

Table 2. Bond lengths (Å) and bond angles (°) with *e.s.d.*'s in parentheses

Cation			
C1—O	1.407 (4)	O—H—O	0.82 (4)
C1—C1a	1.532 (3)	C1a—N2a	1.342 (4)
C1—C1b	1.523 (4)	C3a—N2a	1.338 (3)
C1—C1c	1.548 (5)	C1b—N2b	1.324 (4)
C1a—C6a	1.368 (4)	C3b—N2b	1.338 (4)
C3a—C4a	1.363 (5)	C3c—N2c	1.323 (4)
C4a—C5a	1.360 (5)	C3c—N2c	1.346 (6)
C5a—C6a	1.376 (5)	N2a—Hna	0.874 (35)
C1b—C6b	1.377 (4)	C1c—C6c	1.376 (5)
C3b—C4b	1.367 (5)	C3c—C4c	1.371 (7)
C4b—C5b	1.351 (5)	C4c—C5c	1.377 (6)
C5b—C6b	1.375 (5)	C5c—C6c	1.372 (6)
O—C1—C1a	107.1 (2)	C1a—N2a—C3a	123.3 (3)
O—C1—C1b	108.9 (3)	C1b—N2b—C3b	118.1 (3)
O—C1—C1c	109.4 (2)	C1c—N2c—C3c	117.5 (3)
C1a—C1—C1b	115.1 (2)	C1a—N2a—Hna	115.5 (20)
C1a—C1—C1c	107.8 (2)	C3a—N2a—Hna	121.2 (20)
C1b—C1—C1c	108.5 (2)	C1—O—Ho	102 (3)
C1—C1a—N2a	119.0 (2)	C1—C1b—N2b	117.7 (3)
C1—C1a—C6a	122.7 (2)	C1—C1b—C6b	120.1 (3)
N2a—C1a—C6a	118.1 (2)	N2b—C1b—C6b	122.1 (3)
N2a—C3a—C4a	119.5 (3)	N2b—C3b—C4b	123.0 (3)
C3a—C4a—C5a	118.6 (3)	C3b—C4b—C5b	118.4 (4)
C4a—C5a—C6a	121.1 (3)	C4b—C5b—C6b	119.9 (3)
C1a—C6a—C5a	119.3 (3)	C1b—C6b—C5b	118.6 (3)
C1—C1c—N2c	113.3 (3)	C3c—C4c—C5c	119.2 (4)
C1—C1c—C6c	123.0 (3)	C4c—C5c—C6c	118.9 (4)
N2c—C1c—C6c	123.7 (3)	C1c—C6c—C5c	118.3 (3)
N2c—C3c—C4c	122.4 (4)		
Anion			
Cl—O1	1.401 (4)	Cl—O3	1.408 (3)
Cl—O2	1.413 (3)	Cl—O4	1.392 (4)
O1—Cl—O2	111.8 (2)	O2—Cl—O3	110.9 (2)
O1—Cl—O3	110.9 (2)	O2—Cl—O4	109.0 (2)
O1—Cl—O4	106.3 (2)	O3—Cl—O4	107.8 (2)

*Acta Cryst.* (1992). **C48**, 1329–1331

## Structure of Ethyl 1-*p*-Anisyl-7,8-dimethoxy-4,5-dimethyl-3-phenyl-3a,4,5,9b-tetrahydropyrazolo[3,4-*c*]isoquinoline-3a-carboxylate

BY FRANÇOIS THEOBALD\*

14 Impasse Marguerite, 78000 Versailles, France

NOËL RODIER

Laboratoire de Chimie minérale, Faculté de Pharmacie, 5 rue J. B. Clément,  
92296 Châtenay-Malabry, CEDEX, France

AND KHADIJA MOUSTAID, NGUYEN DINH AN AND BERNARD LAUDE

Laboratoire de Chimie organique, Faculté des Sciences et des Techniques, Université de Franche-Comté,  
La Bouclie, 25030 Besançon, France

