H NMR spectrum for $\mathrm{Na_2[C_2Ph_4]}$ displayed a free rotation for all the phenyl groups at room temperature (Roitershtein *et al.*, 1998). ^{1}H NMR studies of (**1a**) and (**2a**) demonstrated a similar behaviour of the $[\mathrm{Sc}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^-$ anion in a THF-d_8 solution to the Lu and Y analogs. The ^{1}H NMR spectrum of (**2a**) and the NMR numbering scheme are shown in Fig. 4. The signals of atoms H_1, H_2, H_4 and H_5 are very broad at room temperature due to a noticeable rotation of the coordinated phenyl groups in relation the NMR time scale. The structures of these signals become resolved at *ca* 263 K and below (Fig. S6 in the supporting information). We have estimated previously the rotation barrier for coordinated phenyl groups to be *ca* 70 kJ mol^{-1} for $\mathrm{C_2Ph_4}^{2-}$ in $[(1,3\text{-}\mathrm{Ph_2C_5H_3})\mathrm{Lu(C_2Ph_4)(THF)}]$ (Roitershtein, Minyaev *et al.*, 2007) and *ca* 67 kJ mol^{-1} for the coordinated azobenzene dianion in a related complex $\{[(\mathrm{C_5H_5})\mathrm{Lu(THF)}]_2(\mu_2\text{-}\mathrm{N_2Ph_2})_2\}(\mathrm{THF})_2$ (NIXMOW; Roitershtein *et al.*, 1997; Petrov *et al.*, 2002). Qualitatively, the rotation barrier in the $[\mathrm{Sc}(\eta^6\text{-}\mathrm{Ph_4C_2})_2]^-$ anion is noticeably lower.

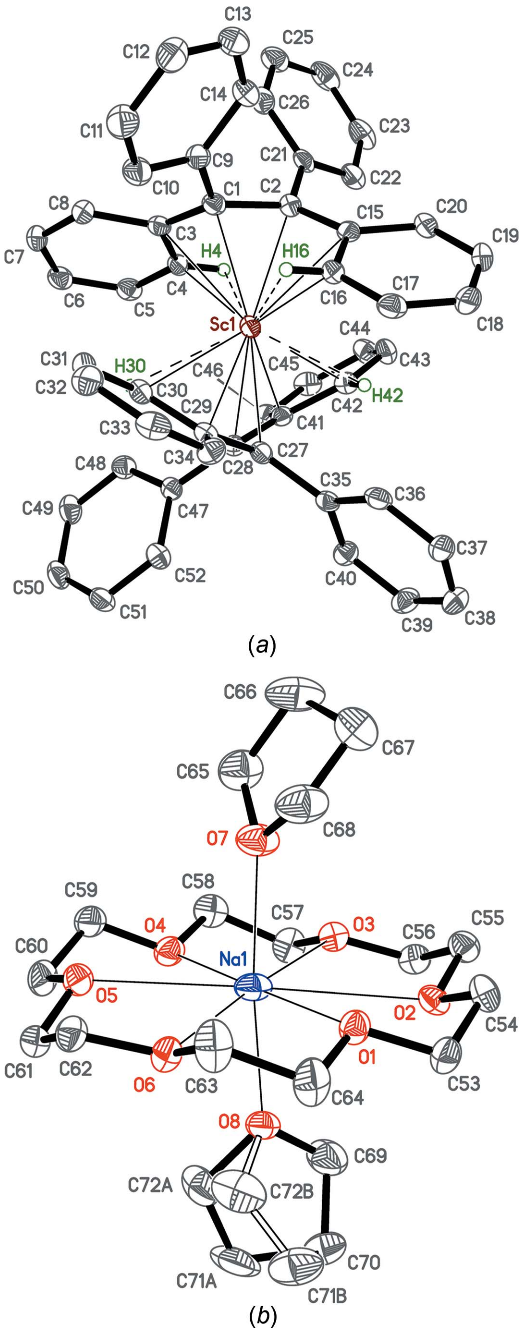


Figure 6
The structures of (*a*) the $[\mathrm{Sc}(\eta^6\text{-}\mathrm{C_2Ph_4})_2]^-$ anion and (*b*) the $[\mathrm{Na(18\text{-}crown\text{-}6)(THF)_2}]^+$ cation in (**1b**). The minor component of the THF disorder is shown with solid open lines. Displacement ellipsoids are drawn at the 50% probability level and all but the H atoms interacting with Sc atoms have been omitted for clarity.

Table 4
The ^{13}C NMR signals for the $[Ln(C_2Ph_4)_2]^-$ anions (Ln = Y, Lu and Sc) in THF-d_8 at room temperature.

Data for $K[Y(C_2Ph_4)_2]$ and $K[Lu(C_2Ph_{42}]$ are taken from the literature and any supporting information files (Roitershtein *et al.*, 1998, 2004).

C atoms	$K[Y(C_2Ph_4)_2]$	$K[Lu(C_2Ph_4)_2]$	(**1a**)	(**2a**)
$C_=$	88.3	86.8	86.1	86.2
C-1	120.1	120.6	118.6	118.6
C-2	131.2	133.2	129.7	129.8
C-3	111.5	112.6	112.0	112.1
C-4	138.9	141.5	137.4	137.4
C-5	99.6	98.1	100.5	100.5
C-6	139.0	141.1	137.8	137.8
C_{ortho}	129.2	130.4	128.1	128.1
C_{meta}	127.7	128.5	125.5	125.5
C_{para}	120.9	122.1	119.4	119.5
C_{ipso}(Ph)	145.9	146.6	144.3	144.3

Table 5
Selected bond lengths (Å) for (**5b**).

Sc1—C1	2.312 (2)	C5—C6	1.366 (4)
Sc1—C2	2.392 (2)	C6—C7	1.404 (4)
Sc1—C3	2.522 (2)	C7—C8	1.366 (3)
Sc1—C4	2.547 (2)	C3—C8	1.432 (3)
Sc1—C15	2.589 (2)	C1—C9	1.498 (3)
Sc1—C16	2.465 (2)	C2—C15	1.433 (3)
Sc1—C27	2.565 (2)	C15—C16	1.434 (3)
Sc1—C28	2.553 (2)	C16—C17	1.406 (4)
Sc1—C29	2.546 (2)	C17—C18	1.366 (4)
Sc1—C30	2.489 (2)	C18—C19	1.400 (4)
Sc1—C31	2.490 (2)	C19—C20	1.367 (4)
Sc1—O1	2.2024 (17)	C15—C20	1.431 (3)
Sc1—$Cp_{centroid}$	2.224 (1)	C2—C21	1.481 (3)
$C_{Cp}\cdots C_{Cp}$ (ave)	1.415 (6)	C27—C32	1.482 (3)
C1—C3	1.434 (3)	C29—C38	1.480 (3)
C3—C4	1.439 (3)	C1—C2	1.506 (3)
C4—C5	1.409 (3)		

The proton signal assignments in (**1a**) and (**2a**) were based on those for $[Ln(C_2Ph_4)_2]^-$ (Ln = Lu and Y) and on the COSY (correlation spectroscopy) ^{1}H–^{1}H spectrum (Fig. S7 in the supporting information). The most upfield shifted signal, *i.e.* H_5 (4.7 ppm; Fig. 4), corresponds to H atoms interacting with Sc^{3+} (H4, H16, H30 and H42; Fig. 6*a*).

The ^{13}C{^{1}H} spectra of (**1a**) and (**2a**), recordered at room temperature, display broad signals of the C_1, C_2, C_4 and C_5 atoms. The 2D ^{1}H–^{13}C HSQC (heteronuclear single quantum coherence; Fig. S8 in the supporting information) and HMBC (heteronuclear multiple-bond correlation; Fig. S9 in the supporting information) spectra of (**1a**) allowed us to make the ^{13}C signal assignments presented in Table 4, along with the data for the related Y and Lu complexes. The highest upfield chemical shifts for the $C_=$ and C_5 atoms in all the title compounds are consistent with the localization of excessive negative charge at these atoms and the bis-allylic coordination type of the dianionic ligand.

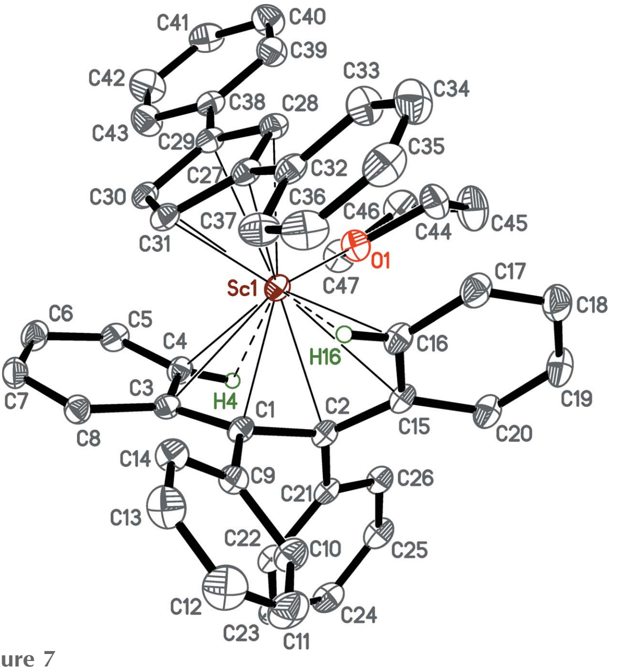


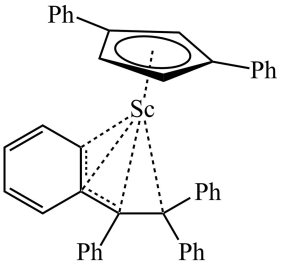
Figure 7
The molecular structure of $[(\eta^5\text{-}1,3\text{-}Ph_2C_5H_3)Sc(\eta^6\text{-}C_2Ph_4)(THF)]$ in (**5b**). Displacement ellipsoids are drawn at the 30% probability level and all but two H atoms have been omitted for clarity.

For a better understanding of the charge redistribution in ate complexes, a ^{45}Sc NMR spectrum of (**2a**) (Fig. S10 in the supporting information) was recorded using $ScCl_3(THF)_3$ (Fig. S11 in the supporting information) as a reference (203 ppm in THF-d_8; Mason, 1987). A strong upfield shift of the scandium signal [64 ppm in (**2a**)] compared to that of $ScCl_3(THF)_3$ suggests that an essential part of the electron density is shifted from the dianionic ligand to the Sc^{3+} cation, which supports the idea of a significant covalent contribution to the Sc^{3+}–$C_2Ph_4{}^{2-}$ ionic interaction (see also UV–Vis spectral data below).

3.3. Heteroleptic tetraphenylethenide complex $[(1,3\text{-}Ph_2C_5H_3)Sc(C_2Ph_4)(THF)]$

The crystal structure of (**5b**) contains the complex $[(\eta^5\text{-}1,3\text{-}Ph_2C_5H_3)Sc(\eta^6\text{-}C_2Ph_4)(THF)]$ (Fig. 7) and a toluene molecule (Fig. S1 in the supporting information). The tetraphenylethylene dianion exhibits the unsymmetrical bis-η^3-allylic coordination type (compare the Sc—C and C—C bond lengths within the two allylic fragments; Table 5). The $1,3\text{-}Ph_2C_5H_3$ ligand is η^5-coordinated. The Sc atom is additionally coordinated by a THF molecule (CN_{Sc} = 8). The C_{Cp}—C_{Cp} distances vary from 1.405 (3) (C30—C31) to 1.423 (3) Å (C28—C29), with an average value of 1.415 (6) Å. The Cp–Ph dihedral angle is 10.49 (18)° for the C32–C37 phenyl ring and 4.08 (18)° for the C38–C43 phenyl ring, and the C_{ipso}(Ph) atom deviation from the Cp plane is 0.150 (4) Å for C32 and 0.073 (4) Å for C38, being somewhat different compared to corresponding data for (**3a**). However, the overall molecular structure of the complex in (**5b**) is very similar to that of $[(\eta^5\text{-}1,3\text{-}Ph_2C_5H_3)\text{-}Lu(\eta^6\text{-}C_2Ph_4)(THF)]$ (CN_{Lu} = 8; IFUHIB; Roitershtein, Minyaev *et al.*, 2007) and $[(\eta^5\text{-}C_5H_5)Lu(\eta^6\text{-}C_2Ph_4)(\kappa^2O,O'\text{-}DME)](DME)$ (DME is dimethoxyethane; CN_{Lu} = 9; KAMZOO; Roitershtein *et al.*, 2004).

According to comparisons of the ^{1}H NMR spectra of (**1**), (**4**) and (**5a**), recordered in THF-d_8, (**5a**) exhibits temperature-dependent hindered rotation of only one phenyl group of the dianionic ligand [unlike complexes (**1a**) and (**2a**)] and the phenyl groups of the Cp ligand. Therefore, the coordination type of the dianion changes in solution from η^6 to η^4 (Scheme 3). This has been observed previously for analogous Lu complexes $[(\eta^5\text{-Cp}')\text{Lu}(\eta^6\text{-C}_2\text{Ph}_4)(\text{THF})_2]$ (Cp′ = C_5H_5 or 1,3-$Ph_2C_5H_3$; Roitershtein *et al.*, 2004; Roitershtein, Minyaev *et al.*, 2007).



Scheme 3

3.4. UV–Vis data of tetraphenylethenide complexes

An efficient interaction between an organic anionic ligand with a metal cation is generally reflected in a strong short wavelength shift of a ligand-to-metal charge transfer (LMCT) absorbance band as compared to alkali-metal salts of the anion (Szwarc, 1972). The LMCT absorbance bands of various tetraphenylethylenide complexes are summarized in Table 6. All tetraphenylethenide Ln^{III} complexes demonstrate a significant short wavelength shift of the LMCT band, indicating the tight and efficient ligand coordination to the metal cation in THF solution. The $[\text{Ln}(\text{C}_2\text{Ph}_4)_2]^-$ anions have closely similar $\Delta\lambda_{max}$ values (80–94 nm). An interesting trend is seen for the Cp derivative, namely that the smallest $\Delta\lambda_{max}$ value is observed for the complex bearing electron-donating substituents (1,3-$TMS_2C_5H_3$), whereas the highest λ_{max} shift is observed for (**5a**), in which the 1,3-$Ph_2C_5H_3$ ligand displays a more pronounced electron-withdrawing property. This is

Table 6
LMCT absorbance bands for tetraphenylethylenide complexes in THF.

$\Delta\lambda_{max}$ is a short wavelength shift of a LMCT band with respect to that of $M_2[C_2Ph_4]$ (M = Na and K).

1	$Na_2[C_2Ph_4]$ and $K_2[C_2Ph_4]$	485	–	Roberts & Szwarc (1965)
2	$Na_2[Yb(C_2Ph_4)_2]$	365, 475	–*	Minyaev *et al.* (2007)
3	$K_2[Yb(C_2Ph_4)_2]$	370, 495	–*	Minyaev *et al.* (2007)
4	$Na[Y(C_2Ph_4)_2]$	405	80	Roitershtein *et al.* (1998)
5	$Na[Lu(C_2Ph_4)_2]$	395	90	Roitershtein *et al.* (2004)
6	$K[Lu(C_2Ph_4)_2]$	397	88	Roitershtein *et al.* (2004)
7	$Na[Sc(C_2Ph_4)_2]$, (**1a**)	392	93	This work
8	$K[Sc(C_2Ph_4)_2]$, (**2a**)	391	94	This work
9	$(1,3\text{-TMS}_2C_5H_3)Lu(C_2Ph_4)$	421	64	Unpublished results
10	$(C_5H_5)Lu(C_2Ph_4)$	410	75	Roitershtein *et al.* (2004)
11	$(1,3\text{-Ph}_2C_5H_3)Lu(C_2Ph_4)$	390	95	Roitershtein, Minyaev *et al.* (2007)
12	$(1,3\text{-Ph}_2C_5H_3)Sc(C_2Ph_4)$, (**5a**)	382	103	This work

Note: (*) the λ_{max} values of labile complexes $M_2[Yb(C_2Ph_4]$ are likely due to superposition of λ_{max} for $M_2[Yb(C_2Ph_4)_2]$ and $M_2[(C_2Ph_4)]$ because of existing dynamic equilibrium in solution (Minyaev *et al.*, 2007): $M_2[Yb(C_2Ph_4)_2] = [Yb(C_2Ph_4)(THF)_4] + M_2[C_2Ph_4]$.

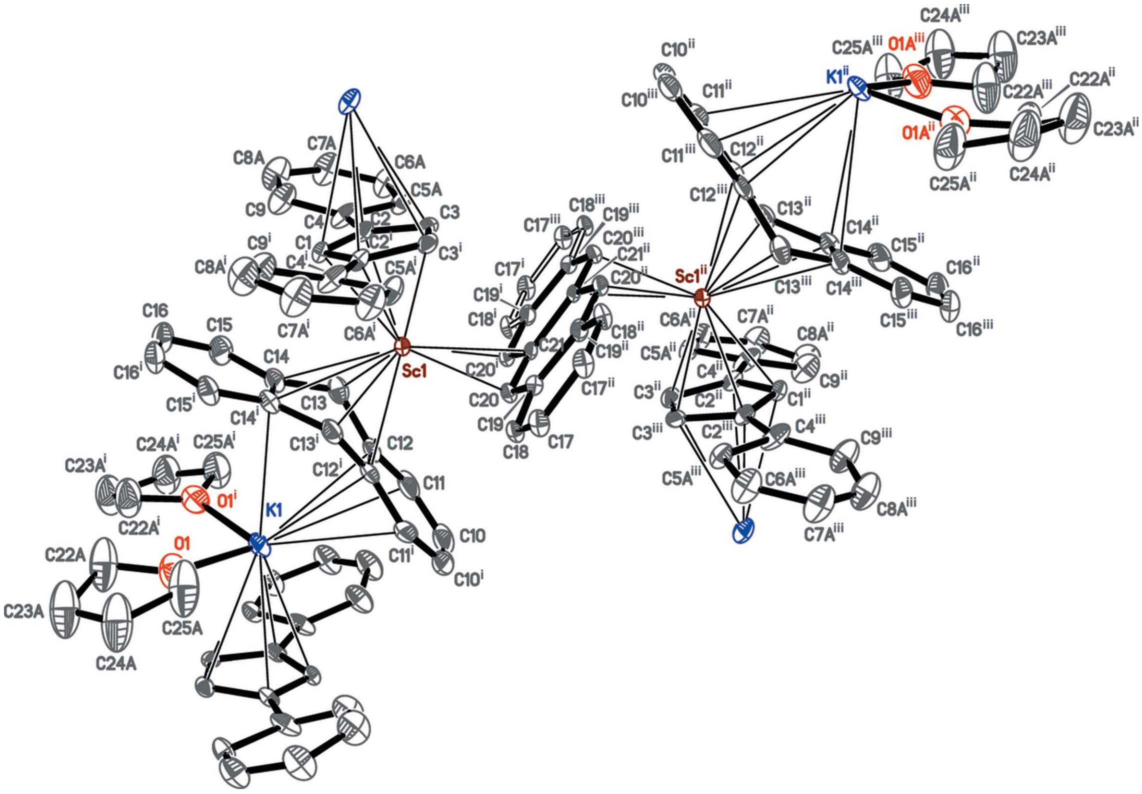


Figure 8
The structure of the $[K(THF)_2]_2[(1,3\text{-Ph}_2C_5H_3)_2Sc_2(\eta^6\text{-}C_{14}H_{10})_2(\mu_2\text{-}\eta^3{:}\eta^3\text{-}C_{14}H_{10})]$ unit in (**6**). H atoms have been omitted and disorder is not shown. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) $x, y, -z + 1$; (ii) $-x + 1, -y, -z + 1$; (iii) $-x + 1, -y, z$.]

Table 7
Selected bond lengths (Å) for (**6**).

Sc1—C1	2.522 (4)	C2—C3	1.418 (4)
Sc1—C2	2.512 (3)	C2—C4	1.478 (4)
Sc1—C2^{i}	2.512 (3)	C3—C3^{i}	1.400 (6)
Sc1—C3	2.470 (3)	C10—C10^{i}	1.362 (10)
Sc1—C3^{i}	2.470 (3)	C10—C11	1.385 (6)
Sc1—C12	2.616 (3)	C11—C12	1.415 (5)
Sc1—C12^{i}	2.616 (3)	C12—C12^{i}	1.428 (7)
Sc1—C13^{i}	2.348 (3)	C12—C13	1.450 (5)
Sc1—C13	2.348 (3)	C13—C14	1.474 (4)
Sc1—C14^{i}	2.874 (3)	C14—C15	1.397 (4)
Sc1—C14	2.874 (3)	C14—C14^{i}	1.418 (6)
Sc1—C20	2.524 (3)	C15—C16	1.383 (5)
Sc1—C20^{i}	2.524 (3)	C16—C16^{i}	1.380 (8)
Sc1—C21	2.582 (3)	C19—C18	1.281 (5)
K1—O1	2.605 (3)	C19—C19^{iii}	1.393 (5)
K1—C1^{ii}	3.079 (4)	C19—C20	1.438 (4)
K1—C2^{ii}	3.106 (3)	C17—C17^{iii}	1.390 (11)
K1—C3^{ii}	3.138 (3)	C17—C18	1.371 (7)
K1—C11	3.431 (3)	C20—C21	1.411 (3)
K1—C12	3.020 (3)	C21—C21^{iv}	1.466 (7)
K1—C14	3.404 (3)	Sc1—Cp$_{centroid}$	2.190 (2)
C1—C2	1.411 (3)		

Symmetry codes: (i) $x, y, -z+1$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z$; (iii) $-x+1, -y, z$; (iv) $-x+1, -y, -z+1$.

consistent with partial electron-density transfer from the dianionic ligand *via* the Ln^{3+} cation to the antibonding orbital of the Cp′ ligand in Cp′Ln(C$_2$Ph$_4$) complexes (Roitershtein, Minyaev *et al.*, 2007).

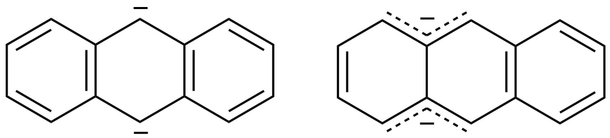
3.5. Crystal structure of (6)

To the best of our knowledge, the crystal structure of (**6**) is entirely unique. The crystal data for (**6**) (crystals were obtained as shown in Fig. 3, upper synthesis) were initially collected on a Bruker SMART CCD Platform. Due to rather high R_{int} value (16.4%) and the presence of many disordered atoms after refinement, its synthesis was repeated under different conditions (Fig. 3, middle synthesis); and better crystal data were recollected on an Bruker APEXII CCD instrument. The second refinement demonstrated that the presence of many disordered atoms is an intrinsic property of the structure.

The crystal structure of (**6**) is highly symmetrical. A quarter of the compound (**6**) (Fig. 8) is crystallographically unique (see Fig. S2 in the supporting information; $Z' = 0.25$). Atoms Sc1, K1, C1, H1 and C21 lie in the same mirror plane. The remaining atoms are generated by this mirror plane (the symmetry operation is $x, y, -z+1$) and by a twofold rotation axis (the symmetry operation is $-x+1, -y, -z+1$). One anthracenide ligand (atoms C17–C21 and their symmetrical equivalents; Fig. 8) lies on both the mirror plane and the twofold rotation axis (see Fig. S4 in the supporting information for disorder details); therefore, the occupancies for atoms Sc1, K1, C1, H1, C17, H17, C18, H18 and H19 are 50%. Selected bond distances in (**6**) are presented in Table 7.

Perhaps, the most striking feature of (**6**) is the presence of two different coordination modes of the anthracene dianion. Two anthracenide ligands display the η^6-coordination mode. Sometimes in the literature, it is also referred to as η^2 or even η^4 for an unsymmetrical bonding type for rare-earth anthracenide compounds. The Sc^{3+} cation in (**6**) is bound to C atoms at the 9- and 10-positions (atoms C13 and C13^{i}; Fig. 8) of the bent anthracene dianion and has four additional short contacts with C atoms (C12, C12^{i}, C14 and C14^{i}). The ligand folding angle between planes defined by the C10–C13/C10^{i}–C13^{i} and C13–C16/C13^{i}–C16^{i} atoms is 33.74 (12)°. The other anthracenide ligand is planar and exhibits the μ_2-η^3:η^3 bridging bis-allylic coordination mode. This coordination type is also common for rare-earth anthracenide complexes. However, (**6**) is the only example displaying both coordination modes for the anthracene dianion, according to the CSD (Groom *et al.*, 2016). The Sc—C and C—C bond distances in the anthracenide ligands are consistent with the anticipated major resonance forms (Scheme 4).

