C9—N1—C10	129.1 (4)	N12—C11—C17	113.3 (4)
N2—N1—C10	120.3 (5)	C11—N12—C16	127.8 (4)
N2—N1—C9	110.0 (4)	C11—N12—N13	121.6 (4)
N1—N2—N3	108.5 (4)	N13—N12—C16	110.7 (4)
N2—N3—C4	108.5 (5)	N12—N13—C14	103.5 (5)
N3—C4—C9	108.2 (5)	N13—C14—N15	111.6 (6)
N3—C4—C5	131.6 (5)	C14—N15—C16	106.6 (5)
C5—C4—C9	120.1 (5)	N12—C16—N15	107.6 (5)
C4—C5—C6	118.3 (6)	C11—C17—C22	123.0 (5)
C5—C6—C7	120.3 (5)	C11—C17—C18	118.9 (5)
C6—C7—C11	120.8 (4)	C18—C17—C22	118.0 (5)
C6—C7—C8	121.6 (5)	C17—C18—C19	121.3 (6)
C8—C7—C11	117.5 (4)	C18—C19—C20	118.9 (5)
C7—C8—C9	116.7 (5)	Cl—C20—C19	118.9 (4)
C4—C9—C8	122.8 (5)	C19—C20—C21	122.6 (6)
N1—C9—C8	132.4 (5)	Cl—C20—C21	118.4 (5)
N1—C9—C4	104.8 (4)	C20—C21—C22	117.8 (6)
C7—C11—C17	114.1 (4)	C17—C22—C21	121.3 (5)
C7—C11—N12	109.9 (4)		

C6—C7—C11—N12	−97.4 (6)	C7—C11—N12—N13	57.4 (6)
C6—C7—C11—C17	31.2 (7)	C7—C11—N12—C16	−120.7 (5)
C8—C7—C11—N12	80.8 (6)	N12—C11—C17—C18	−177.0 (5)
C8—C7—C11—C17	−150.7 (5)	N12—C11—C17—C22	2.6 (7)
C7—C11—C17—C18	56.2 (7)	C17—C11—N12—C16	110.4 (6)
C7—C11—C17—C22	−124.2 (6)	C17—C11—N12—N13	−71.6 (6)

Table 6. *Hydrogen-bonding geometry* (Å, °) *for* (II)

D	H	A	D—H	H···A	D···A	D—H···A
N15	H15	Br	1.027	2.289	3.179 (5)	144.1
C10	H10A	N13^{i}	1.120	2.587	3.444 (7)	132.5
C8	H8	N13^{i}	1.084	2.299	3.298 (7)	152.3
C11	H11	N3^{ii}	1.098	2.518	3.333 (7)	130.1
C16	H16	N3^{ii}	1.100	2.400	3.178 (7)	126.4
C22	H22	N2^{iii}	1.100	2.406	3.320 (8)	139.5
C22	H22	N3^{iii}	1.100	2.705	3.458 (8)	125.2
O	H1W	Br^{iv}	1.084	3.398	3.849 (5)	106.5
O	H1W	Br^{v}	1.084	3.139	3.439 (5)	96.7
O	H2W	Br^{vi}	1.083	3.250	3.473 (5)	92.7
C16	H16	O	1.100	2.296	2.966 (7)	117.3

Symmetry codes: (i) $x + 1, y, z$; (ii) $x, y - 1, z$; (iii) $x - 1, y - 1, z$; (iv) $\frac{1}{2} + x, -\frac{1}{2} - y, -z$; (v) $\frac{1}{2} + x, \frac{1}{2} - y, -z$; (vi) $x, y + 1, z$.

The authors thank Professor Dr P. Janssen of Janssen Research Foundation (Beerse, Belgium) for providing the title compounds.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71325 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1038]

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Structure and Absolute Configuration of (−)-α-[(2-Acetyl-5-methylphenyl)amino]-2,6-dichlorobenzeneacetamide†

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Abstract

The title compound is a potent and highly selective human immunodeficiency virus type 1 (HIV-1) inhibitor. The two rings in the molecule are planar with a dihedral angle between their least-squares planes of 108.29 (8)°. An intramolecular N—H···O hydrogen bond forms a pseudo ring and contributes to the near coplanarity of the acetyl group with the methylphenyl moiety. The molecules are linked together in the **a** direction by N—H···O hydrogen bonds. The absolute configuration at the central asymmetric C atom is *S*.

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† Internal code of Janssen Research Foundation: R90385.

Comment

(−)-α-[(2-Acetyl-5-methylphenyl)amino]-2,6-dichlorobenzeneacetamide belongs to a chemical family of human immunodeficiency virus type 1 (HIV-1) specific reverse transcriptase inhibitors, the α-anilinophenylacetamide (α-APA) derivatives. The HIV-1 inhibition of these derivatives appeared to be stereospecific (Pauwels *et al.*, 1993). To contribute to a better understanding of the mechanism of action of these α-APA compounds, the crystal structure and absolute configuration of R90385 (the title compound), which is the most potent of the series, was determined.