(Received 20 July 1991; accepted 18 November 1991)

**Abstract.** Ethyl 7,8-dimethoxy-1-(4-methoxyphenyl)-4,5-dimethyl-3-phenyl-3a,4,5,9b-tetrahydropyrazolo-

bridging atoms and one pyridyl group of each ligand coordinated to each In<sup>III</sup> and the third non-coordinated (Canty, Titcombe, Skelton & White, 1988). py<sub>3</sub>COH can also coordinate as a bidentate (*N,N*) ligand with dimethylgld (Canty, Minchin, Healy & White, 1982).

The author thanks the Jeffress Memorial Trust (J-20) for support of this work, Professor Michael Sabat of the University of Virginia, and Dr Cynthia Day of Crystallitics Company for helpful discussions.

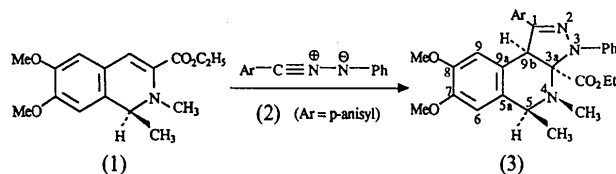
### References

- CANTY, A. J., CHAICHT, N., GATEHOUSE, B. M. & GEORGE, E. E. (1981). *Inorg. Chem.* **20**, 4293–4300.
- CANTY, A. J., MINCHIN, N. J., HEALY, P. C. & WHITE, A. H. (1982). *J. Chem. Soc. Dalton Trans.* pp. 1795–1802.
- CANTY, A. J., TITCOMBE, L., SKELTON, B. W. & WHITE, A. H. (1988). *J. Chem. Soc. Dalton Trans.* pp. 35–45.
- KEENE, F. R., SNOW, M. R. & TIEKINK, E. R. T. (1988). *Acta Cryst.* **C44**, 937–938.
- KEENE, F. R., SZALDA, D. J. & WILSON, T. A. (1987). *Inorg. Chem.* **26**, 2211–2216.
- LARSON, A. E. (1967). *Acta Cryst.* **23**, 664–665.
- MORITZ, P. S., DIAMANTIS, A. A., KEENE, F. R., SNOW, M. R. & TIEKINK, E. R. T. (1988). *Aust. J. Chem.* **41**, 1353–1362.
- SHELDRIK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Germany.
- SZALDA, D. J. & KEENE, F. R. (1986). *Inorg. Chem.* **25**, 2795–2799.
- WHITE, D. L. & FALLER, J. W. (1982). *Inorg. Chem.* **21**, 3119–3122.

\* To whom correspondence should be addressed.

$D_x = 1.277 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ \AA}$ ,  $\mu = 0.67 \text{ mm}^{-1}$ ,  $F(000) = 548$ ,  $T = 294 (1) \text{ K}$ ,  $R = 0.037$  for 3103 independent observed reflections. Aromatic rings *A*, *D* and *E* are found to have good planarity. Ring *C* is approximately flat. C(5) and C(9b) are almost coplanar with *A* [respective distances  $-0.002 (2)$  and  $0.002 (2) \text{ \AA}$ ]. The methyl group at C(5) lies in a pseudo-equatorial position with respect to *B*, whereas the methyl group C(41)H<sub>3</sub> at N(4) lies in a pseudo-axial position [torsion angle C(41)—N(4)—C(5)—C(51) =  $73.4 (2)^\circ$ ]. The *N*-methyl group and the ester moiety [at C(3a)] lie on either side of *B* in a pseudo-axial *trans* configuration [torsion angle C(41)—N(4)—C(3a)—C(31) =  $-156.8 (2)^\circ$ ]. The C(3a)—C(9b) distance [ $1.581 (2) \text{ \AA}$ ] is rather large, possibly as a result of steric effects.