Besides coordination with two dianions, the Sc^{3+} cation is also η^5-coordinated by a 1,3-diphenylcyclopentadienyl ligand with a Cp(centroid)—Sc distance of 2.190 (2) Å. The Cp–Ph dihedral angles in the 1,3-Ph$_2$C$_5$H$_3$ ligand are 19.0 (2)° (for the C4/C5*A*–C8*A*/C9 phenyl ring) and 27.4 (2)° (for the C4/C5*B*–C8*B*/C9 phenyl ring), indicating noticeable steric hindrance [see data for (**3a**)]. The C$_{ipso}$(Ph) deviations from the Cp plane are 0.065 (4) Å (atoms C4 and C4^{i}), directed outward from Sc^{3+}. The K^{+} cation is bound to two THF molecules and displays six short contacts with the bent dianion [atoms C11, C12, C14, C11^{i}, C12^{i} and C14^{i}; symmetry code: (i) $x, y, -z+1$; Table 7]. The K^{+} cation also has contacts to the 1,3-Ph$_2$C$_5$H$_3$ ligand of another [(1,3-Ph$_2$C$_5$H$_3$)$_2$Sc$_2$(C$_{14}$H$_{10}$)$_3$]$^{2-}$ dianionic unit [atoms C1^{iv}, C2^{iv} and C3^{iv}; symmetry code: (iv) $x-\frac{1}{2}$, $-y+\frac{1}{2}$, z; Table 7] with a Cp$_{(centroid)}$—K distance of 2.873 (2) Å. Thus, complex (**6**) itself represents an example of a 2D (two-dimensional) coordination polymer {[K(THF)$_2$]$_2$Sc$_2$(1,3-Ph$_2$C$_5$H$_3$)$_2$(C$_{14}$H$_{10}$)$_3$}$_\infty$, where coordination between neighboring [Sc$_2$(1,3-Ph$_2$C$_5$H$_3$)$_2$(C$_{14}$H$_{10}$)$_3$]$^{2-}$ dianionic units occurs *via* short K···C contacts with [K(THF)$_2$]$^{+}$ units: one unit has interactions with four [K(THF)$_2$]$^{+}$ cations, and each cation connects two units (see Fig. 8 and Fig. S5 in the supporting information). The 2D layers of {[K(THF)$_2$]$_2$Sc$_2$(1,3-Ph$_2$C$_5$H$_3$)$_2$(C$_{14}$H$_{10}$)$_3$}$_\infty$ are parallel to (001) (Fig. S5). Upon formation, crystals of (**6**) are poorly soluble in THF, probably due to its coordination polymer structure.



Scheme 4

3.6. The CSD survey

All known crystal structures with the tetaphenylethylene dianion have been discussed above. No crystal structures of any other rare-earth, alkaline-earth and *d*-metals are known where the tetraphenylethylene ligand bears a negative charge. Moreover, only three structures of Ag^{+} π-complexes with an uncharged C$_2$Ph$_4$ ligand have been reported to date (Ino *et al.*, 2000). Therefore, this ligand remains unique within the organometallic chemistry of *d*- and rare-earth metals.

The crystal structures or rare-earth metals bearing polyphenyl-substituted cyclopentadienyl ligands are still poorly explored (Roitershtein *et al.*, 2012; Minyaev *et al.*, 2016). This especially concerns complexes with 1,3-diphenyl- and 1,2,4-triphenylcyclopentadienyl ligands. Here, we bring attention to known crystal structures containing the 1,3-diphenylcyclopentadienyl ligand and the anthracene dianion.

3.6.1. Crystal structures substituted with 1,4-diphenylcyclopenta-1,3-dienes. The crystal structures of 1,3- or 1,4-diphenylcyclopenta-1,3-dienes have not been determined to date, likely due to the facile 1,2-prototropic shift induced even by slightly elevated temperatures (Prinzhuch & Thyes, 1971) during purification, and leading to a thermodynamic mixture of 1,3- and 1,4-isomers (^{1}H NMR data), whereas three structures of analogous perfluorinated 1,4-diphenylcyclopenta-1,3-dienes are known, namely 1,4-bis(pentafluorophenyl)cyclopenta-1,3-diene (ACEDAN/ACEDAN01; Thornberry *et al.*, 2000), dimethyl 4,4′-cyclopenta-3,5-diene-1,3-diylbis(tetrafluorobenzoate) (POCTIM; Enk *et al.*, 2009) and 1,4-bis(perfluoro-4-tolyl)cyclopentadiene (YEJCAS; Deck *et al.*, 2006). Moreover, the crystal structures of 1,2,4-triphenyl-, 1,2,3,4-tetraphenyl- and 1,2,3,4,5-pentaphenylcyclopenta-1,3-dienes and their substituted derivatives are known (22 structures in the CSD, Version 5.39, latest update of February 2018; Groom *et al.*, 2016).

3.6.2. Crystal structures with the 1,3-diphenylcyclopentadienyl ligand. Rare-earth complexes with the 1,3-$Ph_2C_5H_3$ ligand are not numerous, *viz.* the two isostructural complexes (1,3-$Ph_2C_5H_3$)$LnCl_2(THF)_3$ [Ln = Lu (IFUHEX; Roitershtein, Minyaev *et al.*, 2007) and Yb (OFUHEE; Roitershtein *et al.*, 2012)] and complex (1,3-$Ph_2C_5H_3$)$Lu(C_2Ph_4)(THF)$ (IFUHIB; Roitershtein, Minyaev *et al.*, 2007). The crystal structures of other metal complexes are represented by (1,3-$Ph_2C_5H_3$)$(C_5H_5)ZrCl_2$ (EYUSIA; Thornberry *et al.*, 2004), (1,3-$Ph_2C_5H_3)_2TiCl_2$ (PCYPTC; van Soest *et al.*, 1973), [(1,3-$Ph_2C_5H_3$)$Ru(CO)(\mu_2\text{-}CO)]_2$·(toluene) (VUZYOE; Schumann *et al.*, 2002), [(1,3-$Ph_2C_5H_3$)$Ru_6C(CO)_{12}(\mu_2\text{-}CO)(\mu_3\text{-}CPh)$] (YUZPEO; Haggitt *et al.*, 1995), (1,3-$Ph_2C_5H_3)_2Fe$ (YOCWOC; Schumann *et al.*, 1995), $[(\mu_2\text{-}1,3\text{-}Ph_2C_5H_3)_2Tl_2(\mu_2\text{-}THF)_2]_\infty$ (YOCWIW; Schumann *et al.*, 1995) and $\{(\mu_2\text{-}\eta^5{:}\eta^6\text{-}1,3\text{-}Ph_2C_5H_3)[Mn(CO)_3][Cr(CO)_3]\}$ (NOYCUZ; Kang *et al.*, 1997). There are six related *d*-metal complexes bearing various substituents in the phenyl rings of the 1,3-diarylcyclopentadienyl ligand, including ansa complexes and heterobimetallic derivatives, *i.e.* ACEFAP (Thornberry *et al.*, 2000), NEYDEB and NEYDIF (Muraoka *et al.*, 2007), NOYTIG (Sasamori *et al.*, 2014), QONVUL (Tanaka & Kinbara, 2008) and UHIWOX (Muraoka *et al.*, 2003). Therefore, according to the CSD, the crystal structures of *f*- and even *d*-metal complexes with this ligand are still poorly explored. One possible reason for this is that the corresponding precursor, namely diphenylcyclopentadiene, is less synthetically available than other polyphenyl-substituted cyclopentadienes.

3.6.3. Crystal structures of rare-earth metals with the anthracene dianion. Previously described crystal structures of rare-earth anthracenide complexes exhibit three types of ligand binding modes. The μ_2-η^3:η^3 bis-allylic coordination mode can be found in structures where two Ln^{3+} cations are coordinated by a flat anthracene ligand *via* the C atoms at the 8, 8a and 9, and 4, 4a and 10 ligand positions. This ligand type was found in $\{(\mu_2\text{-}\eta^3{:}\eta^3\text{-}C_{14}H_{10})[La(C_5Me_5)_2]_2\}$ (NAGSOD; Thiele *et al.*, 1996) and $\{(\mu_2\text{-}\eta^3{:}\eta^3\text{-}C_{14}H_{10})[Sm(C_5Me_5)_2]_2\}$·(toluene) (WEVNAM; Evans *et al.*, 1994). The bis-allylic coordination type is also observed in the related tetracene complex $\{(\mu_2\text{-}\eta^3{:}\eta^3\text{-}C_{18}H_{12})[Sm(C_5Me_5)_2]_2\}$·(benzene) (WEVNOA; Evans *et al.*, 1994). Another coordination type of a bent anthracene dianion involves positions 9 and 10 (short contacts *via* positions 4a, 8a, 9a and 10a may also be present). This coordination type is displayed by heavier lanthanide complexes and Y, *i.e.* $[(C_{14}H_{10})Lu(C_5H_4CH_2CH_2PPh_2)(DME)]$ (QOFFIB; Roitershtein, Romanenkov *et al.*, 2007), $[(C_{14}H_{10})Lu(C_5H_5)(THF)_2]$ (VUJDAF; Roitershtein *et al.*, 1992), $[Na(diglyme)_2]^+[(C_{14}H_{10})Lu(C_5H_5)_2]^-$ (YAXSUL; Roitershtein *et al.*, 1993), $[(C_{14}H_{10})TmI(DME)_2]$ (UCOXEP; Fedushkin *et al.*, 2001), and $[K(18\text{-crown-}6)]^+[(C_{14}H_{10})Y(C_5H_4NSiMe_2{}^tBu)_2Fe]^-\cdot Et_2O$ (YEMYEW; Huang *et al.*, 2013).

Bulky dianionic ligands, such as in the latter case (YEMYEW), do not allow two Ln^{3+} cations to be coordinated by the anthracene dianion *via* the allylic positions (μ_2-η^3:η^3 coordination type), even in the case of small Sc^{3+}. Instead, one cation is coordinated *via* the 9- and 10-positions of the bent dianion, whereas the second cation is η^4- or η^6-bound to a nearly flat terminal ring of the anthracenide ligand (the third coordination type): $\{(C_{14}H_{10})[Ln(C_5H_4NSiMe_2{}^tBu)_2Fe(THF)]_2\}$ [Ln = Y (SIRRAO; Huang *et al.*, 2014) and Sc (UXUMIK; Huang *et al.*, 2011)] and $[(C_{14}H_{10})\{Y[PhP(CH_2SiMe_2NSiMe_2CH_2)_2PPh]_2\}]$ (WIYLIZ; Fryzuk *et al.*, 2000). In these complexes with two Ln^{3+} cations, the folding angle of the anthracene dianion is noticeably smaller than in the cases of complexes with one coordinated Ln^{3+} cation. $\{(C_{14}H_{10})[Sc(C_5H_4NSiMe_2{}^tBu)_2Fe(THF)]_2\}$ is the only Sc–anthracenide complex known to date.

4. Concluding remarks

The tetraphenylethenide ligand in (**1b**) demonstrates a symmetrical bis-η^3-allyl coordination mode, which was reported previously for analogous homoleptic anions $[Ln(\eta^6\text{-}C_2Ph_4)_2]^{n-}$ (Ln = Y, Lu, *n* = 1; Ln = Yb, *n* = 2; Roitershtein *et al.*, 1998, 2004; Minyaev *et al.*, 2007). The bis-η^3-allyl coordination mode is retained in THF solutions of (**1a**) and (**2a**). Complex (**5b**) exhibits an unsymmetrical bis-η^3-allyl coordination mode of the dianion in the crystal structure, but this changes to η^4 in THF-d_8 solution, analogous to what is observed for complexes $[(C_5H_5)Lu(C_2Ph_4)(THF)_2]$ and [(1,3-$Ph_2C_5H_3$)$Lu(C_2Ph_4)(THF)$] (Roitershtein *et al.*, 2004; Roitershtein, Minyaev *et al.*, 2007). Based on X-ray diffraction, NMR and UV–Vis studies, we have demonstrated the presence of a significantly covalent contribution to the ionic $Sc^{3+}{-}C_2Ph_4{}^{2-}$ bonding. The unique ate complex (**6**) contains a complex $[Sc_2(1,3\text{-}Ph_2C_5H_3)_2(C_{14}H_{10})_3]^{2-}$ dianionic unit, which displays both the η^6 and μ_2-η^3:η^3 coordination modes of the anthracene ligands. The dianionic unit and $[K(THF)_2]^+$ cations form a 2D coordination polymer structure.

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Scandium complexes with the tetraphenylethylene and anthracene dianions

John E. Ellis, Mikhail E. Minyaev, Ilya E. Nifant'ev and Andrei V. Churakov

Computing details

Data collection: *SMART* (Bruker, 2003) for (1b), (3a), (5b); *APEX2* (Bruker, 2008) for (6). Cell refinement: *SAINT* (Bruker, 2003) for (1b), (3a), (5b); *SAINT* (Bruker, 2008) for (6). Data reduction: *SAINT* (Bruker, 2003) for (1b), (3a), (5b); *SAINT* (Bruker, 2008) for (6). Program(s) used to solve structure: *SHELXS* (Sheldrick, 2008) for (1b), (5b), (6); *SIR2002* (Burla *et al.*, 2003) for (3a). For all structures, program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(18-Crown-6)bis(tetrahydrofuran-κO)sodium bis(η^6-1,1,2,2-tetraphenylethenediyl)scandium(III) tetrahydrofuran disolvate (1b)

Crystal data

$[Na(C_4H_8O)_2(C_{12}H_{24}O_6)][Sc(C_{26}H_{20})_2]\cdot 2C_4H_8O$
M_r = 1285.51
Triclinic, $P\overline{1}$
a = 12.6071 (18) Å
b = 14.764 (2) Å
c = 18.585 (3) Å
α = 84.507 (2)°
β = 88.508 (2)°
γ = 84.033 (2)°
V = 3424.2 (9) Å^3
Z = 2
$F(000)$ = 1376
D_x = 1.247 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4092 reflections
θ = 2.2–27.8°
μ = 0.17 mm^{-1}
T = 173 K
Needle, red
0.40 × 0.25 × 0.25 mm

Data collection

SMART Platform CCD area detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
T_{min} = 0.869, T_{max} = 0.967
32161 measured reflections
11986 independent reflections
6407 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 25.0°, θ_{min} = 1.1°
$h = -14\rightarrow 14$
$k = -17\rightarrow 17$
$l = -22\rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.183
S = 1.02
11986 reflections
860 parameters
46 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 1.9339P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Special details

Experimental. moisture and air sensitive

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Sc1	0.79311 (6)	0.73855 (5)	0.22090 (4)	0.0275 (2)	
C1	0.8864 (3)	0.8495 (3)	0.1545 (2)	0.0311 (10)	
C2	0.9393 (3)	0.7566 (3)	0.1411 (2)	0.0324 (10)	
C3	0.7843 (3)	0.8736 (3)	0.1218 (2)	0.0309 (10)	
C4	0.7245 (3)	0.8055 (3)	0.0991 (2)	0.0306 (10)	
H4	0.755 (3)	0.746 (3)	0.096 (2)	0.037*	
C5	0.6214 (3)	0.8255 (3)	0.0729 (2)	0.0363 (10)	
H5	0.583860	0.777447	0.059214	0.044*	
C6	0.5732 (3)	0.9133 (3)	0.0666 (2)	0.0409 (11)	
H6	0.502078	0.926623	0.050116	0.049*	
C7	0.6312 (3)	0.9824 (3)	0.0849 (2)	0.0407 (11)	
H7	0.599837	1.043934	0.078738	0.049*	
C8	0.7321 (3)	0.9647 (3)	0.1117 (2)	0.0373 (10)	
H8	0.768566	1.014204	0.123904	0.045*	
C9	0.9536 (3)	0.9178 (3)	0.1775 (2)	0.0346 (10)	
C10	0.9163 (4)	0.9895 (3)	0.2191 (2)	0.0432 (11)	
H10	0.843051	0.996628	0.232911	0.052*	
C11	0.9838 (4)	1.0503 (3)	0.2408 (3)	0.0519 (13)	
H11	0.956080	1.098111	0.268884	0.062*	
C12	1.0901 (4)	1.0419 (3)	0.2219 (3)	0.0574 (14)	
H12	1.136157	1.083214	0.237006	0.069*	
C13	1.1289 (4)	0.9731 (3)	0.1809 (3)	0.0557 (14)	
H13	1.201940	0.967962	0.166478	0.067*	
C14	1.0635 (3)	0.9110 (3)	0.1603 (2)	0.0470 (12)	
H14	1.093372	0.862346	0.133722	0.056*	
C15	0.9946 (3)	0.7058 (3)	0.2015 (2)	0.0304 (10)	
C16	0.9779 (3)	0.7327 (3)	0.2729 (2)	0.0324 (10)	
H16	0.949 (3)	0.789 (3)	0.280 (2)	0.039*	
C17	1.0235 (3)	0.6815 (3)	0.3327 (2)	0.0396 (11)	

H17	1.010442	0.702341	0.379273	0.048*
C18	1.0863 (3)	0.6022 (3)	0.3262 (2)	0.0452 (12)
H18	1.114697	0.566147	0.367734	0.054*
C19	1.1082 (3)	0.5749 (3)	0.2570 (2)	0.0439 (12)
H19	1.154394	0.520864	0.251457	0.053*
C20	1.0649 (3)	0.6238 (3)	0.1977 (2)	0.0384 (11)
H20	1.081931	0.602763	0.151591	0.046*
C21	0.9624 (3)	0.7374 (3)	0.0649 (2)	0.0338 (10)
C22	0.9685 (3)	0.6504 (3)	0.0400 (2)	0.0425 (11)
H22	0.955456	0.599280	0.072888	0.051*
C23	0.9933 (3)	0.6370 (4)	−0.0318 (2)	0.0520 (13)
H23	0.997370	0.577090	−0.046942	0.062*
C24	1.0121 (3)	0.7096 (4)	−0.0814 (2)	0.0549 (14)
H24	1.032169	0.699634	−0.129902	0.066*
C25	1.0014 (3)	0.7964 (4)	−0.0597 (2)	0.0524 (13)
H25	1.011378	0.847185	−0.093714	0.063*
C26	0.9761 (3)	0.8103 (3)	0.0120 (2)	0.0452 (12)
H26	0.967787	0.870994	0.025730	0.054*
C27	0.7084 (3)	0.6782 (3)	0.3265 (2)	0.0286 (9)
C28	0.6372 (3)	0.6713 (3)	0.26412 (19)	0.0265 (9)
C29	0.7125 (3)	0.7703 (3)	0.3467 (2)	0.0285 (9)
C30	0.6767 (3)	0.8468 (3)	0.2975 (2)	0.0315 (10)
H30	0.637 (3)	0.838 (3)	0.255 (2)	0.038*
C31	0.6847 (3)	0.9363 (3)	0.3123 (2)	0.0369 (10)
H31	0.659789	0.985192	0.277957	0.044*
C32	0.7273 (4)	0.9557 (3)	0.3750 (2)	0.0444 (12)
H32	0.733697	1.017022	0.384267	0.053*
C33	0.7612 (3)	0.8829 (3)	0.4252 (2)	0.0423 (11)
H33	0.789503	0.895256	0.469715	0.051*
C34	0.7546 (3)	0.7940 (3)	0.4120 (2)	0.0350 (10)
H34	0.779037	0.746424	0.447578	0.042*
C35	0.7355 (3)	0.5949 (3)	0.37686 (19)	0.0282 (9)
C36	0.8236 (3)	0.5817 (3)	0.4230 (2)	0.0366 (10)
H36	0.870475	0.628204	0.422239	0.044*
C37	0.8440 (3)	0.5036 (3)	0.4694 (2)	0.0414 (11)
H37	0.903875	0.497824	0.500098	0.050*
C38	0.7794 (4)	0.4336 (3)	0.4722 (2)	0.0486 (12)
H38	0.793232	0.380137	0.504559	0.058*
C39	0.6931 (4)	0.4439 (3)	0.4259 (2)	0.0427 (11)
H39	0.647778	0.396303	0.426318	0.051*
C40	0.6724 (3)	0.5220 (3)	0.3794 (2)	0.0337 (10)
H40	0.613326	0.526418	0.348145	0.040*
C41	0.6759 (3)	0.6084 (3)	0.2121 (2)	0.0287 (9)
C42	0.7841 (3)	0.5674 (3)	0.2129 (2)	0.0328 (10)
H42	0.829 (3)	0.574 (3)	0.252 (2)	0.039*
C43	0.8241 (3)	0.5133 (3)	0.1593 (2)	0.0385 (11)
H43	0.896566	0.488126	0.161064	0.046*
C44	0.7625 (3)	0.4951 (3)	0.1041 (2)	0.0457 (12)

H44	0.791310	0.458439	0.067595	0.055*
C45	0.6557 (3)	0.5322 (3)	0.1029 (2)	0.0410 (11)
H45	0.611433	0.519908	0.065066	0.049*
C46	0.6138 (3)	0.5853 (3)	0.1546 (2)	0.0331 (10)
H46	0.540514	0.607965	0.152372	0.040*
C47	0.5225 (3)	0.7051 (3)	0.2713 (2)	0.0288 (9)
C48	0.4585 (3)	0.7437 (3)	0.2139 (2)	0.0359 (10)
H48A	0.489123	0.750997	0.166683	0.043*
C49	0.3511 (3)	0.7718 (3)	0.2242 (3)	0.0435 (11)
H49A	0.309601	0.796881	0.183765	0.052*
C50	0.3036 (3)	0.7641 (3)	0.2913 (3)	0.0442 (12)
H50A	0.229768	0.782410	0.297646	0.053*
C51	0.3661 (3)	0.7290 (3)	0.3496 (2)	0.0435 (12)
H51A	0.335092	0.723847	0.396775	0.052*
C52	0.4744 (3)	0.7011 (3)	0.3398 (2)	0.0354 (10)
H52A	0.516297	0.678926	0.380728	0.043*
Na1	0.37921 (13)	0.25410 (12)	0.27731 (9)	0.0483 (5)
O1	0.4239 (2)	0.19929 (19)	0.42279 (14)	0.0400 (7)
O2	0.4224 (2)	0.38248 (19)	0.37589 (14)	0.0388 (7)
O3	0.3400 (2)	0.43418 (19)	0.23880 (14)	0.0394 (7)
O4	0.3337 (2)	0.3067 (2)	0.13954 (15)	0.0453 (8)
O5	0.3433 (2)	0.1224 (2)	0.18819 (15)	0.0433 (8)
O6	0.4299 (2)	0.07066 (19)	0.32220 (15)	0.0417 (7)
O7	0.1962 (2)	0.2560 (2)	0.31551 (17)	0.0585 (9)
O8	0.5614 (2)	0.2612 (2)	0.24862 (18)	0.0551 (9)
C53	0.4701 (4)	0.2602 (3)	0.4642 (2)	0.0448 (12)
H53A	0.546282	0.262147	0.450593	0.054*
H53B	0.465302	0.238997	0.516266	0.054*
C54	0.4114 (4)	0.3534 (3)	0.4502 (2)	0.0460 (12)
H54A	0.335076	0.351478	0.463488	0.055*
H54B	0.441328	0.396738	0.479770	0.055*
C55	0.3665 (4)	0.4693 (3)	0.3583 (2)	0.0445 (12)
H55A	0.392516	0.514507	0.387960	0.053*
H55B	0.289479	0.466699	0.368842	0.053*
C56	0.3835 (4)	0.4977 (3)	0.2804 (2)	0.0435 (11)
H56A	0.347860	0.560033	0.268092	0.052*
H56B	0.460668	0.498522	0.269519	0.052*
C57	0.3510 (4)	0.4584 (3)	0.1635 (2)	0.0502 (12)
H57A	0.427261	0.451027	0.148971	0.060*
H57B	0.323228	0.523179	0.151741	0.060*
C58	0.2906 (4)	0.3985 (3)	0.1234 (2)	0.0523 (13)
H58A	0.214250	0.405629	0.137938	0.063*
H58B	0.296182	0.415984	0.070785	0.063*
C59	0.2810 (4)	0.2441 (3)	0.1034 (2)	0.0505 (13)
H59A	0.275575	0.264961	0.051319	0.061*
H59B	0.207994	0.240489	0.123694	0.061*
C60	0.3429 (4)	0.1532 (3)	0.1133 (2)	0.0513 (13)
H60A	0.310103	0.109376	0.085728	0.062*

