Related literature. Although common for other metals (*e.g.* Ru, Os), triple chloride bridges are rare for Ni which prefers to form doubly bridged species such as $Ni_2Cl_8^{4-}$ (Ross & Stucky, 1970) and $[Ni_2(en)_4Cl_2]^{2+}$ where en = 1,2-diaminoethane (Carlin, Joung, O'Connor & Sinn, 1979). The only other structurally characterized example of a halo-bridged Ni^{II}/Ni^{II} dimer involving thioether ligands is the doubly chloride bridged $[Ni_2\{N(CH_2CH_2S^iPr)_3\}_2Cl_2]^{2+}$ (Carrié, Holm, Muetterties & Stavropoulos, 1991).

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Structure of Ethyl 8,9-Dimethoxy-3-(4-methoxyphenyl)-10b-methyl-1-phenyl-4,5,6,10b-tetrahydro-1,2,4-triazolo[3,4-*a*]isoquinoline-5-carboxylate

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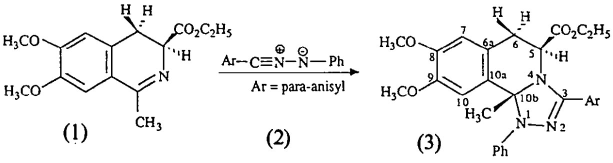
Abstract. $C_{29}H_{31}N_3O_5$, $M_r = 501.6$, orthorhombic, $Pna2_1$, $a = 23.867$ (2), $b = 9.492$ (1), $c = 11.6150$ (6) Å, $V = 2631.2$ (6) Å^3, $Z = 4$, $D_m = 1.27$, $D_x = 1.266$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.67$ mm^{-1}, $F(000) = 1064$, $T = 295$ (1) K, $R = 0.033$ for 1933 independent observed reflections. The title compound has the methyl group at C(10b) and the ester moiety at C(5) in a *cis* disposition; so the name may be specified as ethyl 3-*p*-anisyl-8,9-dimethoxy-*exo*-10b-methyl-1-phenyl-4,5,6,10b-tetrahydro-1,2,4-triazolo[3,4-*a*]isoquinoline-*exo*-5-carboxylate. In the cycloaddition synthesis, the approach of the 1,3-dipole occurs to the less-hindered diastereotopic side of the dipolarophile. Moreover, the conformation of the hexagonal N heterocycle is of the 1,3-diplanar type; the methyl group at C(10b) lies in a pseudo-equatorial disposition, whereas the ester group at C(5) lies in a pseudo-axial one.

Experimental. The 1,3-dipolar cycloaddition of C-*p*-anisyl-*N*-phenylnitrilimine (2) with ethyl 6,7-dimethoxy-1-methyl-3,4-dihydroisoquinoline-3-carboxylate (1) yields a single cycloadduct (3), regio- and stereospecifically.



Data were collected using a crystal with an approximate parallelepipedic shape, 0.19 × 0.30 × 0.34 mm. Density was measured by flotation. Intensi-

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Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (ab\cos\gamma)\beta_{12} + (ac\cos\beta)\beta_{13} + (bc\cos\alpha)\beta_{23}].$$

