

# Mixed cyclopentadienyl—naphthalene complexes of rare-earth elements: mono- and binuclear derivatives.

## Molecular structure of $(\eta^5\text{-C}_5\text{H}_5)\text{YC}_{10}\text{H}_8(\text{DME})$

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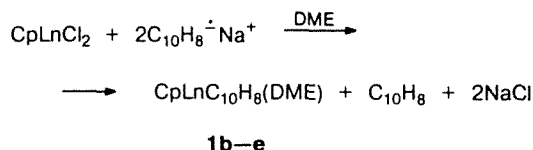
Reaction of  $\text{CpLnCl}_2(\text{THF})_3$  ( $\text{Ln} = \text{Y, Gd, Er, and Tm}$ ) with sodium naphthalenide in 1,2-dimethoxyethane (DME) gives mononuclear complexes  $\text{CpLnC}_{10}\text{H}_8(\text{DME})$ . Binuclear complexes  $(\text{CpLn})_2\text{C}_{10}\text{H}_8(\text{THF})_4$  ( $\text{Ln} = \text{Sm and Yb}$ ) containing the Ln atoms in the oxidation state +2 are formed in similar reactions of Sm and Yb complexes. The structure of  $\text{CpYC}_{10}\text{H}_8(\text{DME})$  was determined by the X-ray diffraction method. The coordinated naphthalene ring linked to the Y atom is nonplanar: it is bent by an angle  $26.1^\circ$  over the C(1)...C(4) line. The existence of two short, Y(1)—C(1) and Y(1)—C(4) (2.438(6) and 2.452(6) Å), and two long, Y(1)—C(2) and Y(1)—C(3) (2.599(7) and 2.598(7) Å), Y—C(C<sub>10</sub>H<sub>8</sub>) bonds testifies to the  $2\eta^1:\eta^2$ -interaction of the Y atom with the naphthalene dianion. The same yttrium complex in a mixture with  $\text{Cp}_3\text{Y}$  is formed in the reaction of  $\text{Cp}_2\text{YCl}$  with sodium naphthalenide.

**Key words:** rare-earth elements, cyclopentadienyl complexes, naphthalene complexes, molecular structure, X-ray diffraction analysis.

Recently, we have obtained a lutetium complex  $\text{CpLuC}_{10}\text{H}_8(\text{DME})$  (**1a**) containing cyclopentadienyl and naphthalene ligands, and its structure has been determined.<sup>1,2</sup> The formation of an yttrium complex of similar composition was established for the reaction of  $\text{CpYCl}_2$  with sodium naphthalenide.<sup>3</sup> To elucidate the effect of the nature of the metal on the composition and the structure of rare-earth element (REE) mixed naphthalene complexes we have studied reactions of the naphthalene radical anion with dichlorides  $\text{CpLnCl}_2(\text{THF})_3$  ( $\text{Ln} = \text{Y, Sm, Gd, Er, Tm, and Yb}$ ), and one of the products obtained,  $\text{CpYC}_{10}\text{H}_8(\text{DME})$  (**1b**), was analyzed by the X-ray diffraction method.

### Results and Discussion

REE-cyclopentadienyl dichlorides react with sodium naphthalenide in different ways, depending on the nature of the metal present in the complex. Typical tervalent lanthanides (Gd, Er, and Tm) and yttrium give soluble naphthalene complexes, whose compositions are similar to that of the previously obtained lutetium complex **1a**.



$\text{Ln} = \text{Y}$  (**1b**),  $\text{Gd}$  (**1c**),  $\text{Er}$  (**1d**),  $\text{Tm}$  (**1e**)

These reactions result in the formation of purple-violet (Er, Tm) or blue-violet (Y, Gd) solutions. Yttrium and gadolinium complexes are unstable in solution: at room temperature, the color of the solution changes to brown within several hours, and after this no individual products could be isolated. Reactions with derivatives of these metals were carried out on cooling to 0 °C. It is likely that partial decomposition results in a lower yield of complexes **1b,c** compared to complexes **1d,e** (Table 1). All compounds of the composition  $\text{CpLnC}_{10}\text{H}_8(\text{DME})$  have close melting points and the same sets of bands in the IR spectra, which testifies to their similar structures.