H60B	0.416864	0.157339	0.095091	0.062*	
C61	0.4060 (4)	0.0373 (3)	0.2027 (2)	0.0469 (12)	
H61A	0.482110	0.044654	0.191774	0.056*	
H61B	0.383213	−0.008139	0.171990	0.056*	
C62	0.3919 (4)	0.0054 (3)	0.2802 (2)	0.0464 (12)	
H62A	0.315570	−0.000438	0.291462	0.056*	
H62B	0.432491	−0.055234	0.291280	0.056*	
C63	0.4157 (5)	0.0465 (3)	0.3969 (3)	0.0643 (15)	
H63A	0.445926	−0.017498	0.409471	0.077*	
H63B	0.338745	0.051406	0.409644	0.077*	
C64	0.4707 (4)	0.1093 (3)	0.4382 (3)	0.0602 (15)	
H64A	0.464550	0.090930	0.490670	0.072*	
H64B	0.547343	0.105395	0.424568	0.072*	
C65	0.1082 (4)	0.3010 (4)	0.2762 (3)	0.0731 (16)	
H65A	0.080143	0.258884	0.244704	0.088*	
H65B	0.130110	0.354570	0.245442	0.088*	
C66	0.0240 (4)	0.3312 (5)	0.3301 (4)	0.094 (2)	
H66A	−0.001074	0.396778	0.319629	0.113*	
H66B	−0.037779	0.294934	0.329708	0.113*	
C67	0.0776 (5)	0.3146 (5)	0.3997 (3)	0.088 (2)	
H67A	0.027559	0.297417	0.439355	0.105*	
H67B	0.111697	0.368811	0.411203	0.105*	
C68	0.1589 (4)	0.2366 (4)	0.3863 (3)	0.0783 (18)	
H68A	0.217873	0.232294	0.420961	0.094*	
H68B	0.126180	0.178156	0.391196	0.094*	
C69	0.6222 (4)	0.3359 (4)	0.2549 (4)	0.0805 (18)	
H69A	0.599596	0.366787	0.298594	0.097*	
H69B	0.612072	0.381018	0.212065	0.097*	
C70	0.7360 (4)	0.2977 (5)	0.2602 (4)	0.090 (2)	
H70A	0.784719	0.343289	0.241559	0.108*	0.639 (12)
H70B	0.755033	0.274563	0.310458	0.108*	0.639 (12)
H70C	0.769194	0.293284	0.211644	0.108*	0.361 (12)
H70D	0.777192	0.335904	0.287787	0.108*	0.361 (12)
C71A	0.7365 (7)	0.2231 (7)	0.2133 (8)	0.109 (5)	0.639 (12)
H71A	0.789115	0.171475	0.230310	0.131*	0.639 (12)
H71B	0.755502	0.244575	0.162961	0.131*	0.639 (12)
C72A	0.6272 (8)	0.1936 (7)	0.2168 (8)	0.076 (4)	0.639 (12)
H72A	0.602463	0.186939	0.167664	0.091*	0.639 (12)
H72B	0.626401	0.134091	0.246171	0.091*	0.639 (12)
C71B	0.7305 (13)	0.2064 (8)	0.2988 (11)	0.087 (7)	0.361 (12)
H71C	0.725105	0.208369	0.351857	0.104*	0.361 (12)
H71D	0.791468	0.162456	0.286388	0.104*	0.361 (12)
C72B	0.6296 (14)	0.1867 (15)	0.2676 (14)	0.088 (7)	0.361 (12)
H72C	0.646660	0.153380	0.224373	0.105*	0.361 (12)
H72D	0.593368	0.145748	0.303356	0.105*	0.361 (12)
O9	0.3893 (4)	0.7876 (3)	−0.0311 (3)	0.1246 (18)	
C73	0.4175 (5)	0.6889 (4)	−0.0294 (4)	0.089 (2)	
H73A	0.397005	0.666681	−0.075196	0.106*	

H73B	0.495396	0.674202	−0.023630	0.106*
C74	0.3612 (4)	0.6457 (4)	0.0306 (3)	0.0615 (14)
H74A	0.403872	0.639506	0.075134	0.074*
H74B	0.343897	0.584413	0.020311	0.074*
C75	0.2627 (5)	0.7087 (4)	0.0382 (3)	0.0778 (18)
H75A	0.238927	0.708262	0.089407	0.093*
H75B	0.204388	0.691288	0.009326	0.093*
C76	0.2940 (5)	0.8011 (5)	0.0101 (4)	0.097 (2)
H76A	0.236902	0.834682	−0.020452	0.117*
H76B	0.305428	0.837329	0.050852	0.117*
O10	0.0414 (7)	0.8450 (6)	0.4779 (4)	0.227 (4)
C77	0.1301 (8)	0.8443 (7)	0.5225 (5)	0.178 (4)
H77A	0.160710	0.780949	0.537321	0.213*
H77B	0.110320	0.875877	0.566342	0.213*
C78	0.2055 (6)	0.8941 (7)	0.4760 (5)	0.125 (3)
H78A	0.257303	0.850963	0.452221	0.150*
H78B	0.245068	0.932398	0.504575	0.150*
C79	0.1375 (7)	0.9521 (7)	0.4214 (5)	0.163 (4)
H79A	0.117257	1.013508	0.437868	0.196*
H79B	0.176044	0.959342	0.374458	0.196*
C80	0.0422 (6)	0.9039 (6)	0.4145 (4)	0.122 (3)
H80A	−0.023166	0.947367	0.411209	0.147*
H80B	0.048045	0.869296	0.371283	0.147*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sc1	0.0240 (4)	0.0292 (5)	0.0282 (4)	0.0010 (3)	0.0009 (3)	−0.0016 (3)
C1	0.027 (2)	0.037 (3)	0.028 (2)	−0.0026 (19)	−0.0004 (18)	0.0013 (19)
C2	0.029 (2)	0.032 (2)	0.035 (2)	0.0009 (19)	0.0020 (19)	0.0003 (19)
C3	0.028 (2)	0.036 (3)	0.028 (2)	−0.0022 (19)	0.0058 (18)	0.0008 (19)
C4	0.027 (2)	0.035 (3)	0.027 (2)	0.002 (2)	0.0022 (18)	0.000 (2)
C5	0.037 (3)	0.047 (3)	0.024 (2)	−0.004 (2)	0.0008 (19)	0.000 (2)
C6	0.029 (2)	0.053 (3)	0.038 (3)	0.000 (2)	0.000 (2)	0.006 (2)
C7	0.031 (3)	0.038 (3)	0.047 (3)	0.009 (2)	−0.001 (2)	0.008 (2)
C8	0.038 (3)	0.032 (3)	0.040 (3)	0.000 (2)	0.005 (2)	0.002 (2)
C9	0.028 (2)	0.038 (3)	0.036 (2)	−0.001 (2)	−0.0027 (19)	0.005 (2)
C10	0.036 (3)	0.042 (3)	0.053 (3)	−0.007 (2)	0.000 (2)	−0.007 (2)
C11	0.047 (3)	0.046 (3)	0.064 (3)	−0.008 (2)	−0.006 (3)	−0.008 (3)
C12	0.039 (3)	0.048 (3)	0.085 (4)	−0.010 (2)	−0.023 (3)	0.009 (3)
C13	0.027 (3)	0.050 (3)	0.088 (4)	−0.010 (2)	−0.003 (3)	0.005 (3)
C14	0.030 (3)	0.047 (3)	0.062 (3)	0.001 (2)	0.003 (2)	0.002 (2)
C15	0.022 (2)	0.032 (2)	0.037 (2)	−0.0071 (18)	−0.0011 (18)	0.0030 (19)
C16	0.025 (2)	0.031 (3)	0.040 (3)	−0.0013 (19)	0.0007 (19)	0.001 (2)
C17	0.031 (2)	0.051 (3)	0.037 (3)	−0.010 (2)	0.000 (2)	0.001 (2)
C18	0.040 (3)	0.049 (3)	0.041 (3)	0.001 (2)	−0.006 (2)	0.015 (2)
C19	0.032 (3)	0.042 (3)	0.052 (3)	0.010 (2)	0.000 (2)	0.008 (2)
C20	0.028 (2)	0.043 (3)	0.042 (3)	0.003 (2)	0.002 (2)	0.002 (2)

C21	0.018 (2)	0.045 (3)	0.036 (2)	0.0041 (19)	0.0003 (18)	−0.001 (2)
C22	0.032 (2)	0.052 (3)	0.042 (3)	0.003 (2)	0.002 (2)	−0.007 (2)
C23	0.038 (3)	0.074 (4)	0.044 (3)	0.009 (3)	0.001 (2)	−0.019 (3)
C24	0.038 (3)	0.093 (4)	0.030 (3)	0.009 (3)	0.001 (2)	−0.007 (3)
C25	0.038 (3)	0.076 (4)	0.036 (3)	0.004 (3)	0.004 (2)	0.013 (3)
C26	0.037 (3)	0.050 (3)	0.045 (3)	0.008 (2)	0.000 (2)	0.004 (2)
C27	0.025 (2)	0.032 (2)	0.029 (2)	0.0001 (18)	0.0023 (17)	−0.0033 (19)
C28	0.028 (2)	0.029 (2)	0.023 (2)	−0.0043 (18)	0.0000 (17)	−0.0033 (18)
C29	0.025 (2)	0.034 (2)	0.028 (2)	−0.0030 (18)	0.0036 (17)	−0.0073 (19)
C30	0.027 (2)	0.033 (3)	0.034 (2)	−0.0008 (19)	0.0022 (19)	−0.006 (2)
C31	0.032 (2)	0.032 (3)	0.044 (3)	0.002 (2)	0.002 (2)	−0.002 (2)
C32	0.053 (3)	0.033 (3)	0.050 (3)	−0.013 (2)	0.008 (2)	−0.014 (2)
C33	0.042 (3)	0.055 (3)	0.034 (3)	−0.015 (2)	0.003 (2)	−0.014 (2)
C34	0.041 (3)	0.034 (3)	0.030 (2)	−0.007 (2)	−0.0014 (19)	−0.002 (2)
C35	0.029 (2)	0.032 (2)	0.023 (2)	−0.0017 (19)	0.0061 (18)	−0.0064 (18)
C36	0.028 (2)	0.046 (3)	0.035 (2)	−0.006 (2)	0.008 (2)	0.000 (2)
C37	0.034 (3)	0.050 (3)	0.036 (3)	0.002 (2)	−0.002 (2)	0.009 (2)
C38	0.056 (3)	0.043 (3)	0.041 (3)	0.005 (3)	0.005 (2)	0.011 (2)
C39	0.051 (3)	0.032 (3)	0.045 (3)	−0.011 (2)	0.003 (2)	0.002 (2)
C40	0.038 (3)	0.033 (3)	0.030 (2)	−0.004 (2)	−0.0016 (19)	−0.003 (2)
C41	0.032 (2)	0.025 (2)	0.030 (2)	−0.0061 (18)	0.0046 (18)	−0.0021 (18)
C42	0.034 (3)	0.033 (3)	0.032 (2)	−0.007 (2)	0.0013 (19)	−0.003 (2)
C43	0.035 (3)	0.036 (3)	0.044 (3)	0.004 (2)	0.004 (2)	−0.007 (2)
C44	0.043 (3)	0.048 (3)	0.048 (3)	−0.003 (2)	0.010 (2)	−0.019 (2)
C45	0.048 (3)	0.046 (3)	0.032 (3)	−0.014 (2)	0.000 (2)	−0.010 (2)
C46	0.032 (2)	0.035 (2)	0.034 (2)	−0.0084 (19)	0.0041 (19)	−0.008 (2)
C47	0.029 (2)	0.023 (2)	0.036 (2)	−0.0052 (18)	0.0028 (19)	−0.0098 (18)
C48	0.034 (3)	0.037 (3)	0.037 (3)	−0.006 (2)	0.000 (2)	−0.003 (2)
C49	0.032 (3)	0.040 (3)	0.057 (3)	0.003 (2)	−0.007 (2)	−0.007 (2)
C50	0.026 (2)	0.041 (3)	0.065 (3)	0.002 (2)	0.003 (2)	−0.010 (2)
C51	0.040 (3)	0.041 (3)	0.049 (3)	−0.003 (2)	0.017 (2)	−0.009 (2)
C52	0.031 (2)	0.038 (3)	0.037 (3)	−0.002 (2)	−0.001 (2)	−0.010 (2)
Na1	0.0311 (9)	0.0619 (12)	0.0506 (11)	−0.0024 (8)	0.0044 (8)	−0.0021 (9)
O1	0.0446 (18)	0.0394 (18)	0.0361 (17)	−0.0033 (15)	−0.0065 (14)	−0.0038 (14)
O2	0.0401 (17)	0.0414 (18)	0.0323 (17)	0.0046 (14)	0.0044 (13)	−0.0013 (14)
O3	0.0408 (17)	0.0394 (18)	0.0369 (17)	−0.0058 (14)	0.0004 (14)	0.0031 (14)
O4	0.0411 (18)	0.056 (2)	0.0389 (18)	−0.0041 (16)	−0.0055 (14)	−0.0060 (16)
O5	0.0420 (18)	0.054 (2)	0.0352 (18)	−0.0070 (16)	0.0031 (14)	−0.0109 (15)
O6	0.0457 (18)	0.0386 (18)	0.0424 (19)	−0.0086 (14)	−0.0067 (14)	−0.0049 (15)
O7	0.043 (2)	0.086 (3)	0.043 (2)	0.0010 (18)	0.0036 (16)	0.0025 (18)
O8	0.0341 (18)	0.050 (2)	0.083 (2)	−0.0024 (17)	0.0015 (17)	−0.0187 (19)
C53	0.050 (3)	0.048 (3)	0.038 (3)	−0.007 (2)	−0.010 (2)	−0.007 (2)
C54	0.057 (3)	0.047 (3)	0.034 (3)	−0.003 (2)	0.009 (2)	−0.009 (2)
C55	0.047 (3)	0.040 (3)	0.045 (3)	0.002 (2)	0.000 (2)	−0.003 (2)
C56	0.043 (3)	0.035 (3)	0.052 (3)	−0.005 (2)	−0.002 (2)	0.003 (2)
C57	0.062 (3)	0.048 (3)	0.040 (3)	−0.011 (3)	−0.002 (2)	0.007 (2)
C58	0.056 (3)	0.058 (3)	0.040 (3)	−0.003 (3)	−0.001 (2)	0.005 (2)
C59	0.045 (3)	0.071 (4)	0.037 (3)	−0.003 (3)	−0.004 (2)	−0.012 (3)

C60	0.056 (3)	0.072 (4)	0.030 (3)	−0.013 (3)	0.002 (2)	−0.018 (2)
C61	0.039 (3)	0.046 (3)	0.058 (3)	−0.006 (2)	0.001 (2)	−0.019 (2)
C62	0.046 (3)	0.039 (3)	0.057 (3)	−0.010 (2)	−0.004 (2)	−0.009 (2)
C63	0.100 (4)	0.046 (3)	0.045 (3)	−0.007 (3)	−0.012 (3)	0.003 (3)
C64	0.090 (4)	0.047 (3)	0.041 (3)	0.009 (3)	−0.027 (3)	0.001 (2)
C65	0.068 (4)	0.089 (4)	0.060 (4)	−0.005 (3)	−0.007 (3)	0.004 (3)
C66	0.048 (4)	0.115 (6)	0.108 (5)	0.017 (4)	−0.003 (4)	0.019 (4)
C67	0.075 (4)	0.108 (5)	0.083 (5)	−0.007 (4)	0.015 (4)	−0.033 (4)
C68	0.056 (4)	0.111 (5)	0.062 (4)	−0.002 (3)	−0.004 (3)	0.013 (3)
C69	0.055 (4)	0.069 (4)	0.121 (5)	−0.014 (3)	0.021 (3)	−0.029 (4)
C70	0.041 (3)	0.119 (6)	0.116 (5)	−0.016 (4)	−0.002 (3)	−0.035 (5)
C71A	0.042 (6)	0.081 (7)	0.206 (13)	0.010 (5)	0.026 (7)	−0.046 (8)
C72A	0.067 (7)	0.042 (6)	0.123 (11)	−0.019 (5)	0.033 (7)	−0.029 (7)
C71B	0.063 (10)	0.075 (12)	0.116 (14)	−0.004 (9)	−0.015 (10)	0.026 (10)
C72B	0.077 (12)	0.060 (11)	0.118 (15)	−0.005 (9)	0.027 (12)	0.020 (12)
O9	0.119 (4)	0.087 (4)	0.160 (5)	0.001 (3)	0.057 (3)	0.004 (3)
C73	0.072 (4)	0.081 (5)	0.118 (6)	−0.016 (4)	0.020 (4)	−0.033 (4)
C74	0.065 (4)	0.066 (4)	0.053 (3)	−0.014 (3)	−0.009 (3)	0.010 (3)
C75	0.065 (4)	0.095 (5)	0.078 (4)	−0.034 (4)	0.007 (3)	−0.010 (4)
C76	0.069 (4)	0.107 (6)	0.107 (5)	0.006 (4)	0.031 (4)	0.010 (4)
O10	0.274 (8)	0.255 (8)	0.164 (6)	−0.155 (7)	−0.102 (6)	0.081 (6)
C77	0.187 (9)	0.175 (9)	0.171 (9)	−0.060 (8)	−0.069 (8)	0.049 (7)
C78	0.103 (6)	0.162 (8)	0.118 (7)	−0.010 (6)	0.004 (6)	−0.057 (6)
C79	0.118 (7)	0.218 (10)	0.157 (8)	−0.074 (7)	0.006 (6)	0.021 (8)
C80	0.140 (7)	0.122 (7)	0.107 (6)	−0.038 (6)	−0.017 (5)	0.009 (5)

Geometric parameters (Å, º)

Sc1—C1	2.351 (4)	Na1—O8	2.355 (3)
Sc1—C2	2.357 (4)	Na1—O7	2.394 (3)
Sc1—C3	2.577 (4)	Na1—O4	2.663 (3)
Sc1—C4	2.518 (4)	Na1—O3	2.685 (3)
Sc1—C15	2.559 (4)	Na1—O5	2.751 (3)
Sc1—C16	2.536 (4)	Na1—O6	2.770 (3)
Sc1—C27	2.354 (4)	Na1—O1	2.801 (3)
Sc1—C28	2.380 (4)	Na1—O2	2.860 (3)
Sc1—C29	2.590 (4)	O1—C64	1.404 (5)
Sc1—C30	2.563 (4)	O1—C53	1.416 (5)
Sc1—C41	2.562 (4)	O2—C55	1.411 (5)
Sc1—C42	2.560 (4)	O2—C54	1.415 (5)
Sc1—H4	2.38 (4)	O3—C57	1.418 (5)
C1—C3	1.434 (5)	O3—C56	1.431 (5)
C3—C4	1.418 (6)	O4—C58	1.414 (5)
C4—C5	1.393 (5)	O4—C59	1.420 (5)
C5—C6	1.370 (6)	O5—C61	1.420 (5)
C6—C7	1.387 (6)	O5—C60	1.423 (5)
C7—C8	1.369 (6)	O6—C63	1.412 (5)
C3—C8	1.432 (5)	O6—C62	1.424 (5)

C1—C9	1.482 (6)	O7—C68	1.399 (6)
C1—C2	1.501 (5)	O7—C65	1.415 (6)
C2—C15	1.442 (5)	O8—C72B	1.35 (2)
C15—C16	1.425 (6)	O8—C72A	1.397 (10)
C16—C17	1.385 (6)	O8—C69	1.422 (6)
C17—C18	1.357 (6)	C53—C54	1.496 (6)
C18—C19	1.397 (6)	C53—H53A	0.9900
C19—C20	1.353 (5)	C53—H53B	0.9900
C15—C20	1.431 (5)	C54—H54A	0.9900
C2—C21	1.486 (5)	C54—H54B	0.9900
C27—C29	1.451 (5)	C55—C56	1.485 (6)
C29—C30	1.426 (5)	C55—H55A	0.9900
C30—C31	1.391 (6)	C55—H55B	0.9900
C31—C32	1.362 (6)	C56—H56A	0.9900
C32—C33	1.393 (6)	C56—H56B	0.9900
C33—C34	1.370 (6)	C57—C58	1.487 (6)
C29—C34	1.424 (5)	C57—H57A	0.9900
C27—C35	1.488 (5)	C57—H57B	0.9900
C27—C28	1.503 (5)	C58—H58A	0.9900
C28—C41	1.444 (5)	C58—H58B	0.9900
C41—C42	1.434 (5)	C59—C60	1.479 (6)
C42—C43	1.388 (5)	C59—H59A	0.9900
C43—C44	1.365 (6)	C59—H59B	0.9900
C44—C45	1.400 (6)	C60—H60A	0.9900
C45—C46	1.360 (5)	C60—H60B	0.9900
C41—C46	1.428 (5)	C61—C62	1.484 (6)
C28—C47	1.487 (5)	C61—H61A	0.9900
C4—H4	0.93 (4)	C61—H61B	0.9900
C5—H5	0.9500	C62—H62A	0.9900
C6—H6	0.9500	C62—H62B	0.9900
C7—H7	0.9500	C63—C64	1.491 (6)
C8—H8	0.9500	C63—H63A	0.9900
C9—C10	1.406 (6)	C63—H63B	0.9900
C9—C14	1.409 (5)	C64—H64A	0.9900
C10—C11	1.393 (6)	C64—H64B	0.9900
C10—H10	0.9500	C65—C66	1.504 (7)
C11—C12	1.372 (6)	C65—H65A	0.9900
C11—H11	0.9500	C65—H65B	0.9900
C12—C13	1.370 (7)	C66—C67	1.462 (8)
C12—H12	0.9500	C66—H66A	0.9900
C13—C14	1.382 (6)	C66—H66B	0.9900
C13—H13	0.9500	C67—C68	1.494 (8)
C14—H14	0.9500	C67—H67A	0.9900
C16—H16	0.89 (4)	C67—H67B	0.9900
C17—H17	0.9500	C68—H68A	0.9900
C18—H18	0.9500	C68—H68B	0.9900
C19—H19	0.9500	C69—C70	1.488 (7)
C20—H20	0.9500	C69—H69A	0.9900

C21—C22	1.401 (6)	C69—H69B	0.9900
C21—C26	1.407 (6)	C70—C71A	1.469 (11)
C22—C23	1.389 (6)	C70—C71B	1.473 (3)
C22—H22	0.9500	C70—H70A	0.9900
C23—C24	1.382 (7)	C70—H70B	0.9900
C23—H23	0.9500	C70—H70C	0.9900
C24—C25	1.372 (7)	C70—H70D	0.9900
C24—H24	0.9500	C71A—C72A	1.485 (12)
C25—C26	1.391 (6)	C71A—H71A	0.9900
C25—H25	0.9500	C71A—H71B	0.9900
C26—H26	0.9500	C72A—H72A	0.9900
C30—H30	0.97 (4)	C72A—H72B	0.9900
C31—H31	0.9500	C71B—C72B	1.478 (3)
C32—H32	0.9500	C71B—H71C	0.9900
C33—H33	0.9500	C71B—H71D	0.9900
C34—H34	0.9500	C72B—H72C	0.9900
C35—C40	1.401 (5)	C72B—H72D	0.9900
C35—C36	1.407 (5)	O9—C76	1.414 (7)
C36—C37	1.379 (6)	O9—C73	1.461 (7)
C36—H36	0.9500	C73—C74	1.440 (7)
C37—C38	1.377 (6)	C73—H73A	0.9900
C37—H37	0.9500	C73—H73B	0.9900
C38—C39	1.390 (6)	C74—C75	1.484 (7)
C38—H38	0.9500	C74—H74A	0.9900
C39—C40	1.379 (5)	C74—H74B	0.9900
C39—H39	0.9500	C75—C76	1.501 (8)
C40—H40	0.9500	C75—H75A	0.9900
C42—H42	0.95 (4)	C75—H75B	0.9900
C43—H43	0.9500	C76—H76A	0.9900
C44—H44	0.9500	C76—H76B	0.9900
C45—H45	0.9500	O10—C80	1.397 (8)
C46—H46	0.9500	O10—C77	1.408 (10)
C47—C52	1.395 (5)	C77—C78	1.475 (3)
C47—C48	1.395 (5)	C77—H77A	0.9900
C48—C49	1.388 (5)	C77—H77B	0.9900
C48—H48A	0.9500	C78—C79	1.491 (10)
C49—C50	1.369 (6)	C78—H78A	0.9900
C49—H49A	0.9500	C78—H78B	0.9900
C50—C51	1.385 (6)	C79—C80	1.473 (3)
C50—H50A	0.9500	C79—H79A	0.9900
C51—C52	1.397 (5)	C79—H79B	0.9900
C51—H51A	0.9500	C80—H80A	0.9900
C52—H52A	0.9500	C80—H80B	0.9900

C1—Sc1—C27	152.76 (14)	C49—C48—H48A	119.3
C1—Sc1—C2	37.20 (13)	C47—C48—H48A	119.3
C27—Sc1—C2	154.47 (13)	C50—C49—C48	121.5 (4)
C1—Sc1—C28	154.60 (13)	C50—C49—H49A	119.2

