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Dichloro(2,2′,4,4′,6,6′-hexamethyl-*m*-terphenyl)-bis(*N*-methylimidazole)(tetrahydrofuran)-samarium(III) toluene disolvate

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The molecular structure of [Sm($C_{24}H_{25}$)Cl_2($C_4H_6N_2$)$_2$(C_4H_8O)]·2C_7H_8, a novel Lewis base adduct of DmpSmCl_2 (Dmp = 2,6-dimesitylphenyl), is reported. It features a monomeric six-coordinate distorted octahedral arrangement (the *mer* isomer) about the metal atom, which is coordinated by two different Lewis bases. A weak hydrogen-bonding interaction is present between neighboring molecules. The molecule lies on a twofold rotation axis and the asymmetric unit also contains a molecule of toluene.

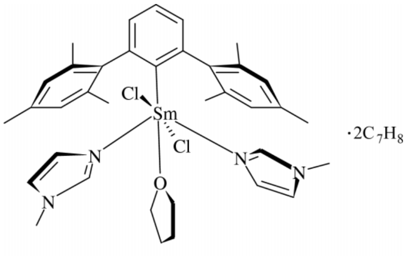
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.008 Å
R factor = 0.043
wR factor = 0.105
Data-to-parameter ratio = 17.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We previously reported the unexpected formation of a mixed Lewis base adduct of DmpYbCl_2 of composition DmpYbCl_2(N-MeIm)$_2$(py) (Rabe *et al.*, 1999), which was obtained from the reaction of equimolar amounts of DmpLi (Ruhlandt-Senge *et al.*, 1993) and YbCl_3 in tetrahydrofuran at room temperature, followed by extraction of the crude product with toluene and addition of small amounts of *N*-methylimidazole (*N*-MeIm), pyridine (py), and tetrahydrofuran. Interestingly, the molecular structure of the ytterbium compound shows the presence of two different Lewis bases at the six-coordinate metal atom, with the weaker base (pyridine) occupying the position *trans* to the Dmp ligand.



(I)

We were interested in investigating the influence of the size of the metal atom and used samarium instead of ytterbium. Again a six-coordinate compound, (I), with two different Lewis bases coordinating to the metal atom, is obtained under the same crystallization conditions. However, in the case of the samarium compound, the position *trans* to the terphenyl ligand is now occupied by a tetrahydrofuran molecule (Fig. 1), and not by a pyridine molecule, as was found in the case of the ytterbium compound. We note that in both compounds the ligand which is *trans* to the Dmp moiety shows an interatomic separation that is approximately 0.2 Å (Ln = Yb) or 0.1 Å (Ln = Sm) longer than the two Ln—N distances for the equatorial ligands. This observation can possibly be rationalized in terms

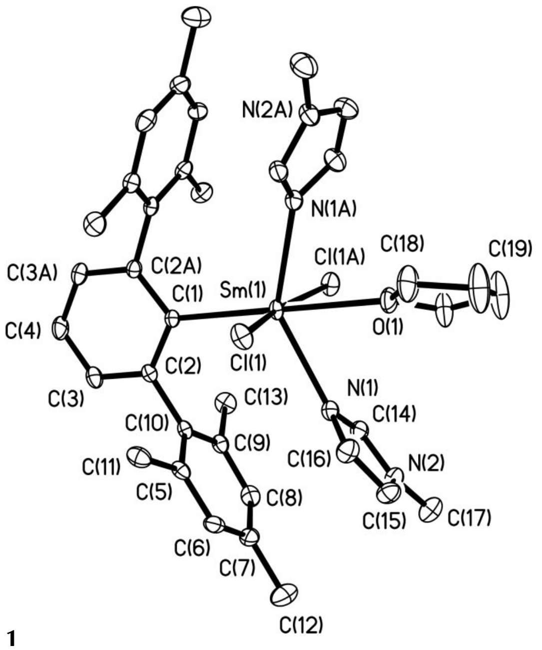


Figure 1
Molecular structure of the title compound, showing the atom labeling scheme. Ellipsoids are drawn at the 30% probability level. H atoms are omitted.

