N(26)	0.4664 (3)	0.3427 (2)	0.5917 (2)	0.064 (1)
O(27)	0.5730 (3)	0.3282 (1)	0.5521 (2)	0.080 (1)
O(28)	0.4396 (3)	0.3066 (2)	0.6569 (2)	0.114 (1)
N(29)	0.1186 (3)	0.5472 (2)	0.6712 (2)	0.068 (1)
O(30)	0.1346 (3)	0.5219 (2)	0.7443 (2)	0.084 (1)
O(31)	0.0375 (3)	0.6038 (2)	0.6501 (2)	0.095 (1)
N(32)	0.2280 (3)	0.5312 (2)	0.3750 (2)	0.061 (1)
O(33)	0.1215 (3)	0.5748 (2)	0.3608 (2)	0.082 (1)
O(34)	0.3141 (3)	0.5159 (2)	0.3217 (2)	0.105 (1)

Table 2. Selected geometric parameters (Å, °)

N(1)—C(2)	1.540 (3)	O(17)—C(18)	1.412 (6)
N(1)—C(10)	1.524 (4)	C(19)—C(20)	1.444 (4)
N(1)—C(11)	1.498 (3)	C(19)—C(24)	1.448 (4)
C(2)—C(3)	1.518 (4)	C(19)—O(25)	1.244 (4)
C(2)—C(12)	1.526 (4)	C(20)—C(21)	1.368 (4)
C(2)—C(13)	1.526 (5)	C(20)—N(26)	1.466 (4)
C(3)—C(4)	1.499 (5)	C(21)—C(22)	1.375 (4)
C(4)—C(5)	1.537 (4)	C(22)—C(23)	1.368 (4)
C(4)—C(14)	1.527 (5)	C(22)—N(29)	1.444 (4)
C(5)—C(6)	1.506 (5)	C(23)—C(24)	1.362 (4)
C(5)—C(10)	1.529 (4)	C(24)—N(32)	1.462 (4)
C(6)—C(7)	1.503 (4)	N(26)—O(27)	1.208 (4)
C(7)—C(8)	1.524 (4)	N(26)—O(28)	1.222 (5)
C(7)—O(15)	1.398 (4)	N(29)—O(30)	1.222 (4)
C(7)—O(17)	1.408 (4)	N(29)—O(31)	1.226 (4)
C(8)—C(9)	1.518 (5)	N(32)—O(33)	1.219 (4)
C(9)—C(10)	1.524 (4)	N(32)—O(34)	1.207 (4)
O(15)—C(16)	1.446 (4)		

C(2)—N(1)—C(10)	114.2 (2)	C(5)—C(10)—C(9)	110.3 (3)
C(2)—N(1)—C(11)	112.5 (2)	C(7)—O(15)—C(16)	116.2 (2)
C(10)—N(1)—C(11)	110.7 (2)	C(7)—O(17)—C(18)	115.4 (3)
N(1)—C(2)—C(3)	107.0 (2)	C(20)—C(19)—C(24)	111.1 (3)
N(1)—C(2)—C(12)	110.7 (2)	C(20)—C(19)—O(25)	123.5 (3)
N(1)—C(2)—C(13)	107.9 (2)	C(24)—C(19)—O(25)	125.3 (3)
C(3)—C(2)—C(12)	111.6 (3)	C(19)—C(20)—C(21)	124.5 (3)
C(3)—C(2)—C(13)	108.4 (3)	C(19)—C(20)—N(26)	118.9 (3)
C(12)—C(2)—C(13)	111.0 (3)	C(21)—C(20)—N(26)	116.6 (3)
C(2)—C(3)—C(4)	116.2 (2)	C(20)—C(21)—C(22)	119.5 (3)
C(3)—C(4)—C(5)	109.2 (3)	C(21)—C(22)—C(23)	120.4 (3)
C(3)—C(4)—C(14)	109.9 (3)	C(21)—C(22)—N(29)	119.8 (3)
C(5)—C(4)—C(14)	112.5 (3)	C(23)—C(22)—N(29)	119.8 (3)
C(4)—C(5)—C(6)	113.3 (3)	C(22)—C(23)—C(24)	120.3 (3)
C(4)—C(5)—C(10)	112.0 (3)	C(19)—C(24)—C(23)	124.1 (3)
C(6)—C(5)—C(10)	108.4 (3)	C(19)—C(24)—N(32)	120.2 (3)
C(5)—C(6)—C(7)	113.5 (3)	C(23)—C(24)—N(32)	115.7 (3)
C(6)—C(7)—C(8)	110.1 (3)	C(20)—N(26)—O(27)	120.2 (3)
C(6)—C(7)—O(15)	113.2 (2)	C(20)—N(26)—O(28)	117.0 (3)
C(6)—C(7)—O(17)	104.7 (3)	O(27)—N(26)—O(28)	122.7 (3)
C(8)—C(7)—O(15)	105.4 (2)	C(22)—N(29)—O(30)	118.6 (3)
C(8)—C(7)—O(17)	112.7 (3)	C(22)—N(29)—O(31)	117.9 (3)
O(15)—C(7)—O(17)	110.9 (3)	O(30)—N(29)—O(31)	123.5 (3)
C(7)—C(8)—C(9)	112.0 (2)	C(24)—N(32)—O(33)	118.3 (3)
C(8)—C(9)—C(10)	110.9 (3)	C(24)—N(32)—O(34)	119.0 (3)
N(1)—C(10)—C(5)	111.2 (2)	O(33)—N(32)—O(34)	122.7 (3)
N(1)—C(10)—C(9)	110.3 (2)		