A *PLUTO* diagram (Motherwell & Clegg, 1978) of the molecule with the atomic numbering scheme is shown in Fig. 1. Bond lengths and angles are within the expected ranges. The distortion of the internal angles of the rings is within 1° of the combined angular substituent parameters given by Domenicano & Murray-Rust (1979). The two phenyl rings are essentially planar (maximum deviations 0.0265 and 0.0137 Å, respectively) with a dihedral angle between their least-squares planes of 108.29 (8)°. The acetyl group is almost coplanar with the anilino moiety [dihedral angle 172.6 (2)°]. An intramolecular N—H···O hydrogen bond stabilizes this conformation [N13···O22 = 2.613 (2), H13···O22 = 1.941 Å, N13—H13···O22 = 120.3°]. The molecules are linked together by the N12—H12B···O22^{i} hydrogen bond [(i) $-\frac{1}{2}+x, \frac{1}{2}-y, -z$; N12···O22^{i} = 2.962 (3), H12B···O22^{i} = 1.949 Å, N12—H12B···O22^{i} = 155.7°], forming infinite chains in the **a** direction.

The absolute configuration is *S* and was determined by calculating the Bijvoet coefficient according to a selection procedure by Beurskens, Noordik & Beurskens (1980).

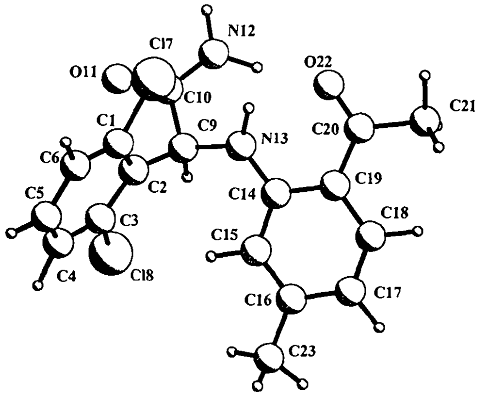

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) diagram of the molecule showing the atomic numbering scheme.

Experimental

Crystal data

$C_{17}H_{16}Cl_2N_2O_2$
M_r = 351.23
Orthorhombic
$P2_12_12_1$
a = 7.3416 (8) Å
b = 10.3924 (8) Å
c = 21.509 (2) Å
V = 1641.0 (3) Å^3
Z = 4
D_x = 1.422 Mg m^{-3}
D_m = 1.42 Mg m^{-3}
Density measured by flotation in CCl_4/n-heptane

Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 24 reflections
θ = 42-45°
μ = 3.7091 mm^{-1}
T = 293 K
Prism
0.84 × 0.49 × 0.38 mm
Colourless
Crystal source: Janssen Research Foundation, Belgium

Data collection

Siemens *P*4 four-circle diffractometer
$\omega/2\theta$ scans (2.02-29.3° min^{-1})
Absorption correction: empirical (*XEMP*, Siemens, 1989*c*)
T_{min} = 0.062, T_{max} = 0.164
5026 measured reflections
2244 independent reflections

2234 observed reflections [$I > 3.0\sigma(I)$]
R_{int} = 0.043
θ_{max} = 57.47°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 11$
$l = -23 \rightarrow 23$
3 standard reflections monitored every 50 reflections intensity variation: <3%

Refinement

Refinement on F
Final R = 0.0333
wR = 0.0575
S = 0.94
2234 reflections
209 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.00400F^2]$
$(\Delta/\sigma)_{max}$ = 0.014
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: Zachariasen (1967) and Larson (1970)

Extinction coefficient: 2.2 (2) μm
Atomic scattering factors from *International Tables for X-ray Crystallography* [1974, Vol. IV, Tables 2.2B, 2.3.1 (C, Cl, O, N) and 2.2B (H)]
Absolute configuration: the Bijvoet coefficient was 0.9315 (6) for the 217 most significant Bijvoet pairs

Data collection: *P3/PC Diffractometer Program* (Siemens, 1989*a*). Cell refinement: *P3/PC Diffractometer Program* (Siemens, 1989*a*). Data reduction: *XDISK* (Siemens, 1989*b*). Program(s) used to solve structure: *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure (full-matrix least squares): *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.0693 (3)	0.0442 (2)	0.1558 (1)	0.0558 (7)
C2	−0.0922 (3)	−0.0284 (2)	0.1575 (9)	0.0487 (6)
C3	−0.0981 (3)	−0.1231 (3)	0.2031 (1)	0.0560 (6)
C4	0.0506 (4)	−0.1530 (3)	0.2411 (1)	0.0677 (9)
C5	0.2049 (4)	−0.0836 (3)	0.2359 (1)	0.0715 (9)