**Experimental.** Source of material: the 1,3-dipolar cycloaddition reaction of *C-p*-anisyl-*N*-phenylnitrilimine (2) with ethyl 6,7-dimethoxy-1,2-dimethyl-1,2-dihydroisoquinoline-3-carboxylate (1) yielded a single cycloadduct, the title product (3), the regiochemistry of which was determined by <sup>1</sup>H and <sup>13</sup>C NMR.



Flat platelet crystal with a trapezoidal shape,  $0.07 \times 0.19 \times 0.30 \text{ mm}$ .  $D_m$  measured by flotation. Cell parameters determined from 25 reflections;  $12.18 \leq \theta \leq 29.65^\circ$ . Enraf-Nonius CAD-4 diffractometer.  $0.022 \leq (\sin \theta) / \lambda \leq 0.562 \text{ \AA}^{-1}$ ,  $-12 \leq h \leq 11$ ,  $0 \leq k \leq 9$ ,  $-16 \leq l \leq 15$ . Intensity control reflections: 411,

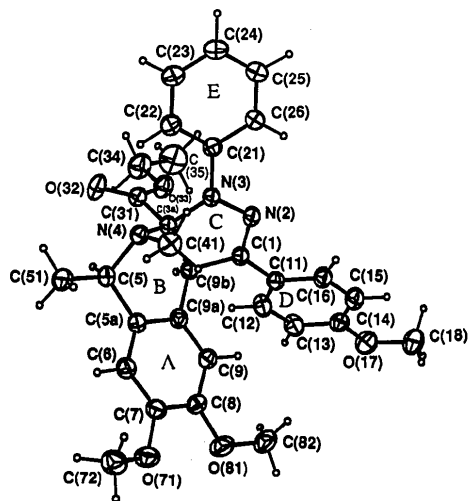


Fig. 1. ORTEP (Johnson, 1976) view of the molecule.

Table 1. Positional parameters and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

$$B_{\text{eq}} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (abc\cos\gamma)\beta_{12} + (acc\cos\beta)\beta_{13} + (bcc\cos\alpha)\beta_{23}]$$