C27—Sc1—C28	37.02 (12)	C48—C49—H49A	119.2
C2—Sc1—C28	154.15 (14)	C49—C50—C51	118.3 (4)
C1—Sc1—C4	61.16 (13)	C49—C50—H50A	120.9
C27—Sc1—C4	132.87 (13)	C51—C50—H50A	120.9
C2—Sc1—C4	71.13 (13)	C50—C51—C52	120.6 (4)
C28—Sc1—C4	97.91 (13)	C50—C51—H51A	119.7
C1—Sc1—C16	71.33 (13)	C52—C51—H51A	119.7
C27—Sc1—C16	96.97 (13)	C47—C52—C51	121.6 (4)
C2—Sc1—C16	61.19 (13)	C47—C52—H52A	119.2
C28—Sc1—C16	132.00 (13)	C51—C52—H52A	119.2
C4—Sc1—C16	129.81 (13)	O8—Na1—O7	174.52 (14)
C1—Sc1—C15	60.94 (13)	O8—Na1—O4	89.11 (12)
C27—Sc1—C15	120.87 (13)	O7—Na1—O4	94.23 (11)
C2—Sc1—C15	33.81 (12)	O8—Na1—O3	90.22 (11)
C28—Sc1—C15	143.51 (13)	O7—Na1—O3	87.50 (11)
C4—Sc1—C15	104.60 (13)	O4—Na1—O3	62.31 (10)
C16—Sc1—C15	32.49 (12)	O8—Na1—O5	97.47 (11)
C1—Sc1—C42	133.49 (13)	O7—Na1—O5	87.95 (11)
C27—Sc1—C42	71.57 (13)	O4—Na1—O5	61.66 (10)
C2—Sc1—C42	97.59 (13)	O3—Na1—O5	123.20 (11)
C28—Sc1—C42	60.86 (13)	O8—Na1—O6	87.50 (11)
C4—Sc1—C42	101.65 (14)	O7—Na1—O6	94.54 (12)
C16—Sc1—C42	99.28 (14)	O4—Na1—O6	120.31 (11)
C15—Sc1—C42	86.41 (13)	O3—Na1—O6	176.47 (11)
C1—Sc1—C41	144.86 (13)	O5—Na1—O6	59.83 (9)
C27—Sc1—C41	61.27 (13)	O8—Na1—O1	91.72 (11)
C2—Sc1—C41	120.73 (13)	O7—Na1—O1	84.92 (10)
C28—Sc1—C41	33.73 (12)	O4—Na1—O1	179.14 (11)
C4—Sc1—C41	87.68 (13)	O3—Na1—O1	117.47 (10)
C16—Sc1—C41	129.24 (13)	O5—Na1—O1	118.41 (10)
C15—Sc1—C41	118.44 (12)	O6—Na1—O1	59.95 (9)
C42—Sc1—C41	32.50 (12)	O8—Na1—O2	81.84 (11)
C1—Sc1—C30	97.59 (13)	O7—Na1—O2	92.70 (11)
C27—Sc1—C30	60.60 (13)	O4—Na1—O2	121.62 (11)
C2—Sc1—C30	133.92 (14)	O3—Na1—O2	60.20 (9)
C28—Sc1—C30	69.38 (13)	O5—Na1—O2	176.57 (11)
C4—Sc1—C30	97.36 (14)	O6—Na1—O2	116.75 (10)
C16—Sc1—C30	104.20 (14)	O1—Na1—O2	58.33 (9)
C15—Sc1—C30	133.68 (13)	C64—O1—C53	111.0 (3)
C42—Sc1—C30	128.47 (13)	C64—O1—Na1	116.2 (2)
C41—Sc1—C30	102.50 (13)	C53—O1—Na1	118.8 (2)
C1—Sc1—C3	33.44 (12)	C55—O2—C54	111.5 (3)
C27—Sc1—C3	143.97 (13)	C55—O2—Na1	112.0 (2)
C2—Sc1—C3	60.15 (13)	C54—O2—Na1	116.1 (2)
C28—Sc1—C3	121.63 (13)	C57—O3—C56	111.8 (3)
C4—Sc1—C3	32.30 (13)	C57—O3—Na1	113.8 (2)
C16—Sc1—C3	104.56 (13)	C56—O3—Na1	119.4 (2)
C15—Sc1—C3	91.35 (12)	C58—O4—C59	112.8 (3)

C42—Sc1—C3	130.93 (13)	C58—O4—Na1	116.7 (2)
C41—Sc1—C3	119.69 (13)	C59—O4—Na1	115.3 (3)
C30—Sc1—C3	86.00 (13)	C61—O5—C60	112.1 (3)
C1—Sc1—C29	119.66 (13)	C61—O5—Na1	115.0 (2)
C27—Sc1—C29	33.70 (12)	C60—O5—Na1	114.3 (3)
C2—Sc1—C29	145.17 (13)	C63—O6—C62	111.5 (3)
C28—Sc1—C29	59.91 (12)	C63—O6—Na1	115.2 (3)
C4—Sc1—C29	127.51 (13)	C62—O6—Na1	117.6 (2)
C16—Sc1—C29	88.99 (13)	C68—O7—C65	106.2 (4)
C15—Sc1—C29	121.22 (12)	C68—O7—Na1	126.1 (3)
C42—Sc1—C29	105.15 (13)	C65—O7—Na1	125.4 (3)
C41—Sc1—C29	91.50 (12)	C72B—O8—C69	104.0 (8)
C30—Sc1—C29	32.12 (12)	C72A—O8—C69	107.6 (5)
C3—Sc1—C29	117.37 (13)	C72B—O8—Na1	117.9 (8)
C1—Sc1—H4	69.3 (9)	C72A—O8—Na1	125.0 (4)
C27—Sc1—H4	133.5 (9)	C69—O8—Na1	127.3 (3)
C2—Sc1—H4	64.4 (9)	O1—C53—C54	108.8 (3)
C28—Sc1—H4	96.7 (9)	O1—C53—H53A	109.9
C4—Sc1—H4	21.6 (9)	C54—C53—H53A	109.9
C16—Sc1—H4	125.3 (9)	O1—C53—H53B	109.9
C15—Sc1—H4	94.2 (9)	C54—C53—H53B	109.9
C42—Sc1—H4	82.2 (9)	H53A—C53—H53B	108.3
C41—Sc1—H4	75.9 (9)	O2—C54—C53	108.4 (3)
C30—Sc1—H4	117.3 (10)	O2—C54—H54A	110.0
C3—Sc1—H4	49.0 (9)	C53—C54—H54A	110.0
C29—Sc1—H4	143.9 (9)	O2—C54—H54B	110.0
C3—C1—C9	123.3 (4)	C53—C54—H54B	110.0
C3—C1—C2	115.3 (3)	H54A—C54—H54B	108.4
C9—C1—C2	118.2 (3)	O2—C55—C56	109.3 (3)
C3—C1—Sc1	82.0 (2)	O2—C55—H55A	109.8
C9—C1—Sc1	131.8 (3)	C56—C55—H55A	109.8
C2—C1—Sc1	71.6 (2)	O2—C55—H55B	109.8
C15—C2—C21	122.9 (3)	C56—C55—H55B	109.8
C15—C2—C1	115.9 (3)	H55A—C55—H55B	108.3
C21—C2—C1	117.8 (3)	O3—C56—C55	108.7 (3)
C15—C2—Sc1	80.8 (2)	O3—C56—H56A	109.9
C21—C2—Sc1	134.2 (3)	C55—C56—H56A	109.9
C1—C2—Sc1	71.2 (2)	O3—C56—H56B	109.9
C4—C3—C8	114.5 (4)	C55—C56—H56B	109.9
C4—C3—C1	120.8 (4)	H56A—C56—H56B	108.3
C8—C3—C1	124.6 (4)	O3—C57—C58	109.3 (4)
C4—C3—Sc1	71.6 (2)	O3—C57—H57A	109.8
C8—C3—Sc1	137.2 (3)	C58—C57—H57A	109.8
C1—C3—Sc1	64.6 (2)	O3—C57—H57B	109.8
C5—C4—C3	122.4 (4)	C58—C57—H57B	109.8
C5—C4—Sc1	131.7 (3)	H57A—C57—H57B	108.3
C3—C4—Sc1	76.1 (2)	O4—C58—C57	108.7 (4)
C5—C4—H4	115 (2)	O4—C58—H58A	110.0

C3—C4—H4	122 (2)	C57—C58—H58A	110.0
Sc1—C4—H4	71 (2)	O4—C58—H58B	110.0
C6—C5—C4	121.1 (4)	C57—C58—H58B	110.0
C6—C5—H5	119.5	H58A—C58—H58B	108.3
C4—C5—H5	119.5	O4—C59—C60	108.7 (4)
C5—C6—C7	118.1 (4)	O4—C59—H59A	110.0
C5—C6—H6	120.9	C60—C59—H59A	110.0
C7—C6—H6	120.9	O4—C59—H59B	110.0
C8—C7—C6	122.1 (4)	C60—C59—H59B	110.0
C8—C7—H7	118.9	H59A—C59—H59B	108.3
C6—C7—H7	118.9	O5—C60—C59	108.6 (4)
C7—C8—C3	121.7 (4)	O5—C60—H60A	110.0
C7—C8—H8	119.1	C59—C60—H60A	110.0
C3—C8—H8	119.1	O5—C60—H60B	110.0
C10—C9—C14	115.6 (4)	C59—C60—H60B	110.0
C10—C9—C1	124.3 (4)	H60A—C60—H60B	108.3
C14—C9—C1	120.0 (4)	O5—C61—C62	108.4 (4)
C11—C10—C9	121.8 (4)	O5—C61—H61A	110.0
C11—C10—H10	119.1	C62—C61—H61A	110.0
C9—C10—H10	119.1	O5—C61—H61B	110.0
C12—C11—C10	120.6 (5)	C62—C61—H61B	110.0
C12—C11—H11	119.7	H61A—C61—H61B	108.4
C10—C11—H11	119.7	O6—C62—C61	108.1 (4)
C13—C12—C11	119.1 (5)	O6—C62—H62A	110.1
C13—C12—H12	120.5	C61—C62—H62A	110.1
C11—C12—H12	120.5	O6—C62—H62B	110.1
C12—C13—C14	121.1 (4)	C61—C62—H62B	110.1
C12—C13—H13	119.5	H62A—C62—H62B	108.4
C14—C13—H13	119.5	O6—C63—C64	109.1 (4)
C13—C14—C9	121.8 (4)	O6—C63—H63A	109.9
C13—C14—H14	119.1	C64—C63—H63A	109.9
C9—C14—H14	119.1	O6—C63—H63B	109.9
C16—C15—C20	113.8 (4)	C64—C63—H63B	109.9
C16—C15—C2	120.9 (4)	H63A—C63—H63B	108.3
C20—C15—C2	125.1 (4)	O1—C64—C63	109.5 (4)
C16—C15—Sc1	72.9 (2)	O1—C64—H64A	109.8
C20—C15—Sc1	133.7 (3)	C63—C64—H64A	109.8
C2—C15—Sc1	65.4 (2)	O1—C64—H64B	109.8
C17—C16—C15	122.3 (4)	C63—C64—H64B	109.8
C17—C16—Sc1	130.7 (3)	H64A—C64—H64B	108.2
C15—C16—Sc1	74.6 (2)	O7—C65—C66	107.5 (4)
C17—C16—H16	116 (3)	O7—C65—H65A	110.2
C15—C16—H16	121 (3)	C66—C65—H65A	110.2
Sc1—C16—H16	77 (3)	O7—C65—H65B	110.2
C18—C17—C16	121.3 (4)	C66—C65—H65B	110.2
C18—C17—H17	119.3	H65A—C65—H65B	108.5
C16—C17—H17	119.3	C67—C66—C65	104.2 (5)
C17—C18—C19	118.4 (4)	C67—C66—H66A	110.9

C17—C18—H18	120.8	C65—C66—H66A	110.9
C19—C18—H18	120.8	C67—C66—H66B	110.9
C20—C19—C18	121.4 (4)	C65—C66—H66B	110.9
C20—C19—H19	119.3	H66A—C66—H66B	108.9
C18—C19—H19	119.3	C66—C67—C68	101.6 (5)
C19—C20—C15	122.6 (4)	C66—C67—H67A	111.5
C19—C20—H20	118.7	C68—C67—H67A	111.5
C15—C20—H20	118.7	C66—C67—H67B	111.5
C22—C21—C26	115.9 (4)	C68—C67—H67B	111.5
C22—C21—C2	124.6 (4)	H67A—C67—H67B	109.3
C26—C21—C2	119.5 (4)	O7—C68—C67	105.4 (5)
C23—C22—C21	121.5 (4)	O7—C68—H68A	110.7
C23—C22—H22	119.2	C67—C68—H68A	110.7
C21—C22—H22	119.2	O7—C68—H68B	110.7
C24—C23—C22	121.0 (5)	C67—C68—H68B	110.7
C24—C23—H23	119.5	H68A—C68—H68B	108.8
C22—C23—H23	119.5	O8—C69—C70	107.0 (5)
C25—C24—C23	119.0 (4)	O8—C69—H69A	110.3
C25—C24—H24	120.5	C70—C69—H69A	110.3
C23—C24—H24	120.5	O8—C69—H69B	110.3
C24—C25—C26	120.3 (5)	C70—C69—H69B	110.3
C24—C25—H25	119.9	H69A—C69—H69B	108.6
C26—C25—H25	119.9	C71A—C70—C69	100.1 (5)
C25—C26—C21	122.2 (4)	C71B—C70—C69	103.4 (8)
C25—C26—H26	118.9	C71A—C70—H70A	111.8
C21—C26—H26	118.9	C69—C70—H70A	111.8
C29—C27—C35	123.1 (3)	C71A—C70—H70B	111.8
C29—C27—C28	114.8 (3)	C69—C70—H70B	111.8
C35—C27—C28	118.2 (3)	H70A—C70—H70B	109.5
C29—C27—Sc1	82.1 (2)	C71B—C70—H70C	111.1
C35—C27—Sc1	132.2 (2)	C69—C70—H70C	111.1
C28—C27—Sc1	72.4 (2)	C71B—C70—H70D	111.1
C41—C28—C47	122.4 (3)	C69—C70—H70D	111.1
C41—C28—C27	116.9 (3)	H70C—C70—H70D	109.0
C47—C28—C27	117.9 (3)	C70—C71A—C72A	106.8 (7)
C41—C28—Sc1	80.1 (2)	C70—C71A—H71A	110.4
C47—C28—Sc1	134.7 (3)	C72A—C71A—H71A	110.4
C27—C28—Sc1	70.5 (2)	C70—C71A—H71B	110.4
C34—C29—C30	114.3 (4)	C72A—C71A—H71B	110.4
C34—C29—C27	125.9 (4)	H71A—C71A—H71B	108.6
C30—C29—C27	119.7 (3)	O8—C72A—C71A	106.9 (7)
C34—C29—Sc1	134.5 (3)	O8—C72A—H72A	110.3
C30—C29—Sc1	72.9 (2)	C71A—C72A—H72A	110.3
C27—C29—Sc1	64.2 (2)	O8—C72A—H72B	110.3
C31—C30—C29	122.1 (4)	C71A—C72A—H72B	110.3
C31—C30—Sc1	132.1 (3)	H72A—C72A—H72B	108.6
C29—C30—Sc1	75.0 (2)	C70—C71B—C72B	97.3 (11)
C31—C30—H30	117 (2)	C70—C71B—H71C	112.3

C29—C30—H30	120 (2)	C72B—C71B—H71C	112.3
Sc1—C30—H30	71 (2)	C70—C71B—H71D	112.3
C32—C31—C30	121.6 (4)	C72B—C71B—H71D	112.3
C32—C31—H31	119.2	H71C—C71B—H71D	109.9
C30—C31—H31	119.2	O8—C72B—C71B	114.7 (15)
C31—C32—C33	118.1 (4)	O8—C72B—H72C	108.6
C31—C32—H32	120.9	C71B—C72B—H72C	108.6
C33—C32—H32	120.9	O8—C72B—H72D	108.6
C34—C33—C32	121.6 (4)	C71B—C72B—H72D	108.6
C34—C33—H33	119.2	H72C—C72B—H72D	107.6
C32—C33—H33	119.2	C76—O9—C73	107.0 (5)
C33—C34—C29	122.3 (4)	C74—C73—O9	107.9 (5)
C33—C34—H34	118.8	C74—C73—H73A	110.1
C29—C34—H34	118.8	O9—C73—H73A	110.1
C40—C35—C36	115.4 (4)	C74—C73—H73B	110.1
C40—C35—C27	120.0 (3)	O9—C73—H73B	110.1
C36—C35—C27	124.6 (4)	H73A—C73—H73B	108.4
C37—C36—C35	122.1 (4)	C73—C74—C75	103.8 (5)
C37—C36—H36	118.9	C73—C74—H74A	111.0
C35—C36—H36	118.9	C75—C74—H74A	111.0
C38—C37—C36	121.4 (4)	C73—C74—H74B	111.0
C38—C37—H37	119.3	C75—C74—H74B	111.0
C36—C37—H37	119.3	H74A—C74—H74B	109.0
C37—C38—C39	117.8 (4)	C74—C75—C76	104.5 (4)
C37—C38—H38	121.1	C74—C75—H75A	110.9
C39—C38—H38	121.1	C76—C75—H75A	110.9
C40—C39—C38	121.1 (4)	C74—C75—H75B	110.9
C40—C39—H39	119.5	C76—C75—H75B	110.9
C38—C39—H39	119.5	H75A—C75—H75B	108.9
C39—C40—C35	122.2 (4)	O9—C76—C75	107.9 (5)
C39—C40—H40	118.9	O9—C76—H76A	110.1
C35—C40—H40	118.9	C75—C76—H76A	110.1
C46—C41—C42	114.6 (3)	O9—C76—H76B	110.1
C46—C41—C28	124.2 (3)	C75—C76—H76B	110.1
C42—C41—C28	121.2 (4)	H76A—C76—H76B	108.4
C46—C41—Sc1	131.8 (3)	C80—O10—C77	115.2 (6)
C42—C41—Sc1	73.7 (2)	O10—C77—C78	103.3 (7)
C28—C41—Sc1	66.2 (2)	O10—C77—H77A	111.1
C43—C42—C41	121.3 (4)	C78—C77—H77A	111.1
C43—C42—Sc1	129.1 (3)	O10—C77—H77B	111.1
C41—C42—Sc1	73.8 (2)	C78—C77—H77B	111.1
C43—C42—H42	119 (2)	H77A—C77—H77B	109.1
C41—C42—H42	120 (2)	C77—C78—C79	104.8 (7)
Sc1—C42—H42	72 (2)	C77—C78—H78A	110.8
C44—C43—C42	122.0 (4)	C79—C78—H78A	110.8
C44—C43—H43	119.0	C77—C78—H78B	110.8
C42—C43—H43	119.0	C79—C78—H78B	110.8
C43—C44—C45	118.1 (4)	H78A—C78—H78B	108.9

C43—C44—H44	121.0	C80—C79—C78	105.8 (7)
C45—C44—H44	121.0	C80—C79—H79A	110.6
C46—C45—C44	121.4 (4)	C78—C79—H79A	110.6
C46—C45—H45	119.3	C80—C79—H79B	110.6
C44—C45—H45	119.3	C78—C79—H79B	110.6
C45—C46—C41	122.5 (4)	H79A—C79—H79B	108.7
C45—C46—H46	118.7	O10—C80—C79	103.6 (7)
C41—C46—H46	118.7	O10—C80—H80A	111.0
C52—C47—C48	116.5 (4)	C79—C80—H80A	111.0
C52—C47—C28	119.0 (3)	O10—C80—H80B	111.0
C48—C47—C28	124.5 (3)	C79—C80—H80B	111.0
C49—C48—C47	121.5 (4)	H80A—C80—H80B	109.0

C3—C1—C2—C15	−140.6 (4)	C28—C27—C35—C40	−19.1 (5)
C9—C1—C2—C15	58.9 (5)	Sc1—C27—C35—C40	−110.5 (4)
Sc1—C1—C2—C15	−69.3 (3)	C29—C27—C35—C36	−43.5 (5)
C3—C1—C2—C21	59.3 (5)	C28—C27—C35—C36	159.7 (4)
C9—C1—C2—C21	−101.1 (4)	Sc1—C27—C35—C36	68.3 (5)
Sc1—C1—C2—C21	130.7 (3)	C40—C35—C36—C37	−2.2 (6)
C3—C1—C2—Sc1	−71.4 (3)	C27—C35—C36—C37	179.0 (4)
C9—C1—C2—Sc1	128.2 (3)	C35—C36—C37—C38	0.8 (6)
C9—C1—C3—C4	178.3 (4)	C36—C37—C38—C39	0.7 (6)
C2—C1—C3—C4	19.0 (5)	C37—C38—C39—C40	−0.7 (6)
Sc1—C1—C3—C4	−46.2 (3)	C38—C39—C40—C35	−0.8 (6)
C9—C1—C3—C8	−4.2 (6)	C36—C35—C40—C39	2.1 (6)
C2—C1—C3—C8	−163.5 (4)	C27—C35—C40—C39	−179.0 (4)
Sc1—C1—C3—C8	131.2 (4)	C47—C28—C41—C46	11.7 (6)
C9—C1—C3—Sc1	−135.4 (4)	C27—C28—C41—C46	172.3 (3)
C2—C1—C3—Sc1	65.3 (3)	Sc1—C28—C41—C46	−125.6 (4)
C8—C3—C4—C5	−3.3 (5)	C47—C28—C41—C42	−171.5 (4)
C1—C3—C4—C5	174.4 (4)	C27—C28—C41—C42	−10.9 (5)
Sc1—C3—C4—C5	130.9 (4)	Sc1—C28—C41—C42	51.2 (3)
C8—C3—C4—Sc1	−134.3 (3)	C47—C28—C41—Sc1	137.3 (4)
C1—C3—C4—Sc1	43.4 (3)	C27—C28—C41—Sc1	−62.1 (3)
C3—C4—C5—C6	1.2 (6)	C46—C41—C42—C43	2.8 (6)
Sc1—C4—C5—C6	101.9 (5)	C28—C41—C42—C43	−174.3 (4)
C4—C5—C6—C7	1.9 (6)	Sc1—C41—C42—C43	−126.3 (4)
C5—C6—C7—C8	−2.8 (6)	C46—C41—C42—Sc1	129.1 (3)
C6—C7—C8—C3	0.5 (6)	C28—C41—C42—Sc1	−48.0 (3)
C4—C3—C8—C7	2.5 (6)	C41—C42—C43—C44	−1.1 (6)
C1—C3—C8—C7	−175.1 (4)	Sc1—C42—C43—C44	−95.4 (5)
Sc1—C3—C8—C7	−86.4 (5)	C42—C43—C44—C45	−0.6 (7)
C3—C1—C9—C10	48.5 (6)	C43—C44—C45—C46	0.5 (7)
C2—C1—C9—C10	−152.8 (4)	C44—C45—C46—C41	1.4 (6)
Sc1—C1—C9—C10	−62.8 (5)	C42—C41—C46—C45	−2.9 (6)
C3—C1—C9—C14	−134.3 (4)	C28—C41—C46—C45	174.0 (4)
C2—C1—C9—C14	24.5 (5)	Sc1—C41—C46—C45	86.7 (5)
Sc1—C1—C9—C14	114.5 (4)	C41—C28—C47—C52	130.7 (4)

C14—C9—C10—C11	0.9 (6)	C27—C28—C47—C52	−29.7 (5)
C1—C9—C10—C11	178.3 (4)	Sc1—C28—C47—C52	−119.4 (4)
C9—C10—C11—C12	−0.1 (7)	C41—C28—C47—C48	−51.3 (6)
C10—C11—C12—C13	0.5 (7)	C27—C28—C47—C48	148.3 (4)
C11—C12—C13—C14	−1.8 (7)	Sc1—C28—C47—C48	58.6 (5)
C12—C13—C14—C9	2.8 (7)	C52—C47—C48—C49	−3.6 (6)
C10—C9—C14—C13	−2.3 (6)	C28—C47—C48—C49	178.3 (4)
C1—C9—C14—C13	−179.8 (4)	C47—C48—C49—C50	1.1 (6)
C21—C2—C15—C16	173.3 (4)	C48—C49—C50—C51	1.2 (6)
C1—C2—C15—C16	14.4 (5)	C49—C50—C51—C52	−0.8 (6)
Sc1—C2—C15—C16	−49.3 (3)	C48—C47—C52—C51	4.0 (6)
C21—C2—C15—C20	−10.0 (6)	C28—C47—C52—C51	−177.8 (4)
C1—C2—C15—C20	−168.9 (4)	C50—C51—C52—C47	−1.9 (6)
Sc1—C2—C15—C20	127.4 (4)	C64—O1—C53—C54	−175.4 (4)
C21—C2—C15—Sc1	−137.4 (4)	Na1—O1—C53—C54	45.9 (4)
C1—C2—C15—Sc1	63.7 (3)	C55—O2—C54—C53	178.5 (4)
C20—C15—C16—C17	−2.1 (6)	Na1—O2—C54—C53	48.6 (4)
C2—C15—C16—C17	175.0 (4)	O1—C53—C54—O2	−61.2 (5)
Sc1—C15—C16—C17	128.8 (4)	C54—O2—C55—C56	178.2 (4)
C20—C15—C16—Sc1	−130.9 (3)	Na1—O2—C55—C56	−49.8 (4)
C2—C15—C16—Sc1	46.2 (3)	C57—O3—C56—C55	178.5 (3)
C15—C16—C17—C18	−0.4 (6)	Na1—O3—C56—C55	−45.0 (4)
Sc1—C16—C17—C18	97.1 (5)	O2—C55—C56—O3	62.7 (4)
C16—C17—C18—C19	2.8 (6)	C56—O3—C57—C58	−171.6 (4)
C17—C18—C19—C20	−2.6 (7)	Na1—O3—C57—C58	49.4 (4)
C18—C19—C20—C15	0.0 (7)	C59—O4—C58—C57	−179.9 (4)
C16—C15—C20—C19	2.3 (6)	Na1—O4—C58—C57	43.2 (4)
C2—C15—C20—C19	−174.7 (4)	O3—C57—C58—O4	−61.1 (5)
Sc1—C15—C20—C19	−86.4 (5)	C58—O4—C59—C60	170.2 (4)
C15—C2—C21—C22	49.3 (6)	Na1—O4—C59—C60	−52.3 (4)
C1—C2—C21—C22	−152.1 (4)	C61—O5—C60—C59	−176.9 (4)
Sc1—C2—C21—C22	−61.9 (5)	Na1—O5—C60—C59	−43.8 (4)
C15—C2—C21—C26	−132.9 (4)	O4—C59—C60—O5	63.4 (5)
C1—C2—C21—C26	25.6 (5)	C60—O5—C61—C62	−173.5 (3)
Sc1—C2—C21—C26	115.9 (4)	Na1—O5—C61—C62	53.7 (4)
C26—C21—C22—C23	3.9 (6)	C63—O6—C62—C61	178.0 (4)
C2—C21—C22—C23	−178.2 (4)	Na1—O6—C62—C61	41.8 (4)
C21—C22—C23—C24	−0.5 (7)	O5—C61—C62—O6	−62.1 (4)
C22—C23—C24—C25	−2.8 (7)	C62—O6—C63—C64	171.9 (4)
C23—C24—C25—C26	2.5 (7)	Na1—O6—C63—C64	−50.8 (5)
C24—C25—C26—C21	1.2 (7)	C53—O1—C64—C63	176.7 (4)
C22—C21—C26—C25	−4.3 (6)	Na1—O1—C64—C63	−43.5 (5)
C2—C21—C26—C25	177.8 (4)	O6—C63—C64—O1	62.4 (5)
C29—C27—C28—C41	139.7 (3)	C68—O7—C65—C66	15.3 (6)
C35—C27—C28—C41	−61.6 (5)	Na1—O7—C65—C66	−148.6 (4)
Sc1—C27—C28—C41	67.4 (3)	O7—C65—C66—C67	9.5 (7)
C29—C27—C28—C47	−58.8 (5)	C65—C66—C67—C68	−28.6 (7)
C35—C27—C28—C47	99.9 (4)	C65—O7—C68—C67	−34.1 (6)