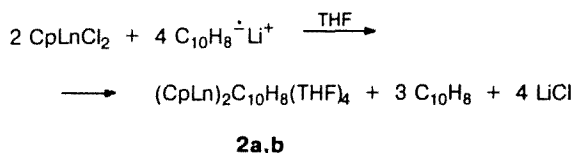
When  $\text{CpLnCl}_2$  complexes of lanthanides with the stable oxidation state +2 react with the naphthalene radical anion, the metal is reduced, and low-solubility naphthalene complexes, whose compositions differ from those of compounds **1a—e** described above, are formed.

<sup>†</sup> Deceased.

**Table 1.** Main properties of mixed REE cyclopentadienyl—naphthalene complexes

Compound	Composition	Ln	Oxidation state of Ln	Magnetic moment, $\mu_{\text{eff}}/\mu_{\text{B}}$	M.p. /°C	Yield (%)
<b>1a*</b>	CpLnC <sub>10</sub> H <sub>8</sub> (DME)	Lu	+3	0	120 (decomp.)	80
<b>1b</b>	The same	Y	+3	0	119 (decomp.)	27
<b>1c</b>	" "	Gd	+3	8.2	116 (decomp.)	7
<b>1d</b>	" "	Er	+3	10.1	122 (decomp.)	62
<b>1e</b>	" "	Tm	+3	8.0	116 (decomp.)	43
<b>2a</b>	(CpLn) <sub>2</sub> C <sub>10</sub> H <sub>8</sub> (THF) <sub>4</sub>	Sm	+2	3.4	—**	94
<b>2b</b>	The same	Yb	+2	0	—**	33

\* According to literature data.<sup>2</sup> \*\* Decomposes upon heating above 100 °C without melting.



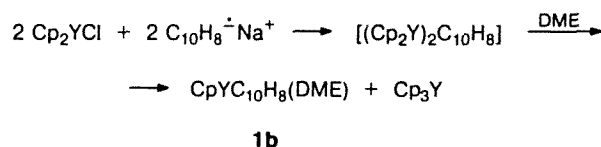
Ln = Sm (**2a**), Yb (**2b**)

In order to separate the low-solubility complexes **2a** and **2b** from the alkaline metal formed, the reaction was carried out in THF, using lithium naphthalenide. Under these conditions, the samarium complex immediately precipitates from solution as a dark-blue powder (black after drying), while the ytterbium complex is not isolated but forms a dark-violet solution. Compound **2b** was synthesized after exchange of the solvent for DME and washing of the precipitate formed with THF. Freshly prepared complex **2b** is soluble in THF in the presence of LiCl excess, but it loses this ability after drying or prolonged storage in the pure solvent.

The magnetic moments of complexes **2a** and **2b** (see Table 1) indicate that they contain a metal in the oxidation state +2. The IR spectra of these complexes differ markedly from those of compounds **1a–e**: they do not contain an intense band at 1250 cm<sup>−1</sup> and exhibit bands at 700, 1350, 1400, and 1500 cm<sup>−1</sup>, which are absent in the spectra of CpLnC<sub>10</sub>H<sub>8</sub>(DME). A similar set of bands (the values of the maxima differ by 3–5 cm<sup>−1</sup>, but the relative intensities and the shape of bands are retained) was observed<sup>4</sup> in the IR spectra of lanthanum and europium complexes with bridged  $\mu\text{-}\eta^4\text{:}\eta^4\text{-naphthalene}$ , [LaI<sub>2</sub>(THF)<sub>3</sub>]<sub>2</sub>( $\mu\text{-}\eta^4\text{:}\eta^4\text{-C}_{10}\text{H}_8$ ) and [EuI(DME)]<sub>2</sub>( $\mu\text{-}\eta^4\text{:}\eta^4\text{-C}_{10}\text{H}_8$ ). This similarity of the IR spectra allows one to suggest that binuclear complexes **2a** and **2b** also contain the bridged naphthalene dianion linked to two lanthanide atoms by the  $\mu\text{-}\eta^4\text{:}\eta^4\text{-type}$ .