The structure was solved by direct methods using the *Xtal*3.0 suite of programs (Hall & Stewart, 1990). Structure refinement was carried out with the program *SHELX*76 (Sheldrick, 1976) using anisotropic displacement factors for non-H atoms. H atoms were generated in idealized positions (CN—H = 0.96 Å) and were then refined either as riding with the atoms to which they are bonded (CH, CH$_2$, NH) or as parts of rigid CH$_3$ groups. The isotropic temperature factors of the H atoms were fixed at either $1.0 \times U_{eq}$ (CH, CH$_2$, NH) or $1.36 \times U_{eq}$ (CH$_3$) of their carrier atoms.

References

Bernstein, J., Regev, H. & Herbstein, F. H. (1980). *Acta Cryst.* **B36**, 1170–1175.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Ferguson, G., Ruhl, B. L., Wieckowski, T., Lloyd, D. & McNab, H. (1984). *Acta Cryst.* **C40**, 1740–1742.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal*3.0 *Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.

Kasemann, O. (1993). PhD thesis, Technical Univ. of Vienna, Austria.

Palenik, G. J. (1972). *Acta Cryst.* **B28**, 1633–1635.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Ergotamine Tartrate Bis(ethanol) Solvate

S. Pakhomova, J. Ondráček, M. Hušák
and B. Kratochvíl

*Department of Solid State Chemistry,
Prague Institute of Chemical Technology, Technická 5,
CZ 155 28, Czech Republic*

A. Jegorov*

*Galena Co., Research Unit, Branišovská 31,
CZ 370 05 České Budějovice, Czech Republic*

J. Stuchlík

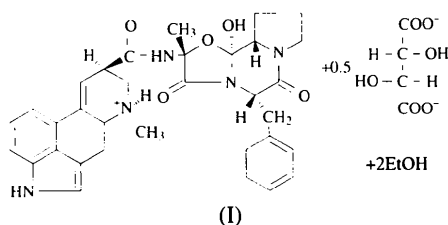*Galena Co., R & D, CZ 747 70 Opava-Komárov,
Czech Republic*

Abstract

The molecule of the title compound 12'-hydroxy-2'-methyl-3',6',18-trioxo-5'α-(phenylmethyl)ergotamanium tartrate bis(ethanol) solvate, $C_{33}H_{36}N_5$-$O_5^+ .0.5C_4H_4O_6^{2-} .2C_2H_6O$, consists of two different substituted polycyclic systems connected by an amide linkage. The partial double-bond character of the C16—N3 amide bond results in conformational rigidity of the molecule. The ergotamine *C*, *F* and *G* rings have regular envelope conformations and the *D* ring possesses a predominant half-chair conformation. An intramolecular O5—HO5···O1 hydrogen bond was found in the structure. The ergotamine and tartrate molecules are joined together through N2—HN2···O6(*x*, *y* + 1, *z*) and N3—HN3···O8(− *x*, − *y* + 1, *z*) hydrogen bonds.