	x	y	z	$B_{\text{eq}}$
C(1)	0.6971 (2)	0.1768 (2)	0.2630 (1)	3.05 (4)
N(2)	0.7995 (1)	0.2597 (2)	0.3156 (1)	3.39 (3)
N(3)	0.8937 (1)	0.2856 (2)	0.26684 (9)	3.34 (3)
N(4)	0.8816 (1)	0.2997 (2)	0.0997 (1)	3.36 (3)
C(3a)	0.8561 (2)	0.2007 (2)	0.1756 (1)	3.03 (4)
C(5)	0.8025 (2)	0.2244 (2)	0.0079 (1)	3.70 (4)
C(5a)	0.6623 (2)	0.2298 (2)	0.0003 (1)	3.35 (4)
C(6)	0.5808 (2)	0.2827 (2)	-0.0786 (1)	3.78 (4)
C(7)	0.4544 (2)	0.2876 (2)	-0.0816 (1)	3.76 (4)
C(8)	0.4078 (2)	0.2393 (2)	-0.0039 (1)	3.69 (4)
C(9)	0.4899 (2)	0.1926 (2)	0.0766 (1)	3.51 (4)
C(9a)	0.6177 (2)	0.1879 (2)	0.0796 (1)	3.09 (4)
C(9b)	0.7095 (2)	0.1323 (2)	0.1662 (1)	3.04 (4)
C(11)	0.5857 (2)	0.1269 (2)	0.3016 (1)	3.22 (4)
C(12)	0.5051 (2)	-0.0143 (2)	0.2735 (1)	4.22 (5)
C(13)	0.4055 (2)	-0.0666 (3)	0.3133 (1)	4.70 (5)
C(14)	0.3811 (2)	0.0216 (2)	0.3815 (1)	4.06 (4)
C(15)	0.4584 (2)	0.1627 (3)	0.4099 (1)	4.43 (5)
C(16)	0.5605 (2)	0.2137 (2)	0.3704 (1)	3.97 (4)
O(17)	0.2795 (1)	-0.0404 (2)	0.4161 (1)	5.79 (4)
C(18)	0.2478 (2)	0.0480 (3)	0.4848 (2)	6.56 (6)
C(21)	1.0206 (2)	0.3462 (2)	0.3170 (1)	3.31 (4)
C(22)	1.1180 (2)	0.3687 (3)	0.2704 (1)	4.41 (5)
C(23)	1.2416 (2)	0.4389 (3)	0.3192 (2)	5.19 (5)
C(24)	1.2691 (2)	0.4875 (3)	0.4119 (2)	5.59 (6)
C(25)	1.1726 (2)	0.4616 (3)	0.4587 (2)	5.19 (5)
C(26)	1.0491 (2)	0.3909 (2)	0.4123 (1)	4.02 (4)
C(31)	0.9322 (2)	0.0671 (2)	0.1831 (1)	3.45 (4)
O(32)	0.9940 (1)	0.0408 (2)	0.1312 (1)	5.17 (3)
O(33)	0.9143 (1)	-0.0190 (1)	0.25545 (9)	4.36 (3)
C(34)	0.9674 (2)	-0.1600 (2)	0.2658 (2)	5.58 (5)
C(35)	0.9343 (3)	-0.2369 (3)	0.3484 (2)	7.84 (8)
C(41)	0.8591 (2)	0.4572 (2)	0.1107 (1)	4.43 (5)
C(51)	0.8534 (2)	0.2903 (3)	-0.0746 (1)	5.53 (6)
O(71)	0.3678 (1)	0.3344 (2)	-0.15800 (9)	5.17 (4)
C(72)	0.4180 (2)	0.4271 (3)	-0.2248 (2)	5.36 (6)
O(81)	0.2802 (1)	0.2410 (2)	-0.0142 (1)	5.36 (4)
C(82)	0.2320 (2)	0.2118 (3)	0.0671 (2)	5.40 (5)