It is of interest that typical trivalent REE, Lu and Y, do not form stable compounds with bridged naphthalene, even when dicyclopentadienyl derivatives Cp<sub>2</sub>LnCl are used, in which case the formation of complexes of (Cp<sub>2</sub>Ln)<sub>2</sub>C<sub>10</sub>H<sub>8</sub> composition could be expected according to the stoichiometry of the reaction.<sup>3</sup> Continuing the study of reactions of Cp<sub>2</sub>YCl with sodium naphthalenide, we found that the yttrium complex CpYC<sub>10</sub>H<sub>8</sub>(DME)

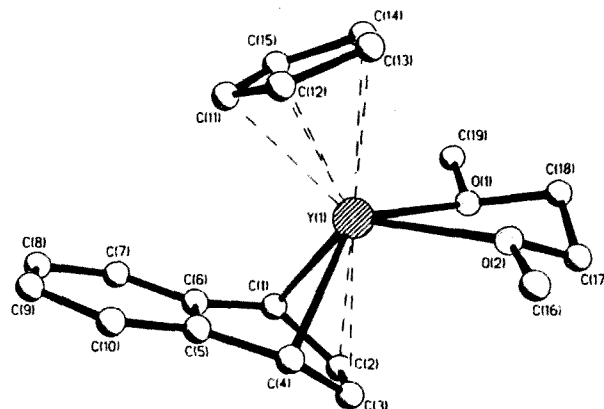
(**1b**) (in a mixture with Cp<sub>3</sub>Y) is isolated upon fast cooling of the concentrated reaction mixture.



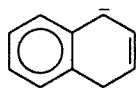
The instability of the complex in solution precluded its isolation in the pure state. However, single crystals for the measurement of unit cell parameters were selected. The unit cell parameters of this compound are similar to those of complex **1b** obtained from CpYCl<sub>2</sub>. It indicates that the structures of these compounds are identical.

The X-ray study confirms that the structure of complex **1b** (Fig. 1) is similar to that of CpLuC<sub>10</sub>H<sub>8</sub>(DME). In the molecule, the Y atom is bonded with the Cp ligand, naphthalene, and the DME molecule.

The six-membered naphthalene ring coordinated with the Y(1) atom in **1b** is nonplanar: the ring is bent by 26.1° over the C(1)...C(4) line. This value is somewhat lower than that in the CpLuC<sub>10</sub>H<sub>8</sub>(DME) molecule, 31.5° (see Refs. 1 and 2). As in the Lu analog, molecule **1b** exhibits a redistribution of values of C—C bond lengths in the coordinated six-membered naphthalene ring compared to their values in the free naphthalene. The C(1)—C(2), C(1)—C(6), C(3)—C(4), and

**Fig. 1.** General view of molecule **1b**.

C(4)—C(5) distances (which are equal to 1.45(1), 1.477(9), 1.44(1), and 1.483(9) Å, respectively) are greater than the C(2)—C(3) distances (1.37(1) Å). The corresponding C—C distances in the free naphthalene (with symmetry  $D_{2h}$ ) are 1.422, 1.406, and 1.364 Å. The C—C bond lengths in the naphthalene ring that are not linked with the Y atom are in the range from 1.36(1) to 1.426(9) Å (the average value is 1.39 Å). Thus, naphthalene in **1b** can be presented as the dianion



The interatomic Y(1)—C(1) and Y(1)—C(4) distances (2.438(6) and 2.452(6) Å, respectively) in **1b** are comparable with the Y—C  $\sigma$ -bond lengths in the other yttrium complexes studied: (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>YMe(THF) 2.44 Å,<sup>5</sup> (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>YCH(SiMe<sub>3</sub>)<sub>2</sub> 2.468 Å,<sup>6</sup> and Cp<sub>2</sub>Y(C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>NMe<sub>2</sub>-*o*) 2.486, 2.362 Å.<sup>7</sup> The Y(1)—C(2) and Y(1)—C(3) distances (2.599(7) and 2.598(6) Å, respectively) are comparable to the values of the  $\eta^5$ -Y(1)—C bond lengths (the average value is 2.65 Å) and are considerably shorter than the Y(1)—C(5) (2.812(6) Å) and Y(1)—C(6) (2.804 Å) distances. Therefore, the bond of naphthalene with the Y atom in **1b** can be presented as the  $2\eta^1:\eta^2(2\sigma,\pi)$ -interaction.