Comment

Ergotamine was the first ergot alkaloid isolated from an ergot crop and subsequently it became one of the most commonly used therapeutic compounds. Ergotamine exhibits several therapeutic effects such as vasoconstriction, sensitization of vascular smooth muscle to nervous and chemical stimuli, and inhibition of circulatory baroreceptor reflexes (Berde, 1980). Ergotamine is mainly administered orally in the form of the tartrate salt, *e.g.* for the treatment of migraine attacks. To obtain a better insight into the interaction of ergotamine with possible receptor sites the present X-ray structure study of the compound (I) was undertaken.

Fig. 1. Molecular structure of ergotamine with the numbering scheme and ring labels. Displacement ellipsoids are shown at 50% probability.

(I)

The structure of the molecule with the atom numbering is shown in Fig. 1. A view of the unit-cell contents is shown in Fig. 2.

The molecule of ergotamine consists of two different substituted polycyclic systems connected by an amide linkage (Fig. 1). The bond distance between the atoms N3 and C16 of 1.351 (8) Å [torsion angle C17—N3—C16—C6 174.2 (5)°] indicates partial double-bond character (bond order 1.4). This causes rigidity of the amide linkage and reduces conformational freedom within the molecule. Rings A and B, which compose an indole moiety, are planar (χ^2 test value is 0.07 for ring A and 14.9 for ring B). The dihedral angle between these two planes is 3.4 (2)°. Ring C adopts an envelope conformation [Table 3 presents puckering parameters according to Cremer & Pople (1975)] with the atom C4 displaced by 0.642 (7) Å below the mean plane of C8, C9, C14, C2 and C3. Ring D has a conformation very close to that of a half chair with a small deviation towards an envelope. The deviations of the atoms N2 and C5 from the plane through C6, C7, C8 and C4 are 0.162 (5) and −0.615 (7) Å, respectively. Ring E approaches planarity (χ^2 test value is 25.03). The piperazine ring F has an envelope conformation with a displacement of −0.637 (7) Å for atom C25. The L-proline residue comprising the G ring forms another envelope. This conformation can alternatively be described as an A conformation with a $C\beta$-*endo* pucker [χ^1(N5—C24—C23—C22) = −37.7 (7)°]. The displacement of the atom C23 from the plane through C22, C21, N5 and C24 is 0.615 (8) Å. The L-phenylalanine residue is only
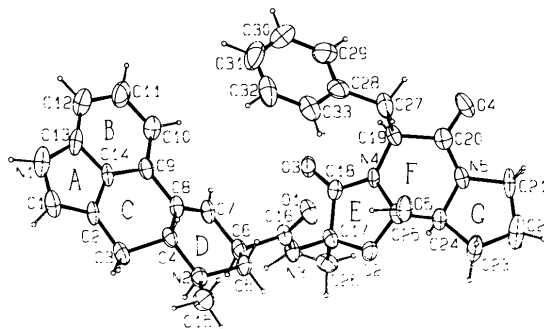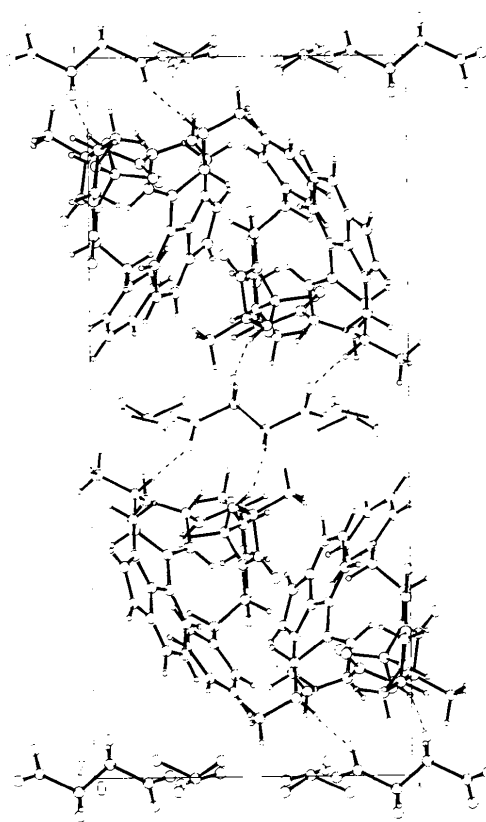
Fig. 2. Projection of the molecular packing onto the *xy* plane.

slightly distorted from its equilibrium orientation [χ^1(N4—C19—C27—C28) = −68.6 (8), χ^2(C19—C27—C28—C29) = −83.2 (8), φ^1(N4—C19—C20—N5) = 0.3 (8)°].