	x	y	z	B_{eq}
N(1)	0.98248 (7)	0.7480 (2)	0.000	4.44 (4)
N(2)	1.03342 (8)	0.8260 (2)	0.0010 (2)	4.83 (5)
C(3)	1.02079 (9)	0.9525 (3)	0.0302 (2)	4.41 (5)
N(4)	0.96295 (7)	0.9794 (2)	0.0372 (2)	4.21 (4)
C(5)	0.9397 (1)	1.0399 (3)	0.1416 (2)	4.26 (5)
C(6)	0.9210 (1)	0.9269 (3)	0.2253 (2)	4.83 (6)
C(6a)	0.8781 (1)	0.8365 (3)	0.1667 (2)	4.44 (5)
C(7)	0.8305 (1)	0.7884 (3)	0.2259 (3)	5.40 (6)
C(8)	0.7888 (1)	0.7170 (3)	0.1692 (3)	6.09 (7)
C(9)	0.7938 (1)	0.6918 (3)	0.0512 (3)	6.23 (7)
C(10)	0.8406 (1)	0.7387 (3)	−0.0075 (3)	5.41 (6)
C(10a)	0.88317 (9)	0.8100 (3)	0.0498 (2)	4.28 (5)
C(10b)	0.93598 (9)	0.8550 (3)	−0.0148 (2)	4.00 (5)
C(11)	0.9829 (1)	0.6295 (3)	−0.0759 (3)	4.75 (5)
C(12)	1.0228 (1)	0.6160 (3)	−0.1622 (3)	6.27 (7)
C(13)	1.0195 (2)	0.4999 (4)	−0.2362 (3)	7.98 (9)
C(14)	0.9788 (2)	0.4005 (4)	−0.2249 (4)	7.78 (9)
C(15)	0.9404 (2)	0.4125 (3)	−0.1371 (3)	6.88 (8)
C(16)	0.9432 (1)	0.5243 (3)	−0.0611 (3)	5.71 (7)
C(31)	1.06234 (9)	1.0638 (3)	0.0480 (2)	4.57 (5)
C(32)	1.0477 (1)	1.2042 (3)	0.0370 (3)	5.59 (6)
C(33)	1.0868 (1)	1.3109 (3)	0.0539 (3)	6.27 (7)
C(34)	1.1413 (1)	1.2744 (4)	0.0820 (3)	6.35 (7)
C(35)	1.1566 (1)	1.1343 (4)	0.0902 (3)	6.23 (7)
C(36)	1.1177 (1)	1.0303 (3)	0.0744 (3)	5.26 (6)
O(37)	1.18305 (9)	1.3704 (3)	0.1024 (3)	8.92 (7)
C(38)	1.1676 (2)	1.5153 (5)	0.1048 (5)	10.4 (1)
C(41)	0.9256 (1)	0.8886 (3)	−0.1415 (2)	4.82 (6)
C(51)	0.8917 (1)	1.1371 (3)	0.1096 (3)	4.44 (5)
O(52)	0.87254 (8)	1.1542 (3)	0.0164 (2)	7.10 (5)
O(53)	0.87227 (8)	1.2034 (2)	0.2017 (2)	6.67 (5)
C(54)	0.8248 (2)	1.2985 (4)	0.1831 (4)	8.43 (9)
C(55)	0.8166 (2)	1.3753 (5)	0.2944 (6)	11.6 (1)
O(81)	0.74075 (9)	0.6657 (3)	0.2183 (3)	8.75 (1)
C(82)	0.7335 (2)	0.6835 (5)	0.3388 (4)	11.6 (1)
O(91)	0.75067 (9)	0.6206 (3)	0.0007 (3)	8.93 (7)
C(92)	0.7602 (2)	0.5732 (6)	−0.1134 (6)	12.2 (1)

Table 2. *Bond distances* (Å) *and angles* (°)