The Y(1)—C(Cp) bond lengths in **1b** range from 2.603(7) to 2.679(7) Å. The average value of 2.65 Å is close to the analogous values in the other trivalent yttrium complexes: for example, 2.67 Å in (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>YCH(SiMe<sub>3</sub>)<sub>2</sub><sup>6</sup> and 2.65 Å in Cp<sub>2</sub>Y(C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>NMe<sub>2</sub>-*o*).<sup>7</sup>

Comparison of the Ln—C bond lengths in complexes **1a** and **1b** shows that the Y—C bonds in **1b** are longer than the analogous Lu—C bonds in **1a** by  $\sim 0.03$ – $0.05$  Å. This difference is comparable with the difference in

ionic radii of these metals of 0.039 Å (0.900 Å for Y<sup>3+</sup> and 0.861 Å for Lu<sup>3+</sup>).<sup>8</sup> The exceptions are the Ln—C(5) and Ln—C(6) distances that are somewhat shorter in the yttrium complex (2.812(6) and 2.804(6) Å) than in the lutetium complex (2.819(8) and 2.830(8) Å). This can be associated with the participation of the C(5) and C(6) atoms in **1b** in the coordination of naphthalene with the Y atom, and, as a consequence, with the decrease in the bending angle of the coordinated naphthalene ring compared to the Lu analog.

## Experimental

All reactions were carried out in evacuated tubes and seamless glassware. Solvents were dried over sodiumbenzophenoneketyl (THF and DME) or sodium mirror (hexane) and were distilled to a reaction flask prior to use. REE were determined by complexometry. Organic volatiles were analyzed on a Tsvet-104 chromatograph (column 3 m, 10 % PEG-20M on Chromaton N-AW). IR spectra were recorded on a Perkin Elmer 577 instrument.

**Synthesis of CpTmC<sub>10</sub>H<sub>8</sub>(DME) (1e).** A solution obtained from sodium (0.066 g, 2.85 g-at.) and naphthalene (0.410 g, 3.2 mmol) in DME (5 mL) was added to a suspension of CpTmCl<sub>2</sub>(THF)<sub>3</sub> (0.742 g, 1.42 mmol) in DME (10 mL). The reaction mixture was stirred at room temperature until complete dissolution of the initial Tm complex, then centrifuged and decanted from a precipitate of NaCl. The solution was concentrated *in vacuo* and cooled to  $-15$  °C. The crystals formed were washed with hexane and dried *in vacuo*. Complex **1e** was obtained (0.275 g, 43 %), m.p. 114–116 °C (decomp.),  $\mu_{\text{eff}} = 8.0 \mu_B$ . Found (%): Tm, 37.0. C<sub>19</sub>H<sub>23</sub>O<sub>2</sub>Tm. Calculated (%): Tm, 37.4.

Complex CpErC<sub>10</sub>H<sub>8</sub>(DME) (**1d**) with m.p. 120–122 °C (decomp.),  $\mu_{\text{eff}} = 10.1 \mu_B$  was obtained in a similar way. Found (%): Er, 36.9. C<sub>19</sub>H<sub>23</sub>O<sub>2</sub>Er. Calculated (%): Er, 37.1.

Complexes **1b** and **1c** were synthesized in a similar way by the reaction at 0 °C. CpYC<sub>10</sub>H<sub>8</sub>(DME) (**1b**) was obtained in a yield of 27 %, m.p. 117–119 °C (decomp.). Found (%): Y, 24.2. C<sub>19</sub>H<sub>23</sub>O<sub>2</sub>Y. Calculated (%): Y, 23.9.