There is an intramolecular hydrogen bond between the hydroxyl group at O5 and the amide carbonyl O atom; the O5···O1 separation is 2.95 (1) Å [HO5···O1 = 1.94 (1) Å] and the angle O5—HO5···O1 is 177.4 (3)°. The intermolecular hydrogen bonds N2—HN2···O6(x, y + 1, z) and N3—HN3···O8($-x$, $-y$

+ 1, z) were found between the tartrate molecule and two ergotamine molecules symmetrically connected by a twofold axis [N2···O6′ = 2.75 (1), NH2···O6′ = 1.77 (1) Å, N2—HN2···O6′ = 166.9 (3)°; N3···O8″ = 2.93 (1), NH3···O8″ = 1.91 (1) Å, N3—HN3···O8″ = 154.6 (3)°].

Experimental

Crystals were obtained by cooling a hot saturated ethanol solution of commercial ergotamine tartrate (Galene Co., Czech Republic). The crystal for data collection was mounted in a glass capillary with mother liquor to prevent desolvation during the analysis.

Crystal data

$C_{33}H_{36}N_5O_5^+.0.5C_4H_4O_6^{2-}.$	Mo $K\alpha$ radiation
$2C_2H_6O$	$\lambda = 0.71073$ Å
$M_r = 748.85$	Cell parameters from 22
Orthorhombic	reflections
$P2_12_12$	$\theta = 18.13–18.81°$
$a = 11.358$ (2) Å	$\mu = 0.085$ mm^{-1}
$b = 25.591$ (5) Å	$T = 295$ K
$c = 13.497$ (2) Å	Prism
$V = 3923.1$ (7) Å^3	$0.70 \times 0.56 \times 0.14$ mm
$Z = 4$	Colourless
$D_x = 1.25$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.028$
diffractometer	$\theta_{max} = 25°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 10$
Absorption correction:	$k = -25 \rightarrow 25$
none	$l = 0 \rightarrow 18$
3081 measured reflections	3 standard reflections
3076 independent reflections	frequency: 120 min
1453 observed reflections	intensity decay: −2.1%
$[I \geq 2.5\sigma(I)]$	

Refinement

Refinement on F	$w = 0.6549/[\sigma^2(F)$
$R = 0.047$	$+ 0.0009F^2]$
$wR = 0.047$	$(\Delta/\sigma)_{max} = 0.004$
$S = 0.839$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
1453 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
475 parameters	Atomic scattering fac-
H-atom parameters not	tors from *SHELX76*
refined	(Sheldrick, 1976)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N1	0.3713 (5)	0.7410 (3)	−0.2122 (4)	0.082 (3)
N2	0.3631 (5)	0.8931 (2)	0.1247 (3)	0.057 (2)
N3	0.0557 (5)	0.8724 (2)	0.3582 (3)	0.055 (2)
N4	0.0259 (4)	0.7932 (2)	0.5576 (3)	0.047 (2)
N5	0.0162 (4)	0.8018 (2)	0.7567 (3)	0.054 (2)
O1	0.2227 (4)	0.8269 (2)	0.3936 (3)	0.074 (2)
O2	0.0604 (4)	0.8801 (2)	0.5334 (3)	0.062 (2)
O3	−0.0770 (4)	0.7745 (2)	0.4172 (3)	0.061 (2)
O4	0.0083 (5)	0.7153 (2)	0.7775 (3)	0.085 (2)