C6	0.2184 (4)	0.0187 (3)	0.1934 (1)	0.0689 (8)
Cl7	0.0849 (8)	0.1731 (6)	0.1051 (3)	0.0673 (2)
Cl8	−0.2972 (9)	−0.2119 (6)	0.2151 (3)	0.0715 (3)
C9	−0.2484 (3)	−0.0057 (2)	0.1131 (9)	0.0478 (6)
C10	−0.3437 (3)	0.1236 (2)	0.1263 (1)	0.0534 (7)
O11	−0.3501 (2)	0.1634 (2)	0.1791 (8)	0.0684 (5)
N12	−0.4243 (3)	0.1808 (2)	0.0780 (1)	0.0665 (7)
N13	−0.2015 (3)	−0.0189 (2)	0.0471 (8)	0.0555 (6)
C14	−0.1889 (3)	−0.1343 (2)	0.0169 (1)	0.0487 (7)
C15	−0.1921 (3)	−0.2505 (2)	0.0493 (1)	0.0521 (7)
C16	−0.1843 (3)	−0.3691 (2)	0.0203 (1)	0.0525 (7)
C17	−0.1771 (4)	−0.3717 (3)	−0.0450 (1)	0.0616 (8)
C18	−0.1729 (3)	−0.2593 (3)	−0.0781 (1)	0.0590 (7)
C19	−0.1761 (3)	−0.1374 (2)	−0.0486 (1)	0.0499 (6)
C20	−0.1673 (4)	−0.0211 (3)	−0.0858 (1)	0.0609 (8)
C21	−0.1723 (7)	−0.0287 (4)	−0.1565 (1)	0.109 (1)
O22	−0.1507 (3)	0.0856 (2)	−0.0621 (8)	0.068 (6)
C23	−0.1770 (4)	−0.4905 (3)	0.0564 (1)	0.072 (9)

Table 2. *Geometric parameters* (Å, °)

C1—C2	1.406 (3)	C10—N12	1.336 (3)
C1—C6	1.387 (3)	N13—C14	1.367 (2)
C1—Cl7	1.731 (2)	C14—C15	1.395 (3)
C2—C3	1.391 (3)	C14—C19	1.412 (3)
C2—C9	1.510 (3)	C15—C16	1.383 (3)
C3—C4	1.398 (3)	C16—C17	1.405 (3)
C3—Cl8	1.748 (2)	C16—C23	1.481 (3)
C4—C5	1.347 (4)	C17—C18	1.368 (3)
C5—C6	1.404 (4)	C18—C19	1.417 (3)
C9—C10	1.541 (3)	C19—C20	1.452 (3)
C9—N13	1.468 (2)	C20—C21	1.521 (3)
C10—O11	1.209 (3)	C20—O22	1.227 (3)
C6—C1—Cl7	117.6 (2)	O11—C10—N12	124.2 (2)
C2—C1—Cl7	119.1 (2)	C9—N13—C14	123.9 (2)
C2—C1—C6	123.3 (2)	N13—C14—C19	119.9 (2)
C1—C2—C9	122.7 (2)	N13—C14—C15	121.4 (2)
C1—C2—C3	115.1 (2)	C15—C14—C19	118.7 (2)
C3—C2—C9	122.2 (2)	C14—C15—C16	123.0 (2)
C2—C3—Cl8	120.3 (2)	C15—C16—C23	121.6 (2)
C2—C3—C4	123.0 (2)	C15—C16—C17	118.0 (2)
C4—C3—Cl8	116.7 (2)	C17—C16—C23	120.4 (2)
C3—C4—C5	119.2 (2)	C16—C17—C18	120.3 (2)
C4—C5—C6	121.3 (3)	C17—C18—C19	122.0 (2)
C1—C6—C5	117.9 (2)	C14—C19—C18	117.9 (2)
C2—C9—N13	114.8 (2)	C18—C19—C20	119.8 (2)
C2—C9—C10	111.4 (2)	C14—C19—C20	122.3 (2)
C10—C9—N13	111.5 (2)	C19—C20—O22	121.8 (2)
C9—C10—N12	116.4 (2)	C19—C20—C21	120.4 (3)
C9—C10—O11	119.2 (2)	C21—C20—O22	117.8 (3)

The authors thank Professor Dr P. Janssen of the Janssen Research Foundation (Beerse, Belgium) for providing the title compound and W. I. Jottier for collecting the data.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71330 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1041]

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Acta Cryst. (1993). C**49**, 1963–1965

Structure of *syn*-7,8-Benzo-9,10-(9′,10′-phenanthro)tricyclo[4.2.2.$2^{2,5}$]dodeca-3,7,9-triene†

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Abstract

The double bond in the title compound, produced as a single stereoisomer in the $4\pi_s + 4\pi_s$ photocycloaddition of dibenz[*a*,*c*]anthracene with 1,3-cyclohexadiene, is established to be *syn* to the phenanthrene moiety, as predicted by the principle of maximum secondary orbital overlap. Two long interannular bonds, 1.610 (5) and 1.612 (5) Å, are present.

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† Alternative name: 9,10,13,14-tetrahydro-9,14-*o*-benzeno-10,13-ethanocycloocta[