**Synthesis of (CpSm)<sub>2</sub>C<sub>10</sub>H<sub>8</sub>(THF)<sub>4</sub> (2a).** A solution of

**Table 2.** Geometric parameters of molecule **1b**

Bond	d/Å	Bond	d/Å	Angle	$\omega/\text{deg}$	Angle	$\omega/\text{deg}$
Y(1)—O(1)	2.406(4)	C(1)—C(2)	1.45(1)	O(1)—Y(1)—O(2)	67.2(1)	C(1)—C(6)—C(5)	118.4(6)
Y(1)—O(2)	2.391(4)	C(1)—C(6)	1.477(9)	Y(1)—O(1)—C(18)	116.6(4)	C(1)—C(6)—C(7)	123.2(6)
Y(1)—C(1)	2.438(6)	C(2)—C(3)	1.37(1)	Y(1)—O(1)—C(19)	121.7(4)	C(5)—C(6)—C(7)	118.4(6)
Y(1)—C(2)	2.599(7)	C(3)—C(4)	1.44(1)	C(18)—O(1)—C(19)	111.4(5)	C(6)—C(7)—C(8)	121.5(7)
Y(1)—C(3)	2.598(6)	C(4)—C(5)	1.483(9)	Y(1)—O(2)—C(16)	123.0(4)	C(7)—C(8)—C(9)	120.3(6)
Y(1)—C(4)	2.452(6)	C(5)—C(6)	1.426(9)	Y(1)—O(2)—C(17)	116.6(3)	C(8)—C(9)—C(10)	120.2(7)
Y(1)—C(5)	2.812(6)	C(5)—C(10)	1.404(9)	C(16)—O(2)—C(17)	113.1(5)	C(5)—C(10)—C(9)	121.4(6)
Y(1)—C(6)	2.804(6)	C(6)—C(7)	1.395(9)	C(2)—C(1)—C(6)	115.8(5)	C(12)—C(11)—C(15)	107.8(6)
Y(1)—C(11)	2.603(7)	C(7)—C(8)	1.38(1)	C(1)—C(2)—C(3)	119.5(6)	C(11)—C(12)—C(13)	108.1(6)
Y(1)—C(12)	2.639(7)	C(8)—C(9)	1.36(1)	C(2)—C(3)—C(4)	121.1(6)	C(12)—C(13)—C(14)	108.8(6)
Y(1)—C(13)	2.675(7)	C(9)—C(10)	1.38(1)	C(3)—C(4)—C(5)	115.3(6)	C(13)—C(14)—C(15)	107.5(6)
Y(1)—C(14)	2.679(7)	C(11)—C(12)	1.39(1)	C(4)—C(5)—C(6)	118.4(6)	C(11)—C(15)—C(14)	107.8(6)
Y(1)—C(15)	2.630(7)	C(11)—C(15)	1.39(1)	C(4)—C(5)—C(10)	123.5(6)	O(2)—C(17)—C(18)	107.2(5)
O(1)—C(18)	1.439(8)	C(12)—C(13)	1.39(1)	C(6)—C(5)—C(10)	118.1(6)	O(1)—C(18)—C(17)	107.1(5)
O(1)—C(19)	1.428(8)	C(13)—C(14)	1.37(1)				
O(2)—C(16)	1.428(9)	C(14)—C(15)	1.415(9)				
O(2)—C(17)	1.426(8)	C(17)—C(18)	1.49(1)				

**Table 3.** Coordinates of atoms ( $\times 10^4$ ) and equivalent isotropic\* temperature parameters ( $\times 10^3$ ) in the structure of **1b**

Atom	x	y	z	$U/\text{\AA}^2$
Y(1)	841(1)	2906(1)	297(1)	20(1)
O(1)	-476(3)	2549(3)	-597(3)	24(1)
O(2)	964(3)	1494(3)	-359(3)	29(1)
C(1)	-193(4)	3440(4)	1434(4)	24(2)
C(2)	-324(5)	2501(5)	1523(4)	29(2)
C(3)	434(5)	1971(4)	1655(4)	28(2)
C(4)	1355(5)	2329(4)	1703(4)	29(2)
C(5)	1412(5)	3247(4)	2011(4)	26(2)
C(6)	633(4)	3805(4)	1873(4)	25(2)
C(7)	694(5)	4674(4)	2144(4)	35(2)
C(8)	1497(6)	5005(4)	2505(5)	42(3)
C(9)	2249(6)	4479(5)	2625(5)	43(3)
C(10)	2207(5)	3606(4)	2399(4)	32(2)
C(11)	1923(5)	4262(5)	152(5)	36(3)
C(12)	2459(5)	3574(5)	-160(5)	37(2)
C(13)	2075(5)	3294(5)	-943(5)	33(2)
C(14)	1315(5)	3804(4)	-1129(4)	31(2)
C(15)	1214(5)	4414(4)	-445(5)	36(2)
C(16)	1626(6)	854(5)	-87(6)	44(3)
C(17)	126(5)	1118(4)	-682(5)	32(2)
C(18)	-372(5)	1815(5)	-1174(4)	33(2)
C(19)	-1050(5)	3211(5)	-972(6)	36(2)

\* Equivalent isotropic  $U$  were determined as one third of the trace of orthogonalized tensor  $U(i, j)$ .