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

C(1)—N(2)	1.289 (2)	C(9a)—C(9b)	1.530 (2)
C(1)—C(9b)	1.519 (3)	C(11)—C(12)	1.395 (2)
C(1)—C(11)	1.469 (3)	C(11)—C(16)	1.386 (3)
N(2)—N(3)	1.384 (2)	C(12)—C(13)	1.373 (3)
N(3)—C(3a)	1.464 (2)	C(13)—C(14)	1.382 (3)
N(3)—C(21)	1.404 (2)	C(14)—C(15)	1.381 (3)
N(4)—C(3a)	1.456 (2)	C(14)—O(17)	1.364 (3)
N(4)—C(5)	1.486 (2)	C(15)—C(16)	1.390 (3)
N(4)—C(41)	1.475 (3)	O(17)—C(18)	1.428 (3)
C(3a)—C(9b)	1.581 (2)	C(21)—C(22)	1.392 (3)
C(3a)—C(31)	1.547 (3)	C(21)—C(26)	1.391 (3)
C(5)—C(5a)	1.516 (3)	C(22)—C(23)	1.391 (3)
C(5)—C(51)	1.522 (3)	C(23)—C(24)	1.366 (3)
C(5a)—C(6)	1.390 (2)	C(24)—C(25)	1.386 (3)
C(5a)—C(9a)	1.390 (3)	C(25)—C(26)	1.383 (3)
C(6)—C(7)	1.380 (3)	C(31)—O(32)	1.189 (3)
C(7)—C(8)	1.393 (3)	C(31)—O(33)	1.329 (2)
C(7)—O(71)	1.372 (2)	O(33)—C(34)	1.455 (3)
C(8)—C(9)	1.386 (2)	C(34)—C(35)	1.471 (4)
C(8)—O(81)	1.366 (2)	O(71)—C(72)	1.421 (3)
C(9)—C(9a)	1.395 (3)	O(81)—C(82)	1.423 (3)
N(2)—C(1)—C(9b)	114.0 (2)	C(5a)—C(9a)—C(9b)	118.7 (2)
N(2)—C(1)—C(11)	119.5 (2)	C(9)—C(9a)—C(9b)	122.0 (2)
C(9b)—C(1)—C(11)	126.5 (1)	C(1)—C(9b)—C(3a)	100.9 (1)
C(1)—N(2)—N(3)	109.4 (1)	C(1)—C(9b)—C(9a)	116.8 (2)
N(2)—N(3)—C(3a)	113.0 (1)	C(3a)—C(9b)—C(9a)	113.4 (1)
N(2)—N(3)—C(21)	118.8 (1)	C(1)—C(11)—C(12)	120.8 (2)
C(3a)—N(3)—C(21)	125.2 (2)	C(1)—C(11)—C(16)	121.6 (1)
C(3a)—N(4)—C(5)	107.9 (1)	C(12)—C(11)—C(16)	117.5 (2)
C(3a)—N(4)—C(41)	112.9 (2)	C(11)—C(12)—C(13)	121.4 (1)
C(5)—N(4)—C(41)	112.2 (1)	C(12)—C(13)—C(14)	120.4 (2)
N(3)—C(3a)—N(4)	111.4 (1)	C(13)—C(14)—C(15)	119.4 (1)
N(3)—C(3a)—C(9b)	102.0 (1)	C(13)—C(14)—O(17)	115.8 (2)
N(3)—C(3a)—C(31)	109.3 (1)	C(15)—C(14)—O(17)	124.8 (2)
N(4)—C(3a)—C(9b)	115.6 (1)	C(14)—C(15)—C(16)	119.8 (2)
N(4)—C(3a)—C(31)	109.1 (2)	C(11)—C(16)—C(15)	121.4 (2)
C(9b)—C(3a)—C(31)	109.1 (1)	C(14)—O(17)—C(18)	118.0 (2)

Table 2 (*cont.*)

N(4)—C(5)—C(5a)	108.4 (2)	N(3)—C(21)—C(22)	120.0 (2)
N(4)—C(5)—C(51)	111.2 (1)	N(3)—C(21)—C(26)	120.5 (1)
C(5a)—C(5)—C(51)	115.9 (2)	C(22)—C(21)—C(26)	119.5 (1)
C(5)—C(5a)—C(6)	123.4 (2)	C(21)—C(22)—C(23)	119.4 (2)
C(5)—C(5a)—C(9a)	117.0 (1)	C(22)—C(23)—C(24)	121.3 (2)
C(6)—C(5a)—C(9a)	119.5 (2)	C(23)—C(24)—C(25)	119.1 (2)
C(5a)—C(6)—C(7)	121.3 (2)	C(24)—C(25)—C(26)	121.0 (2)
C(6)—C(7)—C(8)	119.4 (2)	C(21)—C(26)—C(25)	119.7 (2)
C(6)—C(7)—O(71)	124.3 (2)	C(3a)—C(31)—O(32)	126.3 (2)
C(8)—C(7)—O(71)	116.3 (2)	C(3a)—C(31)—O(33)	109.3 (1)
C(7)—C(8)—C(9)	119.7 (2)	O(32)—C(31)—O(33)	124.4 (1)
C(7)—C(8)—O(81)	115.7 (2)	C(31)—O(33)—C(34)	116.3 (1)
C(9)—C(8)—O(81)	124.6 (2)	O(33)—C(34)—C(35)	106.7 (2)
C(8)—C(9)—C(9a)	120.8 (2)	C(7)—O(71)—C(72)	117.5 (1)
C(5a)—C(9a)—C(9)	119.2 (1)	C(8)—O(81)—C(82)	117.7 (1)