O5	0.2045 (4)	0.8322 (2)	0.6110 (3)	0.069 (2)
C1	0.4004 (6)	0.7917 (3)	−0.1973 (5)	0.078 (3)
C2	0.3664 (6)	0.8073 (3)	−0.1050 (4)	0.058 (3)
C3	0.3706 (7)	0.8575 (3)	−0.0482 (4)	0.069 (3)
C4	0.3654 (6)	0.8435 (3)	0.0648 (4)	0.048 (2)
C5	0.3287 (6)	0.8838 (3)	0.2305 (4)	0.060 (3)
C6	0.2027 (7)	0.8654 (3)	0.2295 (4)	0.058 (3)
C7	0.1896 (6)	0.8176 (3)	0.1658 (4)	0.061 (3)
C8	0.2629 (6)	0.8083 (3)	0.0916 (4)	0.052 (3)
C9	0.2573 (6)	0.7596 (3)	0.0294 (4)	0.053 (3)
C10	0.2061 (6)	0.7119 (3)	0.0529 (5)	0.070 (3)
C11	0.2114 (8)	0.6687 (4)	−0.0112 (5)	0.092 (4)
C12	0.2689 (8)	0.6739 (4)	−0.1044 (6)	0.089 (4)
C13	0.3156 (7)	0.7207 (4)	−0.1283 (5)	0.070 (4)
C14	0.3140 (6)	0.7628 (3)	−0.0627 (4)	0.053 (3)
C15	0.4791 (6)	0.9201 (3)	0.1238 (5)	0.077 (3)
C16	0.1633 (5)	0.8527 (3)	0.3377 (4)	0.051 (3)
C17	−0.0070 (6)	0.8622 (3)	0.4511 (4)	0.052 (3)
C18	−0.0236 (6)	0.8045 (3)	0.4703 (4)	0.050 (3)
C19	0.0201 (6)	0.7449 (3)	0.6110 (4)	0.051 (3)
C20	0.0152 (6)	0.7538 (3)	0.7223 (4)	0.058 (3)
C21	0.0216 (7)	0.8130 (4)	0.8646 (4)	0.080 (4)
C22	0.0319 (8)	0.8716 (4)	0.8716 (5)	0.107 (5)
C23	0.0874 (7)	0.8870 (3)	0.7704 (4)	0.084 (3)
C24	0.0245 (6)	0.8493 (3)	0.6993 (3)	0.056 (3)
C25	0.0838 (6)	0.8385 (3)	0.6006 (4)	0.053 (2)
C26	−0.1235 (6)	0.8914 (3)	0.4461 (5)	0.065 (3)
C27	0.1191 (6)	0.7076 (3)	0.5902 (4)	0.077 (2)
C28	0.1208 (5)	0.6838 (3)	0.4874 (5)	0.070 (3)
C29	0.0518 (5)	0.6400 (3)	0.4668 (5)	0.106 (5)
C30	0.0587 (5)	0.6161 (3)	0.3742 (5)	0.141 (6)
C31	0.1346 (5)	0.6359 (3)	0.3021 (5)	0.130 (6)
C32	0.2036 (5)	0.6796 (3)	0.3227 (5)	0.103 (5)
C33	0.1967 (5)	0.7036 (3)	0.4153 (5)	0.076 (3)
C34	0.1715 (8)	−0.0071 (3)	0.1309 (5)	0.081 (4)
C35	0.0491 (7)	0.0187 (3)	0.1330 (5)	0.080 (4)
O6	0.1890 (5)	−0.0395 (2)	0.0628 (3)	0.091 (2)
O7	0.2387 (5)	0.0019 (3)	0.2000 (4)	0.122 (3)
O8	0.0464 (5)	0.0521 (2)	0.2193 (4)	0.101 (2)
C36	0.3193 (14)	−0.0014 (6)	0.8334 (15)	0.332 (19)
O37	0.4010 (15)	0.0221 (9)	0.9063 (13)	0.464 (19)
C38	0.1967 (14)	0.0234 (9)	0.8459 (14)	0.304 (16)
C39	0.2262 (15)	0.4910 (14)	0.5860 (14)	0.782 (54)
O40	0.1035 (17)	0.5054 (11)	0.6109 (17)	0.514 (19)
C41	0.2345 (24)	0.4798 (9)	0.4741 (17)	0.383 (20)