Sc1—C27—C28—C47	−131.1 (3)	Na1—O7—C68—C67	129.6 (4)
C29—C27—C28—Sc1	72.3 (3)	C66—C67—C68—O7	39.4 (6)
C35—C27—C28—Sc1	−129.0 (3)	C72B—O8—C69—C70	14.4 (13)
C35—C27—C29—C34	8.4 (6)	C72A—O8—C69—C70	−27.2 (9)
C28—C27—C29—C34	165.9 (4)	Na1—O8—C69—C70	157.2 (4)
Sc1—C27—C29—C34	−127.6 (4)	O8—C69—C70—C71A	34.1 (8)
C35—C27—C29—C30	−174.7 (3)	O8—C69—C70—C71B	−32.7 (11)
C28—C27—C29—C30	−17.1 (5)	C69—C70—C71A—C72A	−28.5 (11)
Sc1—C27—C29—C30	49.3 (3)	C69—O8—C72A—C71A	8.2 (12)
C35—C27—C29—Sc1	136.0 (4)	Na1—O8—C72A—C71A	−176.1 (7)
C28—C27—C29—Sc1	−66.4 (3)	C70—C71A—C72A—O8	13.8 (14)
C34—C29—C30—C31	1.1 (5)	C69—C70—C71B—C72B	34.5 (16)
C27—C29—C30—C31	−176.2 (4)	C69—O8—C72B—C71B	10 (2)
Sc1—C29—C30—C31	−130.6 (4)	Na1—O8—C72B—C71B	−137.3 (14)
C34—C29—C30—Sc1	131.7 (3)	C70—C71B—C72B—O8	−29 (2)
C27—C29—C30—Sc1	−45.6 (3)	C76—O9—C73—C74	−18.9 (7)
C29—C30—C31—C32	0.0 (6)	O9—C73—C74—C75	29.4 (6)
Sc1—C30—C31—C32	−98.8 (5)	C73—C74—C75—C76	−28.3 (6)
C30—C31—C32—C33	−1.3 (6)	C73—O9—C76—C75	0.3 (8)
C31—C32—C33—C34	1.5 (6)	C74—C75—C76—O9	17.5 (7)
C32—C33—C34—C29	−0.4 (6)	C80—O10—C77—C78	−12.2 (13)
C30—C29—C34—C33	−0.9 (6)	O10—C77—C78—C79	23.6 (11)
C27—C29—C34—C33	176.2 (4)	C77—C78—C79—C80	−27.3 (11)
Sc1—C29—C34—C33	88.6 (5)	C77—O10—C80—C79	−4.8 (13)
C29—C27—C35—C40	137.7 (4)	C78—C79—C80—O10	19.7 (10)

1,4-Diphenylcyclopenta-1,3-diene (3a)

Crystal data

$C_{17}H_{14}$
$M_r = 218.28$
Monoclinic, $P2_1/n$
$a = 7.3278$ (5) Å
$b = 5.9440$ (4) Å
$c = 26.6551$ (18) Å
$\beta = 94.352$ (1)°
$V = 1157.65$ (14) Å^3
$Z = 4$

$F(000) = 464$
$D_x = 1.252$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7599 reflections
$\theta = 2.8$–28.2°
$\mu = 0.07$ mm^{-1}
$T = 123$ K
Needle, pale yellow
0.60 × 0.20 × 0.20 mm

Data collection

SMART Platform CCD area detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
$T_{\min} = 0.829$, $T_{\max} = 0.985$

13442 measured reflections
2788 independent reflections
2406 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.083$
$\theta_{\max} = 28.3°$, $\theta_{\min} = 1.5°$
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 7$
$l = -33 \rightarrow 35$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.128$
$S = 1.03$
2788 reflections
210 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2 + 0.2815P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.02183 (14)	0.46040 (18)	0.20306 (4)	0.0194 (2)
C2	−0.07683 (15)	0.66331 (18)	0.22021 (4)	0.0220 (2)
H2	−0.122 (2)	0.791 (2)	0.1996 (5)	0.026 (3)*
C3	−0.06521 (15)	0.66279 (18)	0.27494 (4)	0.0218 (2)
H3	−0.1011 (19)	0.789 (2)	0.2962 (5)	0.024 (3)*
C4	−0.00283 (14)	0.45997 (18)	0.29250 (4)	0.0196 (2)
C5	0.03554 (15)	0.31609 (18)	0.24792 (4)	0.0203 (2)
H5A	0.168 (2)	0.273 (3)	0.2479 (6)	0.036 (4)*
H5B	−0.032 (2)	0.170 (2)	0.2473 (5)	0.026 (3)*
C6	−0.02328 (14)	0.38421 (17)	0.15080 (4)	0.0191 (2)
C7	0.05654 (15)	0.17886 (18)	0.13860 (4)	0.0218 (2)
H7A	0.117 (2)	0.086 (2)	0.1654 (5)	0.028 (3)*
C8	0.05238 (16)	0.10492 (19)	0.08895 (4)	0.0247 (3)
H8A	0.110 (2)	−0.037 (2)	0.0822 (5)	0.025 (3)*
C9	−0.03166 (16)	0.2347 (2)	0.05055 (4)	0.0257 (3)
H9	−0.035 (2)	0.183 (3)	0.0154 (6)	0.038 (4)*
C10	−0.11146 (16)	0.4390 (2)	0.06201 (4)	0.0249 (3)
H10	−0.173 (2)	0.532 (2)	0.0351 (5)	0.029 (4)*
C11	−0.10837 (15)	0.51292 (19)	0.11145 (4)	0.0221 (2)
H11	−0.1680 (19)	0.653 (2)	0.1185 (5)	0.024 (3)*
C12	0.01735 (14)	0.38183 (17)	0.34475 (4)	0.0195 (2)
C13	0.10418 (15)	0.17717 (18)	0.35739 (4)	0.0219 (2)
H13	0.157 (2)	0.085 (2)	0.3308 (6)	0.033 (4)*
C14	0.12069 (15)	0.10045 (19)	0.40687 (4)	0.0240 (3)
H14	0.181 (2)	−0.041 (2)	0.4144 (5)	0.030 (4)*

C15	0.04995 (16)	0.2269 (2)	0.44469 (4)	0.0246 (3)
H15	0.059 (2)	0.169 (2)	0.4800 (6)	0.033 (4)*
C16	−0.03760 (16)	0.4298 (2)	0.43276 (4)	0.0253 (3)
H16	−0.091 (2)	0.519 (2)	0.4583 (5)	0.030 (4)*
C17	−0.05411 (15)	0.50682 (19)	0.38350 (4)	0.0225 (2)
H17	−0.116 (2)	0.648 (2)	0.3761 (5)	0.027 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0159 (5)	0.0223 (5)	0.0201 (5)	−0.0014 (4)	0.0016 (4)	0.0010 (4)
C2	0.0217 (5)	0.0217 (5)	0.0226 (5)	0.0003 (4)	0.0012 (4)	0.0018 (4)
C3	0.0221 (5)	0.0217 (5)	0.0218 (5)	0.0004 (4)	0.0023 (4)	−0.0018 (4)
C4	0.0170 (5)	0.0219 (5)	0.0199 (5)	−0.0011 (4)	0.0010 (4)	−0.0015 (4)
C5	0.0207 (5)	0.0217 (5)	0.0184 (5)	0.0023 (4)	0.0012 (4)	−0.0001 (4)
C6	0.0164 (5)	0.0213 (5)	0.0196 (5)	−0.0021 (4)	0.0019 (4)	0.0010 (4)
C7	0.0217 (5)	0.0234 (5)	0.0202 (5)	0.0010 (4)	0.0018 (4)	0.0015 (4)
C8	0.0257 (6)	0.0233 (5)	0.0255 (6)	0.0008 (4)	0.0045 (4)	−0.0023 (4)
C9	0.0276 (6)	0.0298 (6)	0.0197 (5)	−0.0030 (5)	0.0018 (4)	−0.0029 (4)
C10	0.0253 (6)	0.0283 (6)	0.0205 (5)	−0.0005 (4)	−0.0009 (4)	0.0032 (4)
C11	0.0211 (5)	0.0226 (5)	0.0226 (5)	0.0011 (4)	0.0010 (4)	0.0010 (4)
C12	0.0169 (5)	0.0219 (5)	0.0194 (5)	−0.0013 (4)	0.0000 (4)	−0.0012 (4)
C13	0.0212 (5)	0.0230 (5)	0.0214 (5)	0.0017 (4)	0.0008 (4)	−0.0020 (4)
C14	0.0242 (5)	0.0233 (5)	0.0241 (6)	0.0008 (4)	−0.0008 (4)	0.0021 (4)
C15	0.0249 (5)	0.0288 (6)	0.0197 (5)	−0.0022 (4)	−0.0011 (4)	0.0013 (4)
C16	0.0264 (6)	0.0294 (6)	0.0200 (5)	0.0022 (4)	0.0008 (4)	−0.0040 (4)
C17	0.0228 (5)	0.0231 (5)	0.0213 (5)	0.0027 (4)	−0.0005 (4)	−0.0025 (4)

Geometric parameters (Å, º)

C1—C2	1.3615 (15)	C9—C10	1.3920 (17)
C2—C3	1.4548 (16)	C9—H9	0.984 (16)
C3—C4	1.3598 (15)	C10—C11	1.3877 (15)
C4—C5	1.5077 (14)	C10—H10	0.987 (14)
C1—C5	1.5051 (14)	C11—H11	0.963 (14)
C1—C6	1.4641 (14)	C12—C13	1.4020 (15)
C4—C12	1.4651 (14)	C12—C17	1.4053 (15)
C2—H2	0.978 (14)	C13—C14	1.3921 (15)
C3—H3	0.988 (13)	C13—H13	0.998 (15)
C5—H5A	1.003 (17)	C14—C15	1.3888 (16)
C5—H5B	0.998 (15)	C14—H14	0.964 (14)
C6—C7	1.4023 (15)	C15—C16	1.3915 (16)
C6—C11	1.4053 (15)	C15—H15	0.999 (15)
C7—C8	1.3928 (15)	C16—C17	1.3873 (15)
C7—H7A	0.982 (14)	C16—H16	0.967 (15)
C8—C9	1.3878 (16)	C17—H17	0.969 (14)
C8—H8A	0.968 (14)		

C2—C1—C6	127.76 (10)	C8—C9—C10	119.55 (10)
C2—C1—C5	108.07 (9)	C8—C9—H9	120.4 (9)
C6—C1—C5	124.12 (9)	C10—C9—H9	120.0 (9)
C1—C2—C3	109.78 (9)	C11—C10—C9	120.52 (10)
C1—C2—H2	126.4 (8)	C11—C10—H10	119.1 (8)
C3—C2—H2	123.8 (8)	C9—C10—H10	120.4 (8)
C4—C3—C2	109.85 (9)	C10—C11—C6	120.72 (10)
C4—C3—H3	125.0 (8)	C10—C11—H11	118.9 (8)
C2—C3—H3	125.1 (8)	C6—C11—H11	120.4 (8)
C3—C4—C12	127.98 (10)	C13—C12—C17	118.02 (10)
C3—C4—C5	108.03 (9)	C13—C12—C4	120.98 (10)
C12—C4—C5	123.92 (9)	C17—C12—C4	120.98 (10)
C1—C5—C4	104.19 (9)	C14—C13—C12	121.14 (10)
C1—C5—H5A	111.0 (9)	C14—C13—H13	118.9 (8)
C4—C5—H5A	112.6 (9)	C12—C13—H13	119.9 (8)
C1—C5—H5B	112.0 (8)	C15—C14—C13	120.00 (10)
C4—C5—H5B	112.7 (8)	C15—C14—H14	120.8 (8)
H5A—C5—H5B	104.6 (12)	C13—C14—H14	119.2 (8)
C7—C6—C11	118.04 (10)	C14—C15—C16	119.61 (10)
C7—C6—C1	121.11 (9)	C14—C15—H15	119.6 (8)
C11—C6—C1	120.83 (10)	C16—C15—H15	120.8 (8)
C8—C7—C6	121.03 (10)	C17—C16—C15	120.54 (10)
C8—C7—H7A	119.4 (8)	C17—C16—H16	118.3 (9)
C6—C7—H7A	119.6 (8)	C15—C16—H16	121.1 (9)
C9—C8—C7	120.14 (10)	C16—C17—C12	120.69 (10)
C9—C8—H8A	121.6 (8)	C16—C17—H17	119.0 (8)
C7—C8—H8A	118.3 (8)	C12—C17—H17	120.3 (8)

C6—C1—C2—C3	175.73 (10)	C8—C9—C10—C11	−0.35 (18)
C5—C1—C2—C3	−1.74 (13)	C9—C10—C11—C6	0.60 (17)
C1—C2—C3—C4	0.04 (13)	C7—C6—C11—C10	−0.55 (16)
C2—C3—C4—C12	−175.42 (10)	C1—C6—C11—C10	−179.22 (10)
C2—C3—C4—C5	1.67 (12)	C3—C4—C12—C13	−172.37 (11)
C2—C1—C5—C4	2.63 (12)	C5—C4—C12—C13	10.96 (16)
C6—C1—C5—C4	−174.95 (9)	C3—C4—C12—C17	9.37 (17)
C3—C4—C5—C1	−2.61 (11)	C5—C4—C12—C17	−167.30 (10)
C12—C4—C5—C1	174.63 (9)	C17—C12—C13—C14	−0.52 (16)
C2—C1—C6—C7	172.91 (11)	C4—C12—C13—C14	−178.83 (10)
C5—C1—C6—C7	−9.99 (16)	C12—C13—C14—C15	0.23 (17)
C2—C1—C6—C11	−8.46 (17)	C13—C14—C15—C16	0.15 (17)
C5—C1—C6—C11	168.64 (10)	C14—C15—C16—C17	−0.22 (17)
C11—C6—C7—C8	0.27 (16)	C15—C16—C17—C12	−0.08 (17)
C1—C6—C7—C8	178.93 (10)	C13—C12—C17—C16	0.45 (16)
C6—C7—C8—C9	−0.03 (17)	C4—C12—C17—C16	178.76 (10)
C7—C8—C9—C10	0.07 (18)		

(η^5-1,3-Diphenylcyclopentadienyl)(tetrahydrofuran-κO)(η^6-\ 1,1,2,2-tetraphenylethenediyl)scandium(III) toluene hemisolvate (5b)

Crystal data

$[Sc(C_{17}H_{13})(C_{26}H_{20})(C_4H_8O)]\cdot 0.5C_7H_8$	$F(000) = 3016$
$M_r = 712.82$	$D_x = 1.270$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 36.060$ (5) Å	Cell parameters from 4042 reflections
$b = 9.5557$ (14) Å	$\theta = 2.3$–$27.4°$
$c = 23.662$ (3) Å	$\mu = 0.24$ mm^{-1}
$\beta = 113.840$ (2)°	$T = 173$ K
$V = 7457.9$ (19) Å^3	Block, green
$Z = 8$	0.50 × 0.40 × 0.25 mm

Data collection

SMART Platform CCD area detector diffractometer	42147 measured reflections
Radiation source: normal-focus sealed tube	8560 independent reflections
Graphite monochromator	6319 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.044$
Absorption correction: multi-scan (SADABS; Bruker, 2003)	$\theta_{max} = 27.5°$, $\theta_{min} = 1.2°$
$T_{min} = 0.869$, $T_{max} = 0.967$	$h = -46\rightarrow46$
	$k = -12\rightarrow12$
	$l = -30\rightarrow30$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.058$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.159$	$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2 + 11.065P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
8560 reflections	$\Delta\rho_{max} = 1.17$ e Å^{-3}
467 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. moisture and air sensitive

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Sc1	0.16110 (2)	0.20739 (5)	0.10810 (2)	0.02614 (12)	
O1	0.16758 (5)	0.06005 (18)	0.04110 (8)	0.0363 (4)	
C1	0.12072 (6)	0.4045 (2)	0.08963 (10)	0.0272 (5)	

C2	0.10099 (7)	0.3125 (2)	0.03373 (10)	0.0283 (5)
C3	0.15797 (7)	0.4698 (2)	0.09571 (10)	0.0277 (5)
C4	0.17933 (7)	0.4203 (2)	0.05975 (11)	0.0302 (5)
H4	0.1641 (7)	0.369 (3)	0.0218 (12)	0.028 (6)*
C5	0.21883 (7)	0.4668 (3)	0.07138 (11)	0.0338 (5)
H5	0.232083	0.432186	0.046746	0.041*
C6	0.23846 (8)	0.5606 (3)	0.11737 (12)	0.0386 (6)
H6	0.265595	0.587912	0.126333	0.046*
C7	0.21745 (8)	0.6155 (3)	0.15102 (11)	0.0364 (6)
H7	0.230532	0.682895	0.182302	0.044*
C8	0.17882 (7)	0.5753 (2)	0.14025 (10)	0.0316 (5)
H8	0.165406	0.618517	0.162938	0.038*
C9	0.09513 (7)	0.4592 (2)	0.12129 (11)	0.0307 (5)
C10	0.05321 (8)	0.4719 (3)	0.08783 (12)	0.0402 (6)
H10	0.041554	0.445954	0.045468	0.048*
C11	0.02831 (9)	0.5219 (3)	0.11540 (15)	0.0529 (8)
H11	0.000001	0.530979	0.091514	0.063*
C12	0.04423 (10)	0.5584 (4)	0.17692 (16)	0.0569 (8)
H12	0.027137	0.593066	0.195521	0.068*
C13	0.08519 (9)	0.5439 (3)	0.21108 (14)	0.0475 (7)
H13	0.096382	0.567210	0.253750	0.057*
C14	0.11028 (8)	0.4956 (3)	0.18396 (12)	0.0364 (5)
H14	0.138522	0.487014	0.208437	0.044*
C15	0.08409 (7)	0.1823 (3)	0.04194 (10)	0.0305 (5)
C16	0.09492 (7)	0.1250 (3)	0.10254 (11)	0.0328 (5)
H16	0.1071 (8)	0.183 (3)	0.1395 (13)	0.038 (7)*
C17	0.08325 (8)	−0.0106 (3)	0.11169 (13)	0.0417 (6)
H17	0.091533	−0.045500	0.152569	0.050*
C18	0.06024 (9)	−0.0932 (3)	0.06303 (14)	0.0482 (7)
H18	0.053571	−0.186435	0.069344	0.058*
C19	0.04678 (9)	−0.0363 (3)	0.00353 (13)	0.0471 (7)
H19	0.029377	−0.090349	−0.030586	0.057*
C20	0.05791 (7)	0.0946 (3)	−0.00697 (12)	0.0385 (6)
H20	0.047916	0.128635	−0.048184	0.046*
C21	0.09059 (6)	0.3769 (3)	−0.02771 (10)	0.0285 (5)
C22	0.08549 (7)	0.5226 (3)	−0.03458 (11)	0.0326 (5)
H22	0.089402	0.578191	0.000670	0.039*
C23	0.07487 (7)	0.5876 (3)	−0.09155 (12)	0.0381 (6)
H23	0.071291	0.686198	−0.094744	0.046*
C24	0.06946 (8)	0.5097 (3)	−0.14380 (12)	0.0406 (6)
H24A	0.061571	0.553914	−0.182906	0.049*
C25	0.07570 (7)	0.3677 (3)	−0.13801 (11)	0.0387 (6)
H25	0.072635	0.313744	−0.173359	0.046*
C26	0.08641 (7)	0.3015 (3)	−0.08127 (10)	0.0325 (5)
H26	0.091028	0.203404	−0.078467	0.039*
C27	0.18663 (7)	0.1234 (3)	0.22043 (10)	0.0301 (5)
C28	0.20589 (7)	0.0384 (3)	0.19123 (10)	0.0306 (5)
H28	0.204252	−0.065974	0.188389	0.037*

C29	0.23249 (7)	0.1232 (3)	0.17491 (10)	0.0302 (5)	
C30	0.22818 (7)	0.2620 (2)	0.19229 (10)	0.0297 (5)	
H30	0.245906	0.342537	0.192420	0.036*	
C31	0.20000 (7)	0.2622 (3)	0.21949 (10)	0.0303 (5)	
H31	0.194576	0.343102	0.241854	0.036*	
C32	0.16001 (7)	0.0769 (3)	0.25089 (10)	0.0323 (5)	
C33	0.15468 (9)	−0.0642 (3)	0.25971 (13)	0.0445 (6)	
H33	0.168562	−0.132483	0.246411	0.053*	
C34	0.12934 (10)	−0.1068 (3)	0.28770 (14)	0.0522 (7)	
H34	0.125860	−0.203740	0.293010	0.063*	
C35	0.10914 (9)	−0.0092 (3)	0.30790 (13)	0.0496 (7)	
H35	0.091616	−0.038640	0.326657	0.060*	
C36	0.11466 (9)	0.1313 (3)	0.30059 (13)	0.0502 (7)	
H36	0.101115	0.199141	0.314713	0.060*	
C37	0.13999 (8)	0.1737 (3)	0.27260 (12)	0.0416 (6)	
H37	0.143749	0.270878	0.268143	0.050*	
C38	0.26046 (7)	0.0767 (3)	0.14696 (11)	0.0315 (5)	
C39	0.26366 (8)	−0.0619 (3)	0.13261 (13)	0.0404 (6)	
H39	0.246921	−0.130023	0.139959	0.049*	
C40	0.29080 (9)	−0.1039 (3)	0.10767 (14)	0.0471 (7)	
H40	0.292054	−0.199738	0.097911	0.056*	
C41	0.31568 (8)	−0.0095 (3)	0.09697 (13)	0.0451 (7)	
H41A	0.334324	−0.038589	0.080197	0.054*	
C42	0.31305 (9)	0.1285 (3)	0.11110 (15)	0.0524 (7)	
H42	0.330178	0.195331	0.103952	0.063*	
C43	0.28603 (8)	0.1723 (3)	0.13550 (14)	0.0454 (7)	
H43	0.284796	0.268622	0.144611	0.055*	
C44	0.15540 (9)	−0.0865 (3)	0.03788 (14)	0.0457 (7)	
H44A	0.128568	−0.094936	0.039776	0.055*	
H44B	0.175481	−0.140731	0.072329	0.055*	
C45	0.15362 (12)	−0.1375 (4)	−0.02309 (16)	0.0657 (9)	
H45A	0.126839	−0.117983	−0.056741	0.079*	
H45B	0.159097	−0.239275	−0.021871	0.079*	
C46	0.18686 (10)	−0.0541 (3)	−0.03149 (14)	0.0537 (8)	
H46A	0.213818	−0.096600	−0.008255	0.064*	
H46B	0.182027	−0.047169	−0.075660	0.064*	
C47	0.18356 (9)	0.0865 (3)	−0.00555 (12)	0.0425 (6)	
H47A	0.210482	0.131920	0.013273	0.051*	
H47B	0.165015	0.148154	−0.038475	0.051*	
C50	−0.0052 (2)	0.1054 (5)	0.2767 (2)	0.082 (2)*	0.5
C51	−0.0050 (2)	−0.0400 (5)	0.2771 (2)	0.071 (2)*	0.5
H51	−0.009723	−0.089434	0.308357	0.086*	0.5
C52	0.0021 (2)	−0.1132 (4)	0.2316 (3)	0.097 (3)*	0.5
H52	0.002248	−0.212621	0.231908	0.116*	0.5
C53	0.00904 (19)	−0.0409 (5)	0.1859 (2)	0.073 (2)*	0.5
H53	0.013898	−0.090935	0.154817	0.087*	0.5
C54	0.0088 (2)	0.1045 (5)	0.1855 (2)	0.123 (4)*	0.5
H54	0.013577	0.153939	0.154174	0.148*	0.5