N(1)—N(2)	1.423 (3)	C(11)—C(12)	1.388 (4)
N(1)—C(10b)	1.514 (3)	C(11)—C(16)	1.388 (4)
N(1)—C(11)	1.429 (3)	C(12)—C(13)	1.400 (5)
N(2)—C(3)	1.283 (3)	C(13)—C(14)	1.361 (5)
C(3)—N(4)	1.406 (3)	C(14)—C(15)	1.376 (6)
C(3)—C(31)	1.463 (4)	C(15)—C(16)	1.381 (5)
N(4)—C(5)	1.451 (3)	C(31)—C(32)	1.384 (4)
N(4)—C(10b)	1.475 (3)	C(31)—C(36)	1.393 (3)
C(5)—C(6)	1.515 (4)	C(32)—C(33)	1.390 (4)
C(5)—C(51)	1.517 (3)	C(33)—C(34)	1.385 (4)
C(6)—C(6a)	1.500 (4)	C(34)—C(35)	1.382 (5)
C(6a)—C(7)	1.403 (4)	C(34)—O(37)	1.371 (4)
C(6a)—C(10a)	1.386 (4)	C(35)—C(36)	1.368 (4)
C(7)—C(8)	1.373 (4)	O(37)—C(38)	1.424 (5)
C(8)—C(9)	1.396 (5)	C(51)—O(52)	1.186 (4)
C(8)—O(81)	1.371 (4)	C(51)—O(53)	1.325 (4)
C(9)—C(10)	1.384 (4)	O(53)—C(54)	1.464 (4)
C(9)—O(91)	1.364 (4)	C(54)—C(55)	1.496 (8)
C(10)—C(10a)	1.390 (4)	O(81)—C(82)	1.420 (6)
C(10a)—C(10b)	1.528 (3)	O(91)—C(92)	1.418 (7)
C(10b)—C(41)	1.527 (4)		
N(2)—N(1)—C(10b)	106.2 (2)	N(1)—C(10b)—C(41)	111.6 (2)
N(2)—N(1)—C(11)	114.1 (2)	N(4)—C(10b)—C(10a)	112.5 (2)
C(10b)—N(1)—C(11)	117.6 (2)	N(4)—C(10b)—C(41)	107.4 (2)
N(1)—N(2)—C(3)	106.7 (2)	C(10a)—C(10b)—C(41)	113.4 (2)
N(2)—C(3)—N(4)	114.6 (2)	N(1)—C(11)—C(12)	121.5 (2)
N(2)—C(3)—C(31)	123.6 (2)	N(1)—C(11)—C(16)	119.0 (2)
N(4)—C(3)—C(31)	121.7 (2)	C(12)—C(11)—C(16)	119.4 (3)
C(3)—N(4)—C(5)	119.7 (2)	C(11)—C(12)—C(13)	118.6 (3)
C(3)—N(4)—C(10b)	105.0 (2)	C(12)—C(13)—C(14)	121.7 (3)
C(5)—N(4)—C(10b)	119.5 (2)	C(13)—C(14)—C(15)	119.3 (3)
N(4)—C(5)—C(6)	111.7 (2)	C(14)—C(15)—C(16)	120.4 (3)
N(4)—C(5)—C(51)	108.9 (2)	C(11)—C(16)—C(15)	120.4 (3)
C(6)—C(5)—C(51)	111.4 (2)	C(3)—C(31)—C(32)	120.8 (2)
C(5)—C(6)—C(6a)	108.4 (2)	C(3)—C(31)—C(36)	120.6 (2)
C(6)—C(6a)—C(7)	121.1 (2)	C(32)—C(31)—C(36)	118.6 (2)
C(6)—C(6a)—C(10a)	119.3 (2)	C(31)—C(32)—C(33)	121.3 (3)
C(7)—C(6a)—C(10a)	119.5 (2)	C(32)—C(33)—C(34)	118.7 (3)
C(6a)—C(7)—C(8)	120.8 (3)	C(33)—C(34)—C(35)	120.4 (3)
C(7)—C(8)—C(9)	119.6 (3)	C(33)—C(34)—O(37)	123.8 (3)
C(7)—C(8)—O(81)	125.7 (3)	C(35)—C(34)—O(37)	115.8 (3)
C(9)—C(8)—O(81)	114.7 (3)	C(34)—C(35)—C(36)	120.4 (3)
C(8)—C(9)—C(10)	119.8 (3)	C(31)—C(36)—C(35)	120.6 (3)
C(8)—C(9)—O(91)	116.3 (3)	C(34)—O(37)—C(38)	117.3 (3)
C(10)—C(9)—O(91)	123.8 (3)	C(5)—C(51)—O(52)	126.8 (3)
C(9)—C(10)—C(10a)	120.7 (3)	C(5)—C(51)—O(53)	110.8 (2)
C(6a)—C(10a)—C(10)	119.6 (2)	O(52)—C(51)—O(53)	122.4 (2)
C(6a)—C(10a)—C(10b)	120.2 (2)	C(51)—O(53)—C(54)	116.4 (3)
C(10)—C(10a)—C(10b)	120.2 (2)	O(53)—C(54)—C(55)	105.9 (3)
N(1)—C(10b)—N(4)	99.8 (2)	C(8)—O(81)—C(82)	118.0 (3)
N(1)—C(10b)—C(10a)	111.2 (2)	C(9)—O(91)—C(92)	116.1 (3)

ties were measured using an Enraf–Nonius CAD-4 diffractometer with ω scans [amplitude, $s = (0.90 + 0.14\tan\theta)°$]. Cell parameters were determined from 25 reflections; $14.51 \le \theta \le 28.80°$. $0.022 \le \sin\theta/\lambda \le 0.512$ Å^{-1}, $0 \le h \le 28$, $-11 \le k \le 0$, $0 \le l \le 12$. Three intensity control reflections ($14\bar{3}$, 830, $5\bar{1}\bar{3}$) showed no significant intensity variations during measurements. No absorption correction was applied. 2278 independent reflections were measured, of which 345 were unobserved [$I < 2\sigma(I)$]. The structure was solved by direct methods, using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Full-matrix refinements were based on F. Atomic diffusion factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). Refined parameters were x, y, z for all atoms and β_{ij} values for C, N and O atoms; x,y,z were calculated for all H atoms except those bonded to C(55) and C(92); the coordinates of the latter H atoms were fixed, since they could not be stabilized. For H atoms B was chosen equal to $1.30 \times$ (B_{eq} of the neighbouring heavy atom). $R = 0.033$, $wR = 0.042$. According to Killean & Lawrence (1969), $w = 1/[\sigma^2(F) + (0.02F) + 1.0]$, $S = 1.54$. The secondary-extinction coefficient $g = 9\,(1) \times 10^{-7}$. Maximum shift to e.s.d. ratio, $(\Delta/\sigma)_{max} < 0.10$. $\Delta\rho_{max} = 0.11\,(3)$, $\Delta\rho_{min} = -0.13\,(3)$ e Å^{-3}. Calculations were performed using the computer programs of the *SDP* system (B. A. Frenz & Associates, Inc., 1982) Program *ORTEP*II (Johnson, 1976) was used to represent the molecule (Fig. 1). Table 1 lists atomic coordinates and equivalent isotropic thermal parameters; Table 2 gives bond distances and angles.*