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

N1—C1	1.354 (11)	N1—C13	1.398 (10)
N2—C4	1.505 (9)	N2—C5	1.500 (7)
N2—C15	1.488 (9)	N3—C16	1.351 (8)
N3—C17	1.466 (7)	N4—C18	1.337 (7)
N4—C19	1.433 (9)	N4—C25	1.454 (9)
N5—C20	1.313 (9)	N5—C21	1.485 (7)
N5—C24	1.445 (9)	O1—C16	1.209 (8)
O2—C17	1.425 (8)	O2—C25	1.424 (8)
O3—C18	1.213 (8)	O4—C20	1.238 (8)
O5—C25	1.387 (8)	C1—C2	1.364 (9)
C2—C3	1.497 (10)	C2—C14	1.406 (10)
C3—C4	1.568 (8)	C4—C8	1.516 (10)
C5—C6	1.507 (11)	C6—C7	1.502 (10)
C6—C16	1.562 (8)	C7—C8	1.324 (9)
C8—C9	1.504 (10)	C9—C10	1.389 (11)
C9—C14	1.402 (8)	C10—C11	1.405 (12)
C11—C12	1.424 (11)	C12—C13	1.349 (14)
C13—C14	1.394 (11)	C17—C18	1.511 (11)
C17—C20	1.521 (10)	C19—C20	1.520 (8)
C19—C27	1.501 (10)	C21—C22	1.507 (14)
C22—C23	1.555 (10)	C23—C24	1.537 (10)
C24—C25	1.518 (8)	C27—C28	1.515 (9)
C29—C30	1.394 (10)	C29—C28	1.396 (10)
C30—C31	1.396 (9)	C31—C32	1.394 (10)
C32—C33	1.395 (10)	C33—C28	1.396 (9)
C34—C35	1.539 (12)	C34—O6	1.254 (9)
C34—O7	1.227 (10)	C35—O8	1.445 (9)
C35—C35′	1.470 (2)	C36—O37	1.480 (26)
C36—C38	1.539 (24)	C39—O40	1.480 (28)
C39—C41	1.540 (30)		

C1—N1—C13	110.3 (6)	N3—C17—O2	110.4 (5)
C4—N2—C15	111.8 (5)	N3—C17—C18	112.5 (5)
C16—N3—C17	123.3 (5)	N3—C17—C26	107.3 (5)
C18—N4—C25	111.7 (5)	C18—C17—C26	112.3 (6)
C20—N5—C21	121.8 (5)	N4—C18—C17	108.1 (5)
C21—N5—C24	111.2 (6)	N4—C19—C20	111.7 (6)
N1—C1—C2	110.3 (6)	C20—C19—C27	107.9 (5)
C1—C2—C14	104.7 (6)	N5—C20—C19	119.3 (6)
C2—C3—C4	107.5 (6)	N5—C21—C22	104.9 (5)
N2—C4—C8	111.1 (5)	C22—C23—C24	101.6 (6)
N2—C5—C6	106.8 (5)	N5—C24—C25	110.3 (6)
C5—C6—C16	109.2 (6)	N4—C25—O2	104.9 (5)
C6—C7—C8	121.1 (7)	O2—C25—O5	109.6 (5)
C4—C8—C9	113.1 (5)	O2—C25—C24	109.9 (6)
C8—C9—C10	128.2 (5)	C19—C27—C28	115.9 (6)
C10—C9—C14	116.5 (7)	C29—C30—C31	120.1 (6)
C10—C11—C12	119.3 (9)	C31—C32—C33	120.0 (6)
N1—C13—C12	134.6 (7)	C27—C28—C29	119.9 (6)
C12—C13—C14	121.9 (7)	C29—C28—C33	120.0 (6)
C2—C14—C13	111.3 (6)	C35—C34—O7	117.9 (7)
N3—C16—O1	125.5 (5)	C34—C35—O8	106.7 (6)
O1—C16—C6	122.5 (6)	O8—C35—C35ⁱ	111.7 (5)
C4—N2—C5	112.5 (5)	O40—C39—C41	109.1 (17)
C5—N2—C15	108.2 (5)	O2—C17—C18	104.3 (5)
C18—N4—C19	127.6 (5)	O2—C17—C26	110.1 (5)
C19—N4—C25	120.5 (4)	N4—C18—O3	126.4 (6)
C20—N5—C24	126.7 (5)	O3—C18—C17	125.4 (5)
C17—O2—C25	110.9 (5)	N4—C19—C27	114.8 (5)
C1—C2—C3	135.2 (6)	N5—C20—O4	122.2 (5)
C3—C2—C14	120.1 (5)	O4—C20—C19	118.6 (6)
N2—C4—C3	109.3 (6)	C21—C22—C23	103.2 (7)
C3—C4—C8	113.4 (5)	N5—C24—C23	102.9 (5)
C5—C6—C7	110.7 (6)	C23—C24—C25	117.1 (6)
C7—C6—C16	109.7 (6)	N4—C25—O5	113.3 (6)
C4—C8—C7	123.8 (6)	N4—C25—C24	107.1 (5)
C7—C8—C9	123.0 (6)	O5—C25—C24	111.8 (5)
C8—C9—C14	115.3 (6)	C30—C29—C28	120.0 (6)
C9—C10—C11	122.2 (6)	C32—C33—C28	120.0 (6)
C11—C12—C13	118.4 (8)	C27—C28—C33	120.0 (6)
N1—C13—C14	103.5 (7)	C35—C34—O6	116.1 (7)
C2—C14—C9	127.0 (7)	O6—C34—O7	125.7 (8)
C9—C14—C13	121.6 (7)	C34—C35—C35ⁱ	113.8 (9)
N3—C16—C6	111.9 (5)	O37—C36—C38	109.1 (15)