C55	0.0017 (2)	0.1777 (4)	0.2309 (3)	0.0672 (19)*	0.5
H55	0.001606	0.277129	0.230623	0.081*	0.5
C56	−0.0155 (3)	0.1883 (12)	0.3198 (5)	0.109 (3)*	0.5
H56A	0.002670	0.269398	0.333210	0.164*	0.5
H56B	−0.012382	0.131008	0.355728	0.164*	0.5
H56C	−0.043581	0.220523	0.299415	0.164*	0.5

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sc1	0.0241 (2)	0.0307 (2)	0.0234 (2)	−0.00120 (17)	0.00929 (17)	−0.00064 (17)
O1	0.0417 (10)	0.0368 (9)	0.0348 (9)	−0.0016 (8)	0.0201 (8)	−0.0040 (7)
C1	0.0248 (11)	0.0330 (12)	0.0223 (10)	0.0002 (9)	0.0081 (9)	0.0005 (9)
C2	0.0233 (11)	0.0356 (12)	0.0242 (11)	−0.0012 (9)	0.0079 (9)	−0.0014 (9)
C3	0.0279 (11)	0.0294 (12)	0.0237 (11)	0.0012 (9)	0.0082 (9)	0.0020 (9)
C4	0.0299 (12)	0.0332 (12)	0.0268 (11)	−0.0026 (10)	0.0108 (10)	0.0001 (10)
C5	0.0303 (12)	0.0400 (14)	0.0338 (12)	−0.0020 (10)	0.0158 (10)	0.0047 (10)
C6	0.0306 (13)	0.0409 (14)	0.0410 (14)	−0.0081 (11)	0.0111 (11)	0.0048 (11)
C7	0.0389 (13)	0.0318 (12)	0.0327 (12)	−0.0083 (10)	0.0085 (11)	−0.0008 (10)
C8	0.0350 (13)	0.0308 (12)	0.0274 (11)	−0.0015 (10)	0.0110 (10)	−0.0007 (9)
C9	0.0311 (12)	0.0316 (12)	0.0319 (12)	0.0009 (10)	0.0152 (10)	0.0000 (10)
C10	0.0320 (13)	0.0490 (16)	0.0403 (14)	0.0006 (11)	0.0154 (11)	−0.0018 (12)
C11	0.0325 (14)	0.067 (2)	0.064 (2)	0.0034 (13)	0.0243 (14)	−0.0041 (16)
C12	0.0532 (18)	0.069 (2)	0.065 (2)	0.0030 (15)	0.0421 (17)	−0.0118 (16)
C13	0.0560 (18)	0.0532 (17)	0.0436 (15)	−0.0029 (14)	0.0307 (14)	−0.0105 (13)
C14	0.0360 (13)	0.0400 (14)	0.0350 (13)	0.0014 (11)	0.0164 (11)	−0.0015 (11)
C15	0.0218 (10)	0.0398 (13)	0.0281 (11)	−0.0027 (9)	0.0085 (9)	−0.0023 (10)
C16	0.0268 (11)	0.0424 (14)	0.0286 (12)	−0.0046 (10)	0.0107 (10)	0.0001 (10)
C17	0.0341 (13)	0.0499 (16)	0.0398 (14)	−0.0079 (12)	0.0136 (12)	0.0088 (12)
C18	0.0425 (15)	0.0450 (16)	0.0539 (17)	−0.0134 (13)	0.0160 (13)	0.0044 (13)
C19	0.0418 (15)	0.0506 (17)	0.0437 (15)	−0.0176 (13)	0.0120 (13)	−0.0103 (13)
C20	0.0336 (13)	0.0474 (15)	0.0311 (13)	−0.0082 (11)	0.0096 (10)	−0.0015 (11)
C21	0.0195 (10)	0.0389 (13)	0.0247 (11)	−0.0006 (9)	0.0065 (9)	0.0002 (9)
C22	0.0254 (11)	0.0396 (13)	0.0303 (12)	−0.0020 (10)	0.0086 (10)	−0.0014 (10)
C23	0.0288 (12)	0.0424 (14)	0.0405 (14)	−0.0004 (10)	0.0114 (11)	0.0092 (11)
C24	0.0346 (13)	0.0574 (17)	0.0289 (12)	−0.0006 (12)	0.0119 (11)	0.0115 (11)
C25	0.0327 (13)	0.0572 (17)	0.0274 (12)	−0.0020 (12)	0.0133 (10)	−0.0019 (11)
C26	0.0277 (11)	0.0405 (13)	0.0292 (11)	−0.0013 (10)	0.0114 (10)	−0.0026 (10)
C27	0.0267 (11)	0.0391 (13)	0.0217 (10)	0.0006 (10)	0.0068 (9)	0.0038 (9)
C28	0.0277 (11)	0.0349 (13)	0.0271 (11)	0.0022 (10)	0.0088 (9)	0.0051 (9)
C29	0.0249 (11)	0.0360 (13)	0.0275 (11)	0.0015 (9)	0.0082 (9)	0.0034 (9)
C30	0.0232 (11)	0.0346 (12)	0.0261 (11)	−0.0011 (9)	0.0047 (9)	0.0010 (9)
C31	0.0278 (12)	0.0366 (13)	0.0217 (10)	0.0016 (10)	0.0049 (9)	0.0003 (9)
C32	0.0276 (11)	0.0446 (14)	0.0225 (11)	0.0000 (10)	0.0080 (9)	0.0049 (10)
C33	0.0481 (16)	0.0469 (16)	0.0446 (15)	−0.0033 (13)	0.0252 (13)	0.0004 (12)
C34	0.0580 (18)	0.0517 (17)	0.0546 (18)	−0.0127 (15)	0.0306 (15)	0.0050 (14)
C35	0.0404 (15)	0.073 (2)	0.0409 (15)	−0.0055 (14)	0.0218 (13)	0.0081 (14)
C36	0.0496 (17)	0.0636 (19)	0.0465 (16)	0.0114 (14)	0.0287 (14)	0.0087 (14)

C37	0.0436 (15)	0.0477 (16)	0.0374 (14)	0.0082 (12)	0.0203 (12)	0.0101 (11)
C38	0.0253 (11)	0.0381 (13)	0.0301 (12)	0.0029 (10)	0.0099 (9)	0.0050 (10)
C39	0.0366 (14)	0.0387 (14)	0.0499 (16)	−0.0006 (11)	0.0214 (12)	0.0026 (12)
C40	0.0465 (16)	0.0432 (15)	0.0585 (18)	0.0056 (13)	0.0285 (14)	−0.0017 (13)
C41	0.0392 (15)	0.0573 (18)	0.0456 (15)	0.0099 (13)	0.0241 (13)	0.0062 (13)
C42	0.0475 (17)	0.0553 (18)	0.069 (2)	0.0003 (14)	0.0380 (16)	0.0093 (15)
C43	0.0418 (15)	0.0404 (15)	0.0617 (18)	−0.0027 (12)	0.0289 (14)	0.0016 (13)
C44	0.0525 (17)	0.0364 (15)	0.0527 (17)	−0.0046 (12)	0.0261 (14)	−0.0069 (12)
C45	0.080 (2)	0.060 (2)	0.063 (2)	−0.0177 (18)	0.0360 (19)	−0.0278 (17)
C46	0.0538 (18)	0.065 (2)	0.0478 (17)	0.0031 (15)	0.0258 (15)	−0.0149 (14)
C47	0.0449 (15)	0.0534 (17)	0.0361 (14)	−0.0014 (13)	0.0237 (12)	−0.0021 (12)

Geometric parameters (Å, º)

Sc1—C1	2.312 (2)	C24—C25	1.374 (4)
Sc1—C2	2.392 (2)	C24—H24A	0.9500
Sc1—C3	2.522 (2)	C25—C26	1.390 (3)
Sc1—C4	2.547 (2)	C25—H25	0.9500
Sc1—C15	2.589 (2)	C26—H26	0.9500
Sc1—C16	2.465 (2)	C27—C31	1.414 (3)
Sc1—C27	2.565 (2)	C27—C28	1.417 (3)
Sc1—C28	2.553 (2)	C28—C29	1.423 (3)
Sc1—C29	2.546 (2)	C28—H28	1.0000
Sc1—C30	2.489 (2)	C29—C30	1.415 (3)
Sc1—C31	2.490 (2)	C30—C31	1.405 (3)
Sc1—O1	2.2024 (17)	C30—H30	1.0000
Sc1—H16	2.36 (3)	C31—H31	1.0000
C1—C3	1.434 (3)	C32—C33	1.390 (4)
C3—C4	1.439 (3)	C32—C37	1.394 (4)
C4—C5	1.409 (3)	C33—C34	1.389 (4)
C5—C6	1.366 (4)	C33—H33	0.9500
C6—C7	1.404 (4)	C34—C35	1.382 (4)
C7—C8	1.366 (3)	C34—H34	0.9500
C3—C8	1.432 (3)	C35—C36	1.378 (4)
C1—C9	1.498 (3)	C35—H35	0.9500
C2—C15	1.433 (3)	C36—C37	1.388 (4)
C15—C16	1.434 (3)	C36—H36	0.9500
C16—C17	1.406 (4)	C37—H37	0.9500
C17—C18	1.366 (4)	C38—C39	1.384 (4)
C18—C19	1.400 (4)	C38—C43	1.401 (3)
C19—C20	1.367 (4)	C39—C40	1.391 (4)
C15—C20	1.431 (3)	C39—H39	0.9500
C2—C21	1.481 (3)	C40—C41	1.366 (4)
C27—C32	1.482 (3)	C40—H40	0.9500
C29—C38	1.480 (3)	C41—C42	1.373 (4)
C1—C2	1.506 (3)	C41—H41A	0.9500
O1—C47	1.459 (3)	C42—C43	1.383 (4)
O1—C44	1.460 (3)	C42—H42	0.9500

C4—H4	0.97 (3)	C43—H43	0.9500
C5—H5	0.9500	C44—C45	1.500 (4)
C6—H6	0.9500	C44—H44A	0.9900
C7—H7	0.9500	C44—H44B	0.9900
C8—H8	0.9500	C45—C46	1.518 (5)
C9—C10	1.400 (3)	C45—H45A	0.9900
C9—C14	1.401 (3)	C45—H45B	0.9900
C10—C11	1.391 (4)	C46—C47	1.502 (4)
C10—H10	0.9500	C46—H46A	0.9900
C11—C12	1.376 (4)	C46—H46B	0.9900
C11—H11	0.9500	C47—H47A	0.9900
C12—C13	1.375 (4)	C47—H47B	0.9900
C12—H12	0.9500	C50—C51	1.3900
C13—C14	1.383 (4)	C50—C55	1.3900
C13—H13	0.9500	C50—C56	1.452 (11)
C14—H14	0.9500	C51—C52	1.3900
C16—H16	0.98 (3)	C51—H51	0.9500
C17—H17	0.9500	C52—C53	1.3900
C18—H18	0.9500	C52—H52	0.9500
C19—H19	0.9500	C53—C54	1.3900
C20—H20	0.9500	C53—H53	0.9500
C21—C22	1.406 (3)	C54—C55	1.3900
C21—C26	1.411 (3)	C54—H54	0.9500
C22—C23	1.390 (3)	C55—H55	0.9500
C22—H22	0.9500	C56—H56A	0.9800
C23—C24	1.387 (4)	C56—H56B	0.9800
C23—H23	0.9500	C56—H56C	0.9800

O1—Sc1—C1	128.82 (7)	Sc1—C16—H16	72.4 (15)
O1—Sc1—C2	93.85 (7)	C18—C17—C16	121.4 (2)
C1—Sc1—C2	37.30 (7)	C18—C17—H17	119.3
O1—Sc1—C16	98.03 (8)	C16—C17—H17	119.3
C1—Sc1—C16	74.22 (8)	C17—C18—C19	118.0 (3)
C2—Sc1—C16	61.64 (8)	C17—C18—H18	121.0
O1—Sc1—C30	111.00 (7)	C19—C18—H18	121.0
C1—Sc1—C30	108.34 (8)	C20—C19—C18	122.0 (3)
C2—Sc1—C30	143.07 (8)	C20—C19—H19	119.0
C16—Sc1—C30	135.74 (8)	C18—C19—H19	119.0
O1—Sc1—C31	135.90 (7)	C19—C20—C15	122.3 (2)
C1—Sc1—C31	94.08 (8)	C19—C20—H20	118.8
C2—Sc1—C31	130.22 (8)	C15—C20—H20	118.8
C16—Sc1—C31	104.04 (8)	C22—C21—C26	116.1 (2)
C30—Sc1—C31	32.79 (7)	C22—C21—C2	119.6 (2)
O1—Sc1—C3	124.39 (7)	C26—C21—C2	124.2 (2)
C1—Sc1—C3	34.17 (7)	C23—C22—C21	121.8 (2)
C2—Sc1—C3	60.80 (7)	C23—C22—H22	119.1
C16—Sc1—C3	108.33 (8)	C21—C22—H22	119.1
C30—Sc1—C3	82.42 (7)	C24—C23—C22	120.6 (2)

C31—Sc1—C3	83.89 (8)	C24—C23—H23	119.7
O1—Sc1—C29	82.53 (7)	C22—C23—H23	119.7
C1—Sc1—C29	140.91 (8)	C25—C24—C23	118.9 (2)
C2—Sc1—C29	167.86 (8)	C25—C24—H24A	120.6
C16—Sc1—C29	130.25 (8)	C23—C24—H24A	120.6
C30—Sc1—C29	32.62 (8)	C24—C25—C26	121.1 (2)
C31—Sc1—C29	54.10 (8)	C24—C25—H25	119.5
C3—Sc1—C29	111.89 (8)	C26—C25—H25	119.5
O1—Sc1—C4	93.76 (7)	C25—C26—C21	121.5 (2)
C1—Sc1—C4	61.41 (8)	C25—C26—H26	119.2
C2—Sc1—C4	69.87 (8)	C21—C26—H26	119.2
C16—Sc1—C4	130.62 (8)	C31—C27—C28	107.0 (2)
C30—Sc1—C4	81.28 (8)	C31—C27—C32	125.5 (2)
C31—Sc1—C4	99.80 (8)	C28—C27—C32	127.3 (2)
C3—Sc1—C4	32.98 (7)	C31—C27—Sc1	70.86 (12)
C29—Sc1—C4	98.71 (8)	C28—C27—Sc1	73.46 (13)
O1—Sc1—C28	86.11 (7)	C32—C27—Sc1	124.47 (15)
C1—Sc1—C28	144.58 (8)	C27—C28—C29	108.9 (2)
C2—Sc1—C28	159.32 (8)	C27—C28—Sc1	74.40 (13)
C16—Sc1—C28	97.84 (8)	C29—C28—Sc1	73.55 (13)
C30—Sc1—C28	53.72 (8)	C27—C28—H28	125.3
C31—Sc1—C28	53.63 (8)	C29—C28—H28	125.3
C3—Sc1—C28	134.68 (8)	Sc1—C28—H28	125.3
C29—Sc1—C28	32.41 (7)	C30—C29—C28	106.8 (2)
C4—Sc1—C28	130.80 (8)	C30—C29—C38	125.9 (2)
O1—Sc1—C27	116.61 (7)	C28—C29—C38	127.3 (2)
C1—Sc1—C27	112.68 (8)	C30—C29—Sc1	71.46 (13)
C2—Sc1—C27	137.12 (8)	C28—C29—Sc1	74.04 (13)
C16—Sc1—C27	83.66 (8)	C38—C29—Sc1	121.19 (15)
C30—Sc1—C27	53.88 (7)	C31—C30—C29	108.6 (2)
C31—Sc1—C27	32.45 (8)	C31—C30—Sc1	73.63 (13)
C3—Sc1—C27	114.33 (8)	C29—C30—Sc1	75.92 (13)
C29—Sc1—C27	53.73 (7)	C31—C30—H30	125.2
C4—Sc1—C27	131.73 (8)	C29—C30—H30	125.2
C28—Sc1—C27	32.14 (7)	Sc1—C30—H30	125.2
O1—Sc1—C15	84.57 (7)	C30—C31—C27	108.7 (2)
C1—Sc1—C15	61.43 (8)	C30—C31—Sc1	73.58 (13)
C2—Sc1—C15	33.14 (8)	C27—C31—Sc1	76.69 (13)
C16—Sc1—C15	32.83 (7)	C30—C31—H31	125.2
C30—Sc1—C15	163.95 (8)	C27—C31—H31	125.2
C31—Sc1—C15	131.85 (8)	Sc1—C31—H31	125.2
C3—Sc1—C15	92.02 (7)	C33—C32—C37	117.7 (2)
C29—Sc1—C15	156.07 (8)	C33—C32—C27	121.3 (2)
C4—Sc1—C15	102.18 (8)	C37—C32—C27	120.9 (2)
C28—Sc1—C15	126.66 (8)	C34—C33—C32	120.9 (3)
C27—Sc1—C15	116.46 (7)	C34—C33—H33	119.5
O1—Sc1—H16	120.3 (7)	C32—C33—H33	119.5
C1—Sc1—H16	66.7 (6)	C35—C34—C33	120.5 (3)

C2—Sc1—H16	69.9 (7)	C35—C34—H34	119.8
C16—Sc1—H16	23.2 (7)	C33—C34—H34	119.8
C30—Sc1—H16	115.3 (7)	C36—C35—C34	119.4 (3)
C31—Sc1—H16	82.6 (7)	C36—C35—H35	120.3
C3—Sc1—H16	97.6 (6)	C34—C35—H35	120.3
C29—Sc1—H16	121.9 (7)	C35—C36—C37	120.1 (3)
C4—Sc1—H16	128.1 (6)	C35—C36—H36	120.0
C28—Sc1—H16	92.3 (6)	C37—C36—H36	120.0
C27—Sc1—H16	68.8 (7)	C36—C37—C32	121.4 (3)
C15—Sc1—H16	50.3 (7)	C36—C37—H37	119.3
C47—O1—C44	109.21 (19)	C32—C37—H37	119.3
C47—O1—Sc1	128.56 (15)	C39—C38—C43	116.8 (2)
C44—O1—Sc1	122.23 (15)	C39—C38—C29	122.5 (2)
C3—C1—C9	122.6 (2)	C43—C38—C29	120.7 (2)
C3—C1—C2	115.64 (19)	C38—C39—C40	121.5 (2)
C9—C1—C2	118.35 (19)	C38—C39—H39	119.2
C3—C1—Sc1	80.95 (13)	C40—C39—H39	119.2
C9—C1—Sc1	131.12 (15)	C41—C40—C39	121.1 (3)
C2—C1—Sc1	74.22 (12)	C41—C40—H40	119.5
C15—C2—C21	122.7 (2)	C39—C40—H40	119.5
C15—C2—C1	117.64 (19)	C40—C41—C42	118.3 (3)
C21—C2—C1	117.5 (2)	C40—C41—H41A	120.9
C15—C2—Sc1	81.03 (13)	C42—C41—H41A	120.9
C21—C2—Sc1	133.74 (15)	C41—C42—C43	121.5 (3)
C1—C2—Sc1	68.48 (12)	C41—C42—H42	119.3
C8—C3—C1	124.9 (2)	C43—C42—H42	119.3
C8—C3—C4	114.8 (2)	C42—C43—C38	120.9 (3)
C1—C3—C4	120.1 (2)	C42—C43—H43	119.6
C8—C3—Sc1	128.96 (15)	C38—C43—H43	119.6
C1—C3—Sc1	64.87 (12)	O1—C44—C45	104.9 (2)
C4—C3—Sc1	74.48 (13)	O1—C44—H44A	110.8
C5—C4—C3	121.4 (2)	C45—C44—H44A	110.8
C5—C4—Sc1	126.03 (16)	O1—C44—H44B	110.8
C3—C4—Sc1	72.54 (13)	C45—C44—H44B	110.8
C5—C4—H4	119.3 (14)	H44A—C44—H44B	108.8
C3—C4—H4	118.2 (14)	C44—C45—C46	103.1 (2)
Sc1—C4—H4	81.9 (14)	C44—C45—H45A	111.2
C6—C5—C4	121.3 (2)	C46—C45—H45A	111.2
C6—C5—H5	119.3	C44—C45—H45B	111.2
C4—C5—H5	119.3	C46—C45—H45B	111.2
C5—C6—C7	118.2 (2)	H45A—C45—H45B	109.1
C5—C6—H6	120.9	C47—C46—C45	102.7 (2)
C7—C6—H6	120.9	C47—C46—H46A	111.2
C8—C7—C6	122.2 (2)	C45—C46—H46A	111.2
C8—C7—H7	118.9	C47—C46—H46B	111.2
C6—C7—H7	118.9	C45—C46—H46B	111.2
C7—C8—C3	121.8 (2)	H46A—C46—H46B	109.1
C7—C8—H8	119.1	O1—C47—C46	106.0 (2)

C3—C8—H8	119.1	O1—C47—H47A	110.5
C10—C9—C14	116.5 (2)	C46—C47—H47A	110.5
C10—C9—C1	119.4 (2)	O1—C47—H47B	110.5
C14—C9—C1	124.1 (2)	C46—C47—H47B	110.5
C11—C10—C9	121.3 (3)	H47A—C47—H47B	108.7
C11—C10—H10	119.4	C51—C50—C55	120.0
C9—C10—H10	119.4	C51—C50—C56	122.9 (5)
C12—C11—C10	120.7 (3)	C55—C50—C56	117.0 (5)
C12—C11—H11	119.6	C52—C51—C50	120.0
C10—C11—H11	119.6	C52—C51—H51	120.0
C13—C12—C11	119.1 (3)	C50—C51—H51	120.0
C13—C12—H12	120.5	C51—C52—C53	120.0
C11—C12—H12	120.5	C51—C52—H52	120.0
C12—C13—C14	120.7 (3)	C53—C52—H52	120.0
C12—C13—H13	119.7	C54—C53—C52	120.0
C14—C13—H13	119.7	C54—C53—H53	120.0
C13—C14—C9	121.7 (2)	C52—C53—H53	120.0
C13—C14—H14	119.1	C55—C54—C53	120.0
C9—C14—H14	119.1	C55—C54—H54	120.0
C20—C15—C2	125.2 (2)	C53—C54—H54	120.0
C20—C15—C16	114.2 (2)	C54—C55—C50	120.0
C2—C15—C16	120.5 (2)	C54—C55—H55	120.0
C20—C15—Sc1	137.63 (17)	C50—C55—H55	120.0
C2—C15—Sc1	65.83 (12)	C50—C56—H56A	109.5
C16—C15—Sc1	68.82 (12)	C50—C56—H56B	109.5
C17—C16—C15	121.9 (2)	H56A—C56—H56B	109.5
C17—C16—Sc1	129.02 (18)	C50—C56—H56C	109.5
C15—C16—Sc1	78.35 (13)	H56A—C56—H56C	109.5
C17—C16—H16	115.9 (16)	H56B—C56—H56C	109.5
C15—C16—H16	121.5 (16)		

C3—C1—C2—C15	138.2 (2)	C21—C22—C23—C24	−0.9 (4)
C9—C1—C2—C15	−61.9 (3)	C22—C23—C24—C25	−1.5 (4)
Sc1—C1—C2—C15	66.69 (18)	C23—C24—C25—C26	1.4 (4)
C3—C1—C2—C21	−57.7 (3)	C24—C25—C26—C21	1.2 (4)
C9—C1—C2—C21	102.1 (2)	C22—C21—C26—C25	−3.4 (3)
Sc1—C1—C2—C21	−129.24 (18)	C2—C21—C26—C25	178.5 (2)
C3—C1—C2—Sc1	71.52 (17)	C31—C27—C28—C29	2.7 (2)
C9—C1—C2—Sc1	−128.61 (19)	C32—C27—C28—C29	−172.9 (2)
C9—C1—C3—C8	12.2 (3)	Sc1—C27—C28—C29	66.05 (16)
C2—C1—C3—C8	171.1 (2)	C31—C27—C28—Sc1	−63.36 (15)
Sc1—C1—C3—C8	−121.3 (2)	C32—C27—C28—Sc1	121.1 (2)
C9—C1—C3—C4	−174.3 (2)	C27—C28—C29—C30	−2.2 (3)
C2—C1—C3—C4	−15.4 (3)	Sc1—C28—C29—C30	64.42 (15)
Sc1—C1—C3—C4	52.2 (2)	C27—C28—C29—C38	176.1 (2)
C9—C1—C3—Sc1	133.5 (2)	Sc1—C28—C29—C38	−117.3 (2)
C2—C1—C3—Sc1	−67.55 (17)	C27—C28—C29—Sc1	−66.60 (16)
C8—C3—C4—C5	4.2 (3)	C28—C29—C30—C31	0.8 (3)