41 $\bar{3}$ , 420.  $\omega$  scans; amplitude  $s = (0.95 + 0.14 \tan \theta)^\circ$ . Intensity variations during measurements not significant. No absorption correction. 3923 unique reflections measured; 820 unobserved reflections [ $I < 3\sigma(I)$ ]. Direct method (symbolic addition method), program *SIMPEL* (Schenk, Overbeek, van der Putten, Olthof, Schagen, Peschar, Seignette, Driessen & Kiers, 1982). H atoms located from difference Fourier maps. Full-matrix refinements based on *F*. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). Refined parameters:  $x, y, z$  for all atoms and  $\beta_{ij}$  values for C, N and O atoms. For H atoms  $B$  was chosen equal to 1.30 times the value of  $B_{eq}$  of the neighbouring heavy atom.  $R = 0.037$ ,  $wR = 0.052$ ,  $w = 1/\sigma^2(F)$ ,  $S = 1.95$ . Secondary-extinction coefficient,  $g = 2.6(3) \times 10^{-6}$ . Maximum shift to e.s.d. ratio  $(\Delta/\sigma)_{\max} < 0.04$ .  $\Delta\rho_{\max} = 0.17(3)$ ,  $\Delta\rho_{\min} = -0.14(3) \text{ e } \text{Å}^{-3}$ . Computer programs of the *SDP* System (B. A. Frenz & Associates, Inc., 1982). Program *ORTEPII* (Johnson, 1976) used to represent

the molecule (Fig. 1). Atomic coordinates are listed in Table 1, bond lengths and angles in Table 2.\*

**Related literature.** A comparison of experimental torsion angles and data given by Bucourt & Hainaut (1965) shows that ring *B* adopts a quasi 1,3-diplanar conformation. Details of the synthesis are given by Moustaid (1991) and Moustaid, Nguyen Dinh An, Mercier, Sedqui & Laude (1992).

\* 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, and an *ORTEPII* plot of the molecular packing have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54867 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0252]

### References

- B. A. FRENZ & ASSOCIATES, INC. (1982). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.
- BU COURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- MOUSTAID, K. (1991). DSc Thesis. Univ. of Franche-Comté, France.
- MOUSTAID, K., NGUYEN DINH AN, MERCIER, M. F., SEDQUI, A. & LAUDE, B. (1992). *Can. J. Chem.* In the press.
- SCHENK, H., OVERBEEK, O., VAN DER PUTTEN, N., OLTHOF, G., SCHAGEN, J. D., PESCHAR, R., SEIGNETTE, P., DRIESSEN, R. & KIERS, C. (1982). *SIMPEL*. Crystallography Department, Univ. of Amsterdam.

*Acta Cryst.* (1992). **C48**, 1331–1333

## *N*-Benzylmethanesulfonamide

BY DONALD T. ELMORE, JOHN H. JONES AND MARYAM RAISSI

*The Dyson Perrins Laboratory, The University of Oxford, OX1 3QY, England*

AND KEITH PROUT

*Chemical Crystallography Laboratory, The University of Oxford, OX1 3PD, England*

(Received 3 March 1991; accepted 25 November 1991)

**Abstract.**  $\text{C}_8\text{H}_{11}\text{NO}_2\text{S}$ ,  $M_r = 185.24$ , orthorhombic, *Pbc*<sub>21</sub>,  $a = 5.473(2)$ ,  $b = 8.627(4)$ ,  $c = 19.425(5)$  Å,  $V = 917.3$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.341$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 27.7$  cm<sup>-1</sup>,  $F(000) = 392$ ,  $T = 293$  K,  $wR = 4.52$ ,  $R = 3.94\%$  for 824 reflections with  $I > 3\sigma(I)$ . This is the first crystal structure

determination reported for a simple sulfonamide containing the structural element  $\text{C}(sp^3)\text{—SO}_2\text{NH—C}(sp^3)$ .

**Experimental.** The title compound was prepared according to Johnston, Kussner & Holum (1960),