Symmetry code: (i) −*x*, −*y*, *z*.

Table 3. Conformations of the various rings

The atoms comprising the rings are as follows: ring *C* = C8, C9, C14, C2, C3, C4; ring *D* = N2, C5, C6, C7, C8, C4; ring *F* = C24, N5, C20, C19, N4, C25; ring *G* = C23, C22, C21, N5, C24.

Ring	Conformation	Puckering Coordinates		
		Q (Å)	φ (°)	θ (°)
C	E₆	0.468 (7)	−63.8 (10)	56.7 (9)
D	¹H₂	0.530 (7)	48.8 (9)	50.7 (7)
F	E₆	0.454 (7)	−61.8 (9)	55.4 (8)
G	E₁	0.397 (8)	−177.6 (10)	

SDP software (B. A. Frenz & Associates, Inc., 1985) was used for measurement, data reduction and cell refinement. *SHELXS*86 (Sheldrick, 1985) was used to solve the structure. The structure was refined by block-diagonal least squares in six blocks using *SHELX*76 (Sheldrick, 1976). Since a difference map revealed only a few H atoms, their positions were calculated from ideal geometry and held constant during refinement, with displacement factors taken from their bond partners. The H atom of the hydroxy group of the tartrate molecule was not found. The C28–C33 phenyl ring (Fig. 1) was refined by assumption of its ideal geometry. The final difference electron density synthesis revealed the presence of two ethanol molecules. The bond distances and angles involving atoms of the two independent molecules of ethanol were chemically unreasonable, they were fixed at ideal geometry and anisotrop-

ically refined with constraints and unit occupancy factors. The molecular geometry was calculated using *PARST* (Nardelli, 1991).

References

B. A. Frenz & Associates, Inc. (1985). *SDP Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Berde, B. (1980). *Advances in Biochemical Psychopharmacology,* edited by M. Goldstein, A. Lieberman, D. B. Calne & M. O. Thorner, Vol. 23, pp. 3–23. New York: Raven Press.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Nardelli, M. (1991). *PARST. A System of Computer Routines for Calculating Molecular Parameters from the Results of Crystal Structure Analysis.* Univ. of Parma, Italy.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

2-Methoxy-5-(4-nitrostyryl)phenol

Li Zheng-Dong and Su Genbo

Fujian Institute of Research on the Structure of Matter, Academia Sinica, Fuzhou 350002, People's Republic of China

Abstract

The title compound, $C_{15}H_{13}NO_4$, has a *trans* conformation at the central C=C double bond; the dihedral angle between the phenyl rings is 6.1 (1)°.

Comment

The molecular configuration and the packing of molecules in the unit cell of the title compound, (I), are shown in Fig. 1 and Fig. 2, respectively. The H atoms are *trans* in the —C=C— group and the dihedral angle between the phenyl rings is 6.1 (1)ᶜ.

(I)