lithium naphthalenide obtained from metallic lithium (0.008 g, 1.20 g-at.) and naphthalene (0.2 g, 1.56 mmol) in THF (5 mL) was gradually added with stirring to a suspension of  $\text{CpSmCl}_2(\text{THF})_3$  (0.3 g, 0.6 mmol) in THF (5 mL). At first the initial samarium compound was dissolved to form a dark-green solution, from which a dark-blue precipitate was sedimented upon further addition of  $\text{C}_{10}\text{H}_8\text{Li}$ . The precipitate was separated by centrifugation, washed with THF ( $2 \times 5$  mL), and dried *in vacuo*. Complex **2a** (0.240 g, 94 %) was obtained as a finely dispersed paramagnetic powder that decomposed without melting at  $\sim 100^\circ\text{C}$ ,  $\mu_{\text{eff}} = 3.4 \mu_{\text{B}}$ . Hydrolysis of **2a** gives  $\text{Sm}(\text{OH})_3$ ,  $\text{C}_5\text{H}_6$ , THF,  $\text{C}_{10}\text{H}_{10}$  (yield 95 % as a mixture of 1,2- and 1,4-dihydronaphthalenes), and traces of  $\text{C}_{10}\text{H}_8$  and  $\text{C}_{10}\text{H}_{12}$ . Found (%): Sm, 35.7.  $\text{C}_{36}\text{H}_{50}\text{O}_4\text{Sm}_2$ . Calculated (%): Sm, 35.5. IR (Vaseline oil),  $\nu/\text{cm}^{-1}$ : 450, 700, 760, 780, 880, 970, 1000, 1040, 1180, 1360, 1400, 1500.

**Synthesis of  $(\text{CpYb})_2\text{C}_{10}\text{H}_8(\text{THF})_4$  (**2b**).** A solution of lithium naphthalenide obtained from metallic lithium (0.017 g, 2.42 g-at.) and naphthalene (0.341 g, 2.66 mmol) in THF (5 mL) was gradually added with stirring to a suspension of  $\text{CpYbCl}_2(\text{THF})_3$  (0.636 g, 1.21 mmol) in THF (10 mL). The initial ytterbium complex was dissolved to form a bright-red and then dark-violet solution. The major portion of THF was removed *in vacuo*, and DME (10 mL) was added. The suspension obtained was centrifuged off. The residue was washed with THF ( $3 \times 5$  mL), the color of the first portions of the washing solution being intensely violet. The precipitate was dried *in vacuo* to give compound **2b** (0.178 g, 33 %) as a diamag-

netic black powder that decomposed without melting at  $\sim 100^\circ\text{C}$ . Found (%): Yb, 40.0.  $\text{C}_{36}\text{H}_{50}\text{O}_4\text{Yb}_2$ . Calculated (%): Yb, 38.8. IR spectrum is similar to that of complex **2a**.

The X-ray diffraction experiment for compound **1b** was carried out on a Siemens P3/PC diffractometer ( $-120^\circ\text{C}$ , Mo-K $\alpha$  radiation, graphite monochromator,  $\theta/2\theta$ -scanning in the range of  $2 < 2\theta < 52^\circ$ ). Crystals are orthorhombic, at  $-120^\circ\text{C}$   $a = 14.400(3)$ ,  $b = 15.289(3)$ ,  $c = 15.393(3)$  Å,  $V = 3388(2)$  Å<sup>3</sup>, space group  $Pbca$ ,  $Z = 8$ ,  $d_{\text{calc}} = 1.459$  g cm<sup>-3</sup>,  $\mu(\text{Mo-K}\alpha) = 34.65$  cm<sup>-1</sup>. Of 2927 independent reflections measured, 1592 reflections with  $I > 4\sigma(I)$  were used for refinement of the structure. Structure **1b** was solved by direct methods and refined in the full-matrix least-squares method in the anisotropic approximation for all nonhydrogen atoms. The positions of H atoms were calculated from the geometric conditions. Hydrogen atoms were refined in terms of the riding model with nonfixed isotropic thermal parameters. In the final stages of refinement, the following weighted scheme was used:  $w^{-1} = \sigma^2(F) + 0.001F^2$ . Absorption was taken into account by the DIFABS program.<sup>9</sup> The final values of divergence factors were  $R = 0.040$ ,  $R_w = 0.047$ , and  $S = 1.04$ . Calculations were performed on a personal computer using the SHELXTL PLUS program package.<sup>10</sup> Geometric parameters, coordinates of atoms, and their equivalent isotropic temperature factors in structure **1b** are presented in Tables 2 and 3.

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