C1—C3—C4—C5	−170.0 (2)	C38—C29—C30—C31	−177.4 (2)
Sc1—C3—C4—C5	−122.0 (2)	Sc1—C29—C30—C31	66.98 (15)
C8—C3—C4—Sc1	126.19 (19)	C28—C29—C30—Sc1	−66.15 (16)
C1—C3—C4—Sc1	−47.93 (18)	C38—C29—C30—Sc1	115.6 (2)
C3—C4—C5—C6	0.1 (4)	C29—C30—C31—C27	0.8 (3)
Sc1—C4—C5—C6	−90.4 (3)	Sc1—C30—C31—C27	69.33 (16)
C4—C5—C6—C7	−3.2 (4)	C29—C30—C31—Sc1	−68.50 (16)
C5—C6—C7—C8	1.8 (4)	C28—C27—C31—C30	−2.2 (2)
C6—C7—C8—C3	2.8 (4)	C32—C27—C31—C30	173.5 (2)
C1—C3—C8—C7	168.2 (2)	Sc1—C27—C31—C30	−67.25 (15)
C4—C3—C8—C7	−5.6 (3)	C28—C27—C31—Sc1	65.09 (15)
Sc1—C3—C8—C7	84.1 (3)	C32—C27—C31—Sc1	−119.2 (2)
C3—C1—C9—C10	133.8 (2)	C31—C27—C32—C33	−167.5 (2)
C2—C1—C9—C10	−24.6 (3)	C28—C27—C32—C33	7.3 (4)
Sc1—C1—C9—C10	−118.1 (2)	Sc1—C27—C32—C33	102.4 (3)
C3—C1—C9—C14	−48.2 (3)	C31—C27—C32—C37	11.4 (4)
C2—C1—C9—C14	153.5 (2)	C28—C27—C32—C37	−173.8 (2)
Sc1—C1—C9—C14	60.0 (3)	Sc1—C27—C32—C37	−78.7 (3)
C14—C9—C10—C11	1.7 (4)	C37—C32—C33—C34	1.7 (4)
C1—C9—C10—C11	179.9 (3)	C27—C32—C33—C34	−179.3 (2)
C9—C10—C11—C12	−1.0 (5)	C32—C33—C34—C35	−0.6 (5)
C10—C11—C12—C13	−0.4 (5)	C33—C34—C35—C36	−0.6 (5)
C11—C12—C13—C14	1.1 (5)	C34—C35—C36—C37	0.6 (5)
C12—C13—C14—C9	−0.4 (4)	C35—C36—C37—C32	0.6 (4)
C10—C9—C14—C13	−1.0 (4)	C33—C32—C37—C36	−1.7 (4)
C1—C9—C14—C13	−179.1 (2)	C27—C32—C37—C36	179.3 (2)
C21—C2—C15—C20	4.5 (4)	C30—C29—C38—C39	178.4 (2)
C1—C2—C15—C20	167.7 (2)	C28—C29—C38—C39	0.4 (4)
Sc1—C2—C15—C20	−132.4 (2)	Sc1—C29—C38—C39	−92.8 (3)
C21—C2—C15—C16	−179.5 (2)	C30—C29—C38—C43	0.3 (4)
C1—C2—C15—C16	−16.3 (3)	C28—C29—C38—C43	−177.6 (2)
Sc1—C2—C15—C16	43.6 (2)	Sc1—C29—C38—C43	89.1 (3)
C21—C2—C15—Sc1	136.9 (2)	C43—C38—C39—C40	−0.4 (4)
C1—C2—C15—Sc1	−59.87 (17)	C29—C38—C39—C40	−178.5 (2)
C20—C15—C16—C17	5.0 (3)	C38—C39—C40—C41	0.7 (4)
C2—C15—C16—C17	−171.4 (2)	C39—C40—C41—C42	−0.4 (4)
Sc1—C15—C16—C17	−129.0 (2)	C40—C41—C42—C43	−0.1 (5)
C20—C15—C16—Sc1	134.0 (2)	C41—C42—C43—C38	0.3 (5)
C2—C15—C16—Sc1	−42.4 (2)	C39—C38—C43—C42	−0.1 (4)
C15—C16—C17—C18	−1.4 (4)	C29—C38—C43—C42	178.1 (3)
Sc1—C16—C17—C18	−102.8 (3)	C47—O1—C44—C45	−15.4 (3)
C16—C17—C18—C19	−3.2 (4)	Sc1—O1—C44—C45	164.5 (2)
C17—C18—C19—C20	3.8 (4)	O1—C44—C45—C46	32.8 (3)
C18—C19—C20—C15	0.2 (4)	C44—C45—C46—C47	−37.5 (3)
C2—C15—C20—C19	171.8 (3)	C44—O1—C47—C46	−8.5 (3)
C16—C15—C20—C19	−4.4 (4)	Sc1—O1—C47—C46	171.60 (17)
Sc1—C15—C20—C19	80.1 (3)	C45—C46—C47—O1	28.5 (3)
C15—C2—C21—C22	140.7 (2)	C55—C50—C51—C52	0.0

C1—C2—C21—C22	−22.5 (3)	C56—C50—C51—C52	175.1 (7)
Sc1—C2—C21—C22	−108.3 (2)	C50—C51—C52—C53	0.0
C15—C2—C21—C26	−41.3 (3)	C51—C52—C53—C54	0.0
C1—C2—C21—C26	155.5 (2)	C52—C53—C54—C55	0.0
Sc1—C2—C21—C26	69.8 (3)	C53—C54—C55—C50	0.0
C26—C21—C22—C23	3.2 (3)	C51—C50—C55—C54	0.0
C2—C21—C22—C23	−178.6 (2)	C56—C50—C55—C54	−175.4 (7)

Poly[[(μ_2-η^3:η^2-anthracenediyl)bis(η^6-anthracenediyl)bis(η^5-1,3-diphenylcyclopentadienyl)tetrakis(tetrahydrofuran)dipotassiumdiscandium(III)] tetrahydrofuran monosolvate] (6)

Crystal data

$[K_2Sc_2(C_{14}H_{10})_3(C_{17}H_{13})_2(C_4H_8O)_4]\cdot C_4H_8O$
$M_r = 1497.84$
Orthorhombic, *Ibam*
$a = 18.1056$ (12) Å
$b = 19.9164$ (13) Å
$c = 21.8633$ (14) Å
$V = 7883.9$ (9) Å^3
$Z = 4$
$F(000) = 3168$
$D_x = 1.262$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3572 reflections
$\theta = 2.3$–21.1°
$\mu = 0.33$ mm^{-1}
$T = 150$ K
Needle, black
0.25 × 0.08 × 0.07 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan SADABS (Bruker, 2008)
$T_{min} = 0.922$, $T_{max} = 0.977$
28915 measured reflections
3990 independent reflections
2831 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.3°$
$h = -22\rightarrow22$
$k = -24\rightarrow24$
$l = -26\rightarrow26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.150$
$S = 1.02$
3990 reflections
307 parameters
128 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 17.0202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.77$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Special details

Experimental. moisture and air sensitive

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
Sc1	0.48353 (4)	0.15226 (3)	0.500000	0.01985 (19)	
K1	0.26085 (6)	0.31531 (5)	0.500000	0.0534 (3)	
C1	0.5969 (2)	0.22584 (18)	0.500000	0.0240 (8)	
H1	0.596616	0.276053	0.500000	0.029*	
C2	0.60150 (15)	0.18498 (13)	0.44739 (12)	0.0269 (6)	
C3	0.60876 (14)	0.11778 (13)	0.46797 (13)	0.0293 (6)	
H3	0.620551	0.078264	0.441498	0.035*	
C4	0.60264 (18)	0.20723 (17)	0.38291 (13)	0.0412 (8)	
C5A	0.6009 (4)	0.1498 (4)	0.3372 (4)	0.0348 (14)	0.501 (5)
H5A	0.594113	0.104615	0.349868	0.042*	0.501 (5)
C6A	0.6097 (5)	0.1666 (4)	0.2766 (3)	0.0491 (15)	0.501 (5)
H6A	0.604240	0.132697	0.246384	0.059*	0.501 (5)
C7A	0.6261 (6)	0.2312 (5)	0.2587 (4)	0.058 (2)	0.501 (5)
H7A	0.630523	0.242182	0.216524	0.069*	0.501 (5)
C8A	0.6363 (16)	0.2813 (10)	0.3045 (14)	0.062 (4)	0.501 (5)
H8A	0.653422	0.324162	0.291663	0.074*	0.501 (5)
C5B	0.5733 (4)	0.1764 (4)	0.3345 (4)	0.0348 (14)	0.499 (5)
H5B	0.549443	0.134647	0.341532	0.042*	0.499 (5)
C6B	0.5744 (5)	0.2003 (4)	0.2742 (3)	0.0491 (15)	0.499 (5)
H6B	0.558307	0.172589	0.241483	0.059*	0.499 (5)
C7B	0.5991 (6)	0.2639 (5)	0.2633 (4)	0.058 (2)	0.499 (5)
H7B	0.601600	0.280349	0.222572	0.069*	0.499 (5)
C8B	0.6194 (16)	0.3028 (10)	0.3094 (15)	0.062 (4)	0.499 (5)
H8B	0.631027	0.348901	0.303722	0.074*	0.499 (5)
C9	0.6229 (2)	0.27186 (19)	0.36731 (15)	0.0526 (9)	
H9A	0.627430	0.306948	0.396507	0.063*	
C10	0.2393 (2)	0.12700 (17)	0.4688 (2)	0.089 (2)	
H10	0.201536	0.103772	0.447311	0.106*	
C11	0.2938 (2)	0.16058 (16)	0.4366 (2)	0.0625 (12)	
H11	0.295732	0.156101	0.391069	0.075*	
C12	0.35222 (17)	0.19362 (14)	0.46734 (16)	0.0420 (8)	
C13	0.41487 (18)	0.22528 (14)	0.43777 (15)	0.0383 (7)	
H13	0.419812	0.221162	0.392373	0.046*	
C14	0.44205 (17)	0.28693 (14)	0.46756 (14)	0.0380 (7)	
C15	0.4708 (2)	0.34257 (16)	0.43683 (18)	0.0531 (9)	
H15	0.469698	0.343641	0.393403	0.064*	
C16	0.5009 (2)	0.39611 (16)	0.46843 (19)	0.0648 (12)	
H16	0.521595	0.432896	0.446700	0.078*	
C19	0.47132 (14)	0.02329 (13)	0.61377 (14)	0.0294 (6)	
H19	0.450960	0.038584	0.651314	0.035*	0.5

C17	0.4702 (3)	0.0220 (3)	0.7174 (2)	0.0289 (12)	0.5
H17	0.448897	0.037324	0.755065	0.035*	0.5
C18	0.4412 (3)	0.0444 (2)	0.6632 (2)	0.0239 (11)	0.5
H18	0.400401	0.074380	0.662291	0.029*	0.5
C20	0.44307 (15)	0.04811 (13)	0.55660 (14)	0.0319 (7)	
H20	0.406241	0.082074	0.556967	0.038*	
C21	0.46865 (19)	0.02331 (17)	0.500000	0.0290 (9)	
O1	0.27986 (16)	0.39032 (13)	0.59634 (15)	0.0765 (9)	
C22A	0.2907 (7)	0.4599 (3)	0.5888 (3)	0.103 (2)	0.728 (10)
H22A	0.258655	0.477611	0.555794	0.123*	0.728 (10)
H22B	0.342796	0.469800	0.578545	0.123*	0.728 (10)
C23A	0.2703 (6)	0.4904 (3)	0.6491 (3)	0.111 (2)	0.728 (10)
H23A	0.305852	0.525911	0.660733	0.133*	0.728 (10)
H23B	0.220107	0.510052	0.647517	0.133*	0.728 (10)
C24A	0.2730 (6)	0.4335 (4)	0.6935 (3)	0.1126 (18)	0.728 (10)
H24A	0.312204	0.441235	0.724285	0.135*	0.728 (10)
H24B	0.225149	0.428964	0.714941	0.135*	0.728 (10)
C25A	0.2890 (7)	0.3727 (3)	0.6577 (3)	0.106 (2)	0.728 (10)
H25A	0.340181	0.357361	0.665331	0.127*	0.728 (10)
H25B	0.254576	0.336160	0.669033	0.127*	0.728 (10)
C22B	0.2731 (16)	0.4602 (4)	0.6075 (7)	0.109 (2)	0.272 (10)
H22C	0.245119	0.482686	0.574358	0.130*	0.272 (10)
H22D	0.322119	0.481747	0.611478	0.130*	0.272 (10)
C23B	0.2309 (12)	0.4625 (9)	0.6675 (6)	0.110 (2)	0.272 (10)
H23C	0.242202	0.504061	0.690530	0.132*	0.272 (10)
H23D	0.176983	0.459786	0.660516	0.132*	0.272 (10)
C24B	0.2587 (13)	0.4019 (10)	0.7003 (4)	0.112 (2)	0.272 (10)
H24C	0.298095	0.414304	0.729603	0.134*	0.272 (10)
H24D	0.218167	0.379714	0.722977	0.134*	0.272 (10)
C25B	0.288 (2)	0.3567 (8)	0.6521 (5)	0.109 (2)	0.272 (10)
H25C	0.340960	0.346758	0.659833	0.131*	0.272 (10)
H25D	0.260468	0.313870	0.651645	0.131*	0.272 (10)
O2	0.4944 (15)	0.4366 (7)	0.7338 (7)	0.117 (4)*	0.25
C26	0.5447 (17)	0.4858 (13)	0.7042 (12)	0.117 (4)*	0.25
H26A	0.591402	0.463647	0.692107	0.140*	0.25
H26B	0.521243	0.504693	0.667069	0.140*	0.25
C27	0.5579 (16)	0.5336 (13)	0.7438 (12)	0.117 (4)*	0.25
H27A	0.611622	0.533295	0.752104	0.140*	0.25
H27B	0.546568	0.576770	0.723475	0.140*	0.25
C28	0.5242 (18)	0.5339 (18)	0.7967 (16)	0.117 (4)*	0.25
H28A	0.476639	0.558394	0.796452	0.140*	0.25
H28B	0.555831	0.548865	0.831052	0.140*	0.25
C29	0.5172 (18)	0.4687 (17)	0.7928 (13)	0.117 (4)*	0.25
H29A	0.480925	0.454844	0.824208	0.140*	0.25
H29B	0.565237	0.448766	0.804621	0.140*	0.25

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sc1	0.0237 (4)	0.0164 (3)	0.0194 (3)	0.0002 (3)	0.000	0.000
K1	0.0370 (6)	0.0315 (5)	0.0918 (9)	0.0156 (4)	0.000	0.000
C1	0.0283 (19)	0.0198 (18)	0.0238 (19)	−0.0065 (15)	0.000	0.000
C2	0.0280 (14)	0.0294 (14)	0.0231 (14)	−0.0104 (12)	0.0016 (11)	−0.0036 (11)
C3	0.0223 (13)	0.0257 (14)	0.0398 (15)	−0.0040 (11)	0.0071 (12)	−0.0079 (12)
C4	0.0434 (18)	0.058 (2)	0.0221 (15)	−0.0269 (16)	0.0007 (13)	−0.0008 (14)
C5A	0.045 (4)	0.032 (4)	0.0274 (19)	−0.013 (3)	0.009 (3)	−0.006 (3)
C6A	0.072 (5)	0.053 (4)	0.022 (2)	−0.020 (3)	−0.002 (3)	−0.004 (3)
C7A	0.086 (7)	0.063 (6)	0.024 (2)	−0.022 (4)	0.000 (3)	0.010 (4)
C8A	0.085 (12)	0.062 (11)	0.038 (5)	−0.016 (8)	−0.001 (6)	0.025 (9)
C5B	0.045 (4)	0.032 (4)	0.0274 (19)	−0.013 (3)	0.009 (3)	−0.006 (3)
C6B	0.072 (5)	0.053 (4)	0.022 (2)	−0.020 (3)	−0.002 (3)	−0.004 (3)
C7B	0.086 (7)	0.063 (6)	0.024 (2)	−0.022 (4)	0.000 (3)	0.010 (4)
C8B	0.085 (12)	0.062 (11)	0.038 (5)	−0.016 (8)	−0.001 (6)	0.025 (9)
C9	0.068 (2)	0.059 (2)	0.0314 (18)	−0.0211 (19)	−0.0033 (17)	0.0140 (16)
C10	0.0351 (19)	0.0310 (17)	0.200 (7)	0.0096 (15)	−0.019 (2)	−0.013 (2)
C11	0.043 (2)	0.0345 (18)	0.110 (3)	0.0169 (16)	−0.027 (2)	−0.018 (2)
C12	0.0345 (16)	0.0214 (14)	0.070 (2)	0.0133 (13)	−0.0111 (15)	−0.0022 (14)
C13	0.0489 (19)	0.0295 (15)	0.0367 (17)	0.0095 (14)	−0.0101 (14)	0.0042 (13)
C14	0.0392 (17)	0.0275 (15)	0.0472 (18)	0.0093 (13)	−0.0042 (14)	0.0056 (13)
C15	0.058 (2)	0.0352 (18)	0.066 (2)	0.0059 (16)	−0.0046 (18)	0.0183 (17)
C16	0.064 (2)	0.0275 (16)	0.103 (3)	−0.0023 (17)	0.002 (2)	0.0148 (18)
C19	0.0266 (14)	0.0189 (12)	0.0427 (17)	−0.0016 (11)	0.0064 (12)	−0.0018 (12)
C17	0.048 (3)	0.024 (3)	0.014 (3)	0.002 (2)	0.005 (2)	−0.003 (2)
C18	0.036 (3)	0.018 (2)	0.017 (3)	0.005 (2)	0.010 (2)	0.001 (2)
C20	0.0228 (13)	0.0165 (12)	0.056 (2)	0.0020 (11)	0.0017 (13)	−0.0047 (13)
C21	0.0179 (18)	0.0119 (16)	0.057 (3)	−0.0019 (14)	0.000	0.000
O1	0.0724 (19)	0.0558 (16)	0.101 (2)	0.0085 (14)	−0.0261 (17)	−0.0044 (16)
C22A	0.164 (5)	0.053 (3)	0.092 (4)	−0.011 (3)	−0.061 (4)	−0.002 (3)
C23A	0.178 (5)	0.059 (3)	0.095 (3)	−0.006 (3)	−0.065 (3)	−0.003 (3)
C24A	0.177 (4)	0.062 (3)	0.099 (3)	−0.010 (3)	−0.068 (3)	−0.005 (2)
C25A	0.160 (4)	0.058 (3)	0.099 (3)	−0.006 (4)	−0.078 (3)	−0.001 (3)
C22B	0.172 (5)	0.059 (3)	0.095 (4)	−0.010 (3)	−0.068 (4)	−0.005 (3)
C23B	0.172 (5)	0.061 (4)	0.097 (4)	−0.008 (4)	−0.071 (4)	−0.009 (3)
C24B	0.171 (5)	0.061 (4)	0.102 (4)	−0.009 (4)	−0.071 (4)	−0.003 (3)
C25B	0.166 (5)	0.062 (4)	0.101 (4)	−0.006 (4)	−0.074 (4)	−0.002 (3)

Geometric parameters (Å, º)

Sc1—C1	2.522 (4)	C5B—C6B	1.401 (11)
Sc1—C2	2.512 (3)	C5B—H5B	0.9500
Sc1—C2^{i}	2.512 (3)	C6B—C7B	1.365 (12)
Sc1—C3	2.470 (3)	C6B—H6B	0.9500
Sc1—C3^{i}	2.470 (3)	C7B—C8B	1.32 (3)
Sc1—C12	2.616 (3)	C7B—H7B	0.9500

Sc1—C12^{i}	2.616 (3)	C8B—C9	1.41 (3)
Sc1—C13^{i}	2.348 (3)	C8B—H8B	0.9500
Sc1—C13	2.348 (3)	C9—H9A	0.9500
Sc1—C14^{i}	2.874 (3)	C10—H10	0.9500
Sc1—C14	2.874 (3)	C11—H11	1.0000
Sc1—C20	2.524 (3)	C13—H13	1.0000
Sc1—C20^{i}	2.524 (3)	C15—H15	0.9500
Sc1—C21	2.582 (3)	C16—H16	0.9500
K1—O1	2.605 (3)	C19—H19	0.9500
K1—C1^{ii}	3.079 (4)	C17—C17^{vi}	1.674 (10)
K1—C2^{ii}	3.106 (3)	C17—C17^{vii}	1.788 (11)
K1—C3^{ii}	3.138 (3)	C17—H17	0.9598
K1—C11	3.431 (3)	C18—H18	0.9500
K1—C12	3.020 (3)	C20—H20	0.9500
K1—C14	3.404 (3)	O1—C25A	1.397 (6)
C1—C2	1.411 (3)	O1—C25B	1.400 (8)
C2—C3	1.418 (4)	O1—C22A	1.410 (5)
C2—C4	1.478 (4)	O1—C22B	1.419 (8)
C3—C3^{i}	1.400 (6)	C22A—C23A	1.498 (7)
C10—C10^{i}	1.362 (10)	C22A—H22A	0.9900
C10—C11	1.385 (6)	C22A—H22B	0.9900
C11—C12	1.415 (5)	C23A—C24A	1.491 (7)
C12—C12^{i}	1.428 (7)	C23A—H23A	0.9900
C12—C13	1.450 (5)	C23A—H23B	0.9900
C13—C14	1.474 (4)	C24A—C25A	1.470 (7)
C14—C15	1.397 (4)	C24A—H24A	0.9900
C14—C14^{i}	1.418 (6)	C24A—H24B	0.9900
C15—C16	1.383 (5)	C25A—H25A	0.9900
C16—C16^{i}	1.380 (8)	C25A—H25B	0.9900
C19—C18	1.281 (5)	C22B—C23B	1.518 (9)
C19—C19^{iii}	1.393 (5)	C22B—H22C	0.9900
C19—C20	1.438 (4)	C22B—H22D	0.9900
C17—C17^{iii}	1.390 (11)	C23B—C24B	1.493 (8)
C17—C18	1.371 (7)	C23B—H23C	0.9900
C20—C21	1.411 (3)	C23B—H23D	0.9900
C21—C21^{iv}	1.466 (7)	C24B—C25B	1.484 (8)
K1—O1^{i}	2.605 (3)	C24B—H24C	0.9900
K1—C12^{i}	3.020 (3)	C24B—H24D	0.9900
K1—C2^{v}	3.106 (3)	C25B—H25C	0.9900
K1—C3^{v}	3.138 (3)	C25B—H25D	0.9900
K1—C14^{i}	3.404 (3)	O2—C26	1.49 (3)
K1—C11^{i}	3.431 (3)	O2—C29	1.50 (3)
C1—C2^{i}	1.411 (3)	C26—C27	1.309 (17)
C1—H1	1.0000	C26—C29	2.03 (4)
C3—H3	1.0000	C26—H26A	0.9900
C4—C5B	1.334 (9)	C26—H26B	0.9900
C4—C9	1.381 (5)	C27—C28	1.306 (17)
C4—C5A	1.518 (8)	C27—C29	1.83 (5)

C5A—C6A	1.375 (11)	C27—H27A	0.9900
C5A—H5A	0.9500	C27—H27B	0.9900
C6A—C7A	1.379 (12)	C28—C29	1.308 (17)
C6A—H6A	0.9500	C28—H28A	0.9900
C7A—C8A	1.43 (3)	C28—H28B	0.9900
C7A—H7A	0.9500	C29—H29A	0.9900
C8A—C9	1.41 (3)	C29—H29B	0.9900
C8A—H8A	0.9500		