Related literature. Details on the synthesis are given in two publications by Moustaid (1991) and Moustaid, Nguyen Dinh An, Mercier, Sedqui & Laude (1992). ^{1}H and ^{13}C NMR data allowed the determination of the regiochemistry of the cycloadduct

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* Lists of structure factors, coordinates of H atoms, anisotropic thermal parameters for non-H atoms, bond distances involving H atoms, average planes of rings, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55088 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0267]

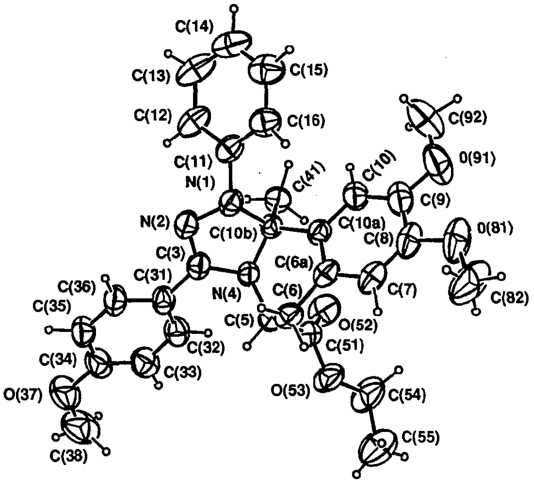

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecule.

(3), but not the stereochemistry; thus the crystal structure determination of (3) was undertaken.

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Ethanol Complex of 1,3-Diethoxy-*p*-*tert*-butylcalix[4]arene

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Abstract. 26,28-Diethoxy-5,11,17,23-tetrakis(1,1-dimethylethyl)pentacyclo[19.3.1.$1^{3,7}$.$1^{9,13}$.$1^{15,19}$]octacosa-1(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-25,27-diol–ethanol (1/1), $C_{48}H_{64}O_4.C_2H_5OH$, $M_r = 751.1$, triclinic, $P\overline{1}$, $a = 12.384$ (1), $b = 17.786$ (1), $c = 21.903$ (2) Å, $\alpha = 97.68$ (1), $\beta = 96.96$ (1), $\gamma = 105.73$ (1)°, $V = 4539$ (1) Å^3, $Z = 4$, $D_x = 1.099$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 5.04$ cm^{-1}, $F(000) = 1640$, $T = 148$ (5) K, final $R = 0.080$ for 8510 observed reflections. The unit cell contains two independent calixarene molecules, with the same cone conformation. In both calixarenes an ethanol molecule could be located, with rather high thermal parameters, indicating disorder.

Experimental. The title compound was obtained as colourless crystals, with dimensions 0.4 × 0.5 × 0.7 mm, by recrystallization of 1,3-diethoxy-*p*-*tert*-butylcalix[4]arene (Ghidini, Ugozolli, Ungaro, Harkema, Abu El-Fadl & Reinhoudt, 1990) from a 1:1 mixture of chloroform and ethanol. The complex loses ethanol at about 395 K and has an ultimate m.p. of 551–554 K. A preliminary structure determination at room temperature indicated disorder. Therefore intensities were measured at 148 (5) K on a Philips PW 1100 diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Lattice parameters were determined from 21 reflections ($6 < \theta < 15°$) by repeated measurement.

A total of 11384 unique reflections up to $\theta = 55°$ ($-13 \leq h \leq 12$, $-18 \leq k \leq 18$, $0 \leq l \leq 23$) were measured in the $\omega/2\theta$ scan mode [scan width (ω) = $(1.90 + 0.80\tan\theta)°$, scan speed (ω) = 0.2° s^{-1}]; 8510 were considered observed [$F_o^2 > 2\sigma(F_o^2)$]. Three reference reflections were measured every 60 min; a correction for long-term variations (Dam, Harkema & Feil, 1983) was applied (maximum variation 5%). No absorption correction was applied.

The crystal contains two independent calixarene molecules in a cone conformation. In the cavity of both calixarenes an ethanol molecule was found and refined, resulting in rather high thermal parameters

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