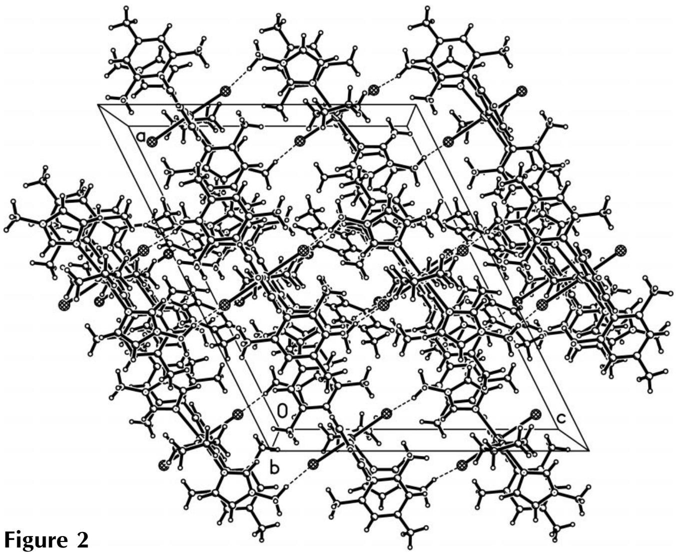


Figure 2
Packing diagram, showing the hydrogen-bonding interaction.

of a *trans*-directing influence of the anionic Dmp ligand. An alternative view is that the steric bulk of the terphenyl ligand and resultant bending back of the two *N*-MeIm ligands results in the 'backing off' of the *trans* thf or pyridine ligand. A similar observation was also previously made in the case of the six-coordinate $DmpYCl_2(thf)_3$ (Rabe *et al.*, 2002). Again the Y—O distance *trans* to the anionic Dmp ligand is approximately 0.2 Å longer than the other two Y—O distances.

A closer inspection of the packing diagram (Fig. 2) reveals the presence of an interaction between neighboring molecules through a chlorine atom and one of the H atoms of the methyl group of the N-MeIm ligand. The relevant data are: C17—H17*A* 0.96, H17*A*···Cl1^{i} 2.89, C17···Cl1^{i} 3.763 (6) Å, with C17—H17*A*···Cl1^{i} 152° [symmetry code: (i) $x, -y, \frac{1}{2} + z$].

Experimental

Crystalline $DmpSmCl_2$(*N*-MeIm)$_2$(thf)·2toluene was obtained from the reaction of equimolar amounts of DmpLi and $SmCl_3$ in tetrahydrofuran at room temperature, followed by extraction of the crude product with toluene and addition of small amounts of *N*-MeIm, pyridine, and tetrahydrofuran.

Crystal data

$[Sm(C_{24}H_{25})Cl_2(C_4H_6N_2)_2(C_4H_8O)]\cdot 2C_7H_8$
$M_r = 955.28$
Monoclinic, $C2/c$
$a = 21.5031$ (2) Å
$b = 13.9259$ (2) Å
$c = 17.6668$ (2) Å
$\beta = 116.5360$ (4)°
$V = 4733.01$ (10) Å^3
$Z = 4$
$D_x = 1.341$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7462 reflections
$\theta = 2.1$–28.2°
$\mu = 1.39$ mm^{-1}
$T = 173$ (2) K
Block, yellow
0.50 × 0.40 × 0.20 mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.543$, $T_{max} = 0.768$
10236 measured reflections
4686 independent reflections
4054 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 26.5$°
$h = -26 \rightarrow 23$
$k = -15 \rightarrow 17$
$l = -22 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.105$
$S = 1.21$
4686 reflections
267 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 28.1537P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.95$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sm1—N1	2.505 (4)	Sm1—O1	2.601 (4)
Sm1—C1	2.532 (5)	Sm1—Cl1	2.6754 (10)
N1^{ii}—Sm1—N1	140.56 (16)	C1—Sm1—O1	180
N1—Sm1—C1	109.72 (8)	N1—Sm1—Cl1^{ii}	85.48 (9)
N1—Sm1—O1	70.28 (8)	Cl1—Sm1—Cl1^{ii}	167.50 (5)

Symmetry code: (ii) $1 - x, y, \frac{3}{2} - z$.

All H atoms were placed in calculated positions and refined with riding model constraints, with U(H) set to 1.2 (1.5 for methyl groups) times U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (2001). *SMART, SAINT, SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Rabe, G. W.; Bérubé, C. D.; Yap, G. P. A.; Lam, K.-C., Concolino, T. E. & Rheingold, A. L. (2002). *Inorg. Chem.* **41**, 1446–1453.

Rabe, G. W.; Strissel, C. S.; Liable-Sands, L. M.; Concolino, T. E. & Rheingold, A. L. (1999). *Inorg. Chem.* **38**, 3446–3447.

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Ruhlandt-Senge, K., Ellison, J.; Wehmschulte, R. J., Pauer, F. & Power, P. P. (1993). *J. Am. Chem. Soc.* **115**, 11353–11357.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Acta Cryst. (2003). E**59**, m99–m101 Gerd W. Rabe *et al.* • $[Sm(C_{24}H_{25})Cl_2(C_4H_6N_2)_2(C_4H_8O)]\cdot 2C_7H_8$ **m101**