C13^{i}—Sc1—C13	70.83 (16)	C6A—C5A—C4	116.7 (6)
C13^{i}—Sc1—C3^{i}	119.60 (11)	C6A—C5A—H5A	121.7
C13—Sc1—C3^{i}	145.34 (10)	C4—C5A—H5A	121.7
C13^{i}—Sc1—C3	145.34 (10)	C5A—C6A—C7A	121.7 (7)
C13—Sc1—C3	119.60 (11)	C5A—C6A—H6A	119.1
C3^{i}—Sc1—C3	32.93 (13)	C7A—C6A—H6A	119.1
C13^{i}—Sc1—C2^{i}	91.39 (11)	C6A—C7A—C8A	118.8 (13)
C13—Sc1—C2^{i}	123.71 (10)	C6A—C7A—H7A	120.6
C3^{i}—Sc1—C2^{i}	33.06 (9)	C8A—C7A—H7A	120.6
C3—Sc1—C2^{i}	54.66 (9)	C9—C8A—C7A	124.7 (18)
C13^{i}—Sc1—C2	123.71 (10)	C9—C8A—H8A	117.6
C13—Sc1—C2	91.39 (11)	C7A—C8A—H8A	117.6
C3^{i}—Sc1—C2	54.66 (9)	C4—C5B—C6B	125.8 (7)
C3—Sc1—C2	33.06 (9)	C4—C5B—H5B	117.1
C2^{i}—Sc1—C2	54.50 (12)	C6B—C5B—H5B	117.1
C13^{i}—Sc1—C1	94.07 (10)	C7B—C6B—C5B	119.0 (7)
C13—Sc1—C1	94.07 (10)	C7B—C6B—H6B	120.5
C3^{i}—Sc1—C1	54.17 (10)	C5B—C6B—H6B	120.5
C3—Sc1—C1	54.17 (10)	C8B—C7B—C6B	120.1 (14)
C2^{i}—Sc1—C1	32.57 (7)	C8B—C7B—H7B	119.9
C2—Sc1—C1	32.57 (7)	C6B—C7B—H7B	119.9
C13^{i}—Sc1—C20	94.09 (10)	C7B—C8B—C9	116.2 (17)
C13—Sc1—C20	129.76 (11)	C7B—C8B—H8B	121.9
C3^{i}—Sc1—C20	84.20 (9)	C9—C8B—H8B	121.9
C3—Sc1—C20	100.19 (9)	C4—C9—C8A	114.3 (10)
C2^{i}—Sc1—C20	103.62 (9)	C4—C9—C8B	128.1 (12)
C2—Sc1—C20	133.20 (9)	C4—C9—H9A	122.9
C1—Sc1—C20	135.54 (9)	C8A—C9—H9A	122.9
C13^{i}—Sc1—C20^{i}	129.76 (11)	C10^{i}—C10—C11	120.6 (3)
C13—Sc1—C20^{i}	94.09 (10)	C10^{i}—C10—H10	119.7
C3^{i}—Sc1—C20^{i}	100.19 (9)	C11—C10—H10	119.7
C3—Sc1—C20^{i}	84.20 (9)	C10—C11—C12	121.0 (4)
C2^{i}—Sc1—C20^{i}	133.19 (9)	C10—C11—K1	96.0 (2)
C2—Sc1—C20^{i}	103.62 (9)	C12—C11—K1	61.35 (16)
C1—Sc1—C20^{i}	135.54 (9)	C10—C11—H11	119.2
C20—Sc1—C20^{i}	58.72 (13)	C12—C11—H11	119.2
C13^{i}—Sc1—C21	124.11 (10)	K1—C11—H11	119.2
C13—Sc1—C21	124.11 (10)	C11—C12—C12^{i}	118.4 (3)
C3^{i}—Sc1—C21	79.59 (10)	C11—C12—C13	125.1 (4)

C3—Sc1—C21	79.59 (10)	C12^{i}—C12—C13	116.48 (18)
C2^{i}—Sc1—C21	110.29 (9)	C11—C12—Sc1	131.5 (2)
C2—Sc1—C21	110.29 (9)	C12^{i}—C12—Sc1	74.16 (8)
C1—Sc1—C21	131.52 (12)	C13—C12—Sc1	63.10 (15)
C20—Sc1—C21	32.05 (7)	C11—C12—K1	94.37 (19)
C20^{i}—Sc1—C21	32.04 (7)	C12^{i}—C12—K1	76.33 (7)
C13^{i}—Sc1—C12	58.78 (11)	C13—C12—K1	100.66 (17)
C13—Sc1—C12	33.42 (11)	Sc1—C12—K1	133.33 (12)
C3^{i}—Sc1—C12	177.74 (9)	C12—C13—C14	115.2 (3)
C3—Sc1—C12	147.62 (10)	C12—C13—Sc1	83.48 (17)
C2^{i}—Sc1—C12	144.71 (9)	C14—C13—Sc1	94.77 (18)
C2—Sc1—C12	124.49 (10)	C12—C13—H13	118.5
C1—Sc1—C12	123.83 (11)	C14—C13—H13	118.5
C20—Sc1—C12	97.40 (10)	Sc1—C13—H13	118.5
C20^{i}—Sc1—C12	82.02 (9)	C15—C14—C14^{i}	118.8 (2)
C21—Sc1—C12	102.61 (10)	C15—C14—C13	124.9 (3)
C13^{i}—Sc1—C12^{i}	33.42 (11)	C14^{i}—C14—C13	116.21 (17)
C13—Sc1—C12^{i}	58.78 (11)	C15—C14—Sc1	139.6 (2)
C3^{i}—Sc1—C12^{i}	147.62 (10)	C14^{i}—C14—Sc1	75.71 (6)
C3—Sc1—C12^{i}	177.74 (9)	C13—C14—Sc1	54.49 (14)
C2^{i}—Sc1—C12^{i}	124.49 (10)	C15—C14—K1	109.1 (2)
C2—Sc1—C12^{i}	144.71 (9)	C14^{i}—C14—K1	77.97 (5)
C1—Sc1—C12^{i}	123.83 (11)	C13—C14—K1	84.75 (18)
C20—Sc1—C12^{i}	82.02 (9)	Sc1—C14—K1	110.82 (10)
C20^{i}—Sc1—C12^{i}	97.40 (10)	C16—C15—C14	121.2 (4)
C21—Sc1—C12^{i}	102.61 (10)	C16—C15—H15	119.4
C12—Sc1—C12^{i}	31.68 (15)	C14—C15—H15	119.4
C13^{i}—Sc1—C14^{i}	30.74 (10)	C16^{i}—C16—C15	120.0 (2)
C13—Sc1—C14^{i}	55.01 (10)	C16^{i}—C16—H16	120.0
C3^{i}—Sc1—C14^{i}	115.43 (9)	C15—C16—H16	120.0
C3—Sc1—C14^{i}	124.70 (9)	C18—C19—C19^{iii}	122.4 (3)
C2^{i}—Sc1—C14^{i}	82.36 (9)	C18—C19—C20	118.0 (3)
C2—Sc1—C14^{i}	95.34 (9)	C19^{iii}—C19—C20	119.62 (15)
C1—Sc1—C14^{i}	70.76 (10)	C19^{iii}—C19—H19	120.2
C20—Sc1—C14^{i}	124.76 (9)	C20—C19—H19	120.2
C20^{i}—Sc1—C14^{i}	144.30 (9)	C18—C17—C17^{iii}	120.2 (3)
C21—Sc1—C14^{i}	154.27 (9)	C18—C17—C17^{vi}	155.1 (4)
C12—Sc1—C14^{i}	62.35 (9)	C17^{iii}—C17—C17^{vi}	70.7 (4)
C12^{i}—Sc1—C14^{i}	53.22 (9)	C18—C17—C17^{vii}	157.0 (4)
C13^{i}—Sc1—C14	55.01 (10)	C17^{iii}—C17—C17^{vii}	62.1 (4)
C13—Sc1—C14	30.74 (10)	C17^{vi}—C17—C17^{vii}	47.2 (4)
C3^{i}—Sc1—C14	124.70 (9)	C18—C17—H17	119.0
C3—Sc1—C14	115.43 (9)	C17^{iii}—C17—H17	120.8
C2^{i}—Sc1—C14	95.34 (9)	C17^{vi}—C17—H17	55.6
C2—Sc1—C14	82.36 (9)	C17^{vii}—C17—H17	63.7
C1—Sc1—C14	70.76 (10)	C19—C18—C17	117.3 (5)
C20—Sc1—C14	144.30 (9)	C19—C18—H18	121.3
C20^{i}—Sc1—C14	124.76 (9)	C17—C18—H18	121.3

C21—Sc1—C14	154.27 (9)	C21—C20—C19	121.7 (2)
C12—Sc1—C14	53.22 (9)	C21—C20—Sc1	76.25 (18)
C12^{i}—Sc1—C14	62.35 (9)	C19—C20—Sc1	127.26 (18)
C14^{i}—Sc1—C14	28.57 (13)	C21—C20—H20	119.2
O1—K1—O1^{i}	107.90 (14)	C19—C20—H20	119.2
O1—K1—C12	125.38 (9)	Sc1—C20—H20	67.8
O1^{i}—K1—C12	101.34 (9)	C20^{i}—C21—C20	122.6 (3)
O1—K1—C12^{i}	101.34 (9)	C20^{i}—C21—C21^{iv}	118.41 (17)
O1^{i}—K1—C12^{i}	125.38 (9)	C20—C21—C21^{iv}	118.41 (17)
C12—K1—C12^{i}	27.35 (13)	C20^{i}—C21—Sc1	71.71 (17)
O1—K1—C1^{ii}	106.26 (8)	C20—C21—Sc1	71.70 (17)
O1^{i}—K1—C1^{ii}	106.26 (8)	C21^{iv}—C21—Sc1	123.3 (3)
C12—K1—C1^{ii}	108.33 (9)	C25A—O1—C22A	110.0 (4)
C12^{i}—K1—C1^{ii}	108.33 (9)	C25B—O1—C22B	109.2 (7)
O1—K1—C2^{ii}	115.04 (8)	C25A—O1—K1	130.4 (3)
O1^{i}—K1—C2^{ii}	79.89 (9)	C25B—O1—K1	116.4 (8)
C12—K1—C2^{ii}	114.82 (8)	C22A—O1—K1	119.2 (3)
C12^{i}—K1—C2^{ii}	126.50 (9)	C22B—O1—K1	133.6 (8)
C1^{ii}—K1—C2^{ii}	26.38 (6)	O1—C22A—C23A	105.1 (4)
O1—K1—C2^{v}	79.89 (9)	O1—C22A—K1	40.3 (3)
O1^{i}—K1—C2^{v}	115.04 (8)	C23A—C22A—K1	141.3 (5)
C12—K1—C2^{v}	126.50 (9)	O1—C22A—H22A	110.7
C12^{i}—K1—C2^{v}	114.82 (8)	C23A—C22A—H22A	110.7
C1^{ii}—K1—C2^{v}	26.38 (6)	K1—C22A—H22A	78.4
C2^{ii}—K1—C2^{v}	43.47 (10)	O1—C22A—H22B	110.7
O1—K1—C3^{ii}	93.03 (8)	C23A—C22A—H22B	110.7
O1^{i}—K1—C3^{ii}	72.06 (8)	K1—C22A—H22B	100.6
C12—K1—C3^{ii}	140.24 (8)	H22A—C22A—H22B	108.8
C12^{i}—K1—C3^{ii}	150.95 (9)	C24A—C23A—C22A	104.9 (4)
C1^{ii}—K1—C3^{ii}	42.88 (8)	C24A—C23A—H23A	110.8
C2^{ii}—K1—C3^{ii}	26.25 (7)	C22A—C23A—H23A	110.8
C2^{v}—K1—C3^{ii}	42.98 (7)	C24A—C23A—H23B	110.8
O1—K1—C3^{v}	72.06 (8)	C22A—C23A—H23B	110.8
O1^{i}—K1—C3^{v}	93.03 (8)	H23A—C23A—H23B	108.8
C12—K1—C3^{v}	150.95 (9)	C25A—C24A—C23A	106.6 (4)
C12^{i}—K1—C3^{v}	140.24 (8)	C25A—C24A—H24A	110.4
C1^{ii}—K1—C3^{v}	42.88 (8)	C23A—C24A—H24A	110.4
C2^{ii}—K1—C3^{v}	42.98 (7)	C25A—C24A—H24B	110.4
C2^{v}—K1—C3^{v}	26.25 (7)	C23A—C24A—H24B	110.4
C3^{ii}—K1—C3^{v}	25.78 (10)	H24A—C24A—H24B	108.6
O1—K1—C14^{i}	78.43 (8)	O1—C25A—C24A	106.3 (4)
O1^{i}—K1—C14^{i}	97.83 (9)	O1—C25A—H25A	110.5
C12—K1—C14^{i}	52.27 (8)	C24A—C25A—H25A	110.5
C12^{i}—K1—C14^{i}	44.72 (8)	O1—C25A—H25B	110.5
C1^{ii}—K1—C14^{i}	152.24 (8)	C24A—C25A—H25B	110.5
C2^{ii}—K1—C14^{i}	166.45 (7)	H25A—C25A—H25B	108.7
C2^{v}—K1—C14^{i}	144.87 (7)	O1—C22B—C23B	102.8 (7)
C3^{ii}—K1—C14^{i}	164.31 (8)	O1—C22B—H22C	111.2

C3^{v}—K1—C14^{i}	150.43 (7)	C23B—C22B—H22C	111.2
O1—K1—C14	97.84 (9)	O1—C22B—H22D	111.2
O1^{i}—K1—C14	78.43 (8)	C23B—C22B—H22D	111.2
C12—K1—C14	44.72 (8)	H22C—C22B—H22D	109.1
C12^{i}—K1—C14	52.27 (8)	C24B—C23B—C22B	102.8 (6)
C1^{ii}—K1—C14	152.24 (8)	C24B—C23B—H23C	111.2
C2^{ii}—K1—C14	144.87 (7)	C22B—C23B—H23C	111.2
C2^{v}—K1—C14	166.45 (7)	C24B—C23B—H23D	111.2
C3^{ii}—K1—C14	150.43 (7)	C22B—C23B—H23D	111.2
C3^{v}—K1—C14	164.31 (8)	H23C—C23B—H23D	109.1
C14^{i}—K1—C14	24.05 (11)	C25B—C24B—C23B	105.7 (5)
O1—K1—C11	144.95 (10)	C25B—C24B—H24C	110.6
O1^{i}—K1—C11	99.52 (10)	C23B—C24B—H24C	110.6
C12—K1—C11	24.28 (8)	C25B—C24B—H24D	110.6
C12^{i}—K1—C11	43.90 (9)	C23B—C24B—H24D	110.6
C1^{ii}—K1—C11	85.91 (9)	H24C—C24B—H24D	108.7
C2^{ii}—K1—C11	90.59 (8)	O1—C25B—C24B	106.8 (5)
C2^{v}—K1—C11	108.03 (9)	O1—C25B—K1	42.4 (6)
C3^{ii}—K1—C11	116.35 (8)	C24B—C25B—K1	140.7 (12)
C3^{v}—K1—C11	128.62 (8)	O1—C25B—H25C	110.4
C14^{i}—K1—C11	76.55 (8)	C24B—C25B—H25C	110.4
C14—K1—C11	66.36 (8)	K1—C25B—H25C	104.7
O1—K1—C11^{i}	99.52 (10)	O1—C25B—H25D	110.4
O1^{i}—K1—C11^{i}	144.95 (10)	C24B—C25B—H25D	110.4
C12—K1—C11^{i}	43.90 (9)	K1—C25B—H25D	73.2
C12^{i}—K1—C11^{i}	24.28 (8)	H25C—C25B—H25D	108.6
C1^{ii}—K1—C11^{i}	85.91 (9)	C26—O2—C29	85.7 (19)
C2^{ii}—K1—C11^{i}	108.03 (9)	C27—C26—O2	108 (2)
C2^{v}—K1—C11^{i}	90.59 (8)	C27—C26—C29	62.3 (19)
C3^{ii}—K1—C11^{i}	128.62 (8)	O2—C26—C29	47.4 (10)
C3^{v}—K1—C11^{i}	116.35 (8)	C27—C26—H26A	110.2
C14^{i}—K1—C11^{i}	66.36 (8)	O2—C26—H26A	110.2
C14—K1—C11^{i}	76.55 (8)	C29—C26—H26A	112.9
C11—K1—C11^{i}	47.65 (16)	C27—C26—H26B	110.2
C2—C1—C2^{i}	109.2 (3)	O2—C26—H26B	110.2
C2—C1—Sc1	73.32 (17)	C29—C26—H26B	137.9
C2^{i}—C1—Sc1	73.32 (17)	H26A—C26—H26B	108.5
C2—C1—K1^{viii}	77.86 (19)	C28—C27—C26	120 (3)
C2^{i}—C1—K1^{viii}	77.86 (19)	C28—C27—C29	45.6 (16)
Sc1—C1—K1^{viii}	129.04 (14)	C26—C27—C29	79 (2)
C2—C1—H1	125.2	C28—C27—H27A	107.2
C2^{i}—C1—H1	125.2	C26—C27—H27A	107.2
Sc1—C1—H1	125.2	C29—C27—H27A	106.5
K1^{viii}—C1—H1	105.7	C28—C27—H27B	107.2
C1—C2—C3	106.9 (2)	C26—C27—H27B	107.2
C1—C2—C4	127.2 (3)	C29—C27—H27B	142.4
C3—C2—C4	125.8 (3)	H27A—C27—H27B	106.9
C1—C2—Sc1	74.11 (18)	C27—C28—C29	89 (3)

C3—C2—Sc1	71.86 (15)	C27—C28—H28A	113.8
C4—C2—Sc1	121.76 (19)	C29—C28—H28A	113.8
C1—C2—K1^{viii}	75.77 (18)	C27—C28—H28B	113.8
C3—C2—K1^{viii}	78.15 (16)	C29—C28—H28B	113.8
C4—C2—K1^{viii}	109.93 (18)	H28A—C28—H28B	111.0
Sc1—C2—K1^{viii}	128.30 (10)	C28—C29—O2	120 (3)
C3^{i}—C3—C2	108.50 (15)	C28—C29—C27	45.5 (17)
C3^{i}—C3—Sc1	73.53 (7)	O2—C29—C27	84.7 (18)
C2—C3—Sc1	75.08 (16)	C28—C29—C26	83 (2)
C3^{i}—C3—K1^{viii}	77.11 (5)	O2—C29—C26	46.9 (10)
C2—C3—K1^{viii}	75.60 (15)	C27—C29—C26	39.2 (11)
Sc1—C3—K1^{viii}	128.61 (10)	C28—C29—H29A	107.2
C3^{i}—C3—H3	125.4	O2—C29—H29A	107.2
C2—C3—H3	125.4	C27—C29—H29A	150.3
Sc1—C3—H3	125.4	C26—C29—H29A	150.7
K1^{viii}—C3—H3	106.0	C28—C29—H29B	107.2
C5B—C4—C9	109.8 (4)	O2—C29—H29B	107.2
C5B—C4—C2	127.8 (4)	C27—C29—H29B	94.8
C9—C4—C2	121.2 (3)	C26—C29—H29B	95.8
C9—C4—C5A	123.0 (4)	H29A—C29—H29B	106.8
C2—C4—C5A	113.7 (4)		

C2^{i}—C1—C2—C3	0.2 (4)	C11—C12—C13—Sc1	−123.5 (3)
Sc1—C1—C2—C3	−64.85 (19)	C12^{i}—C12—C13—Sc1	53.51 (12)
K1^{viii}—C1—C2—C3	72.63 (19)	K1—C12—C13—Sc1	133.43 (11)
C2^{i}—C1—C2—C4	−176.80 (19)	C12—C13—C14—C15	−145.0 (3)
Sc1—C1—C2—C4	118.1 (3)	Sc1—C13—C14—C15	130.0 (3)
K1^{viii}—C1—C2—C4	−104.4 (3)	C12—C13—C14—C14^{i}	38.6 (3)
C2^{i}—C1—C2—Sc1	65.1 (3)	Sc1—C13—C14—C14^{i}	−46.43 (16)
K1^{viii}—C1—C2—Sc1	137.48 (8)	C12—C13—C14—Sc1	85.0 (2)
C2^{i}—C1—C2—K1^{viii}	−72.4 (3)	C12—C13—C14—K1	−35.2 (2)
Sc1—C1—C2—K1^{viii}	−137.48 (8)	Sc1—C13—C14—K1	−120.26 (11)
C1—C2—C3—C3^{i}	−0.1 (2)	C14^{i}—C14—C15—C16	1.9 (4)
C4—C2—C3—C3^{i}	177.0 (2)	C13—C14—C15—C16	−174.4 (3)
Sc1—C2—C3—C3^{i}	−66.52 (7)	Sc1—C14—C15—C16	−100.2 (4)
K1^{viii}—C2—C3—C3^{i}	70.80 (7)	K1—C14—C15—C16	88.4 (4)
C1—C2—C3—Sc1	66.4 (2)	C14—C15—C16—C16^{i}	−1.9 (4)
C4—C2—C3—Sc1	−116.5 (3)	C19^{iii}—C19—C18—C17	3.4 (7)
K1^{viii}—C2—C3—Sc1	137.32 (8)	C20—C19—C18—C17	−178.0 (4)
C1—C2—C3—K1^{viii}	−70.9 (2)	C17^{iii}—C17—C18—C19	−0.2 (9)
C4—C2—C3—K1^{viii}	106.2 (3)	C17^{vi}—C17—C18—C19	−110.4 (12)
Sc1—C2—C3—K1^{viii}	−137.32 (8)	C17^{vii}—C17—C18—C19	88.9 (13)
C1—C2—C4—C5B	−145.3 (6)	C18—C19—C20—C21	−174.5 (4)
C3—C2—C4—C5B	38.2 (7)	C19^{iii}—C19—C20—C21	4.2 (5)
Sc1—C2—C4—C5B	−51.5 (6)	C18—C19—C20—Sc1	88.7 (4)
K1^{viii}—C2—C4—C5B	127.7 (5)	C19^{iii}—C19—C20—Sc1	−92.6 (3)
C1—C2—C4—C9	21.3 (5)	C19—C20—C21—C20^{i}	−178.4 (2)
C3—C2—C4—C9	−155.3 (3)	Sc1—C20—C21—C20^{i}	−52.8 (3)

Sc1—C2—C4—C9	115.1 (3)	C19—C20—C21—C21^{iv}	−7.0 (5)
K1^{viii}—C2—C4—C9	−65.7 (4)	Sc1—C20—C21—C21^{iv}	118.6 (4)
C1—C2—C4—C5A	−174.7 (4)	C19—C20—C21—Sc1	−125.5 (3)
C3—C2—C4—C5A	8.8 (5)	C25A—O1—C22A—C23A	−27.5 (10)
Sc1—C2—C4—C5A	−80.9 (4)	K1—O1—C22A—C23A	158.5 (5)
K1^{viii}—C2—C4—C5A	98.3 (4)	C25A—O1—C22A—K1	174.0 (8)
C9—C4—C5A—C6A	−9.7 (9)	O1—C22A—C23A—C24A	18.4 (10)
C2—C4—C5A—C6A	−173.4 (6)	K1—C22A—C23A—C24A	40.7 (12)
C4—C5A—C6A—C7A	5.9 (12)	C22A—C23A—C24A—C25A	−4.0 (11)
C5A—C6A—C7A—C8A	1.9 (19)	C22A—O1—C25A—C24A	25.0 (10)
C6A—C7A—C8A—C9	−7 (3)	K1—O1—C25A—C24A	−161.8 (5)
C9—C4—C5B—C6B	10.8 (9)	C23A—C24A—C25A—O1	−12.0 (11)
C2—C4—C5B—C6B	178.6 (6)	C25B—O1—C22B—C23B	−35 (2)
C4—C5B—C6B—C7B	−8.7 (13)	K1—O1—C22B—C23B	134.7 (10)
C5B—C6B—C7B—C8B	−1.9 (19)	O1—C22B—C23B—C24B	33.8 (18)
C6B—C7B—C8B—C9	8 (3)	C22B—C23B—C24B—C25B	−22 (2)
C2—C4—C9—C8A	167.2 (13)	C22B—O1—C25B—C24B	21 (3)
C5A—C4—C9—C8A	4.7 (14)	K1—O1—C25B—C24B	−150.3 (15)
C5B—C4—C9—C8B	−3.9 (14)	C22B—O1—C25B—K1	171.4 (17)
C2—C4—C9—C8B	−172.7 (13)	C23B—C24B—C25B—O1	2 (3)
C7A—C8A—C9—C4	4 (3)	C23B—C24B—C25B—K1	−30 (3)
C7B—C8B—C9—C4	−5 (3)	C29—O2—C26—C27	−17 (2)
C10^{i}—C10—C11—C12	2.3 (4)	O2—C26—C27—C28	−5 (4)
C10^{i}—C10—C11—K1	−57.75 (16)	C29—C26—C27—C28	−19 (3)
C10—C11—C12—C12^{i}	−2.2 (4)	O2—C26—C27—C29	14 (2)
K1—C11—C12—C12^{i}	76.81 (11)	C26—C27—C28—C29	26 (4)
C10—C11—C12—C13	174.7 (3)	C27—C28—C29—O2	−43 (4)
K1—C11—C12—C13	−106.3 (3)	C27—C28—C29—C26	−14 (2)
C10—C11—C12—Sc1	91.4 (4)	C26—O2—C29—C28	41 (4)
K1—C11—C12—Sc1	170.4 (4)	C26—O2—C29—C27	11.6 (16)
C10—C11—C12—K1	−79.0 (3)	C26—C27—C29—C28	−157 (4)
C11—C12—C13—C14	144.3 (3)	C28—C27—C29—O2	144 (4)
C12^{i}—C12—C13—C14	−38.7 (3)	C26—C27—C29—O2	−13 (2)
Sc1—C12—C13—C14	−92.2 (2)	C28—C27—C29—C26	157 (4)
K1—C12—C13—C14	41.2 (3)		

Symmetry codes: (i) *x*, *y*, −*z*+1; (ii) *x*−1/2, −*y*+1/2, *z*; (iii) −*x*+1, −*y*, *z*; (iv) −*x*+1, −*y*, −*z*+1; (v) *x*−1/2, −*y*+1/2, −*z*+1; (vi) *x*, −*y*, −*z*+3/2; (vii) −*x*+1, *y*, −*z*+3/2; (viii) *x*+1/2, −*y*+1/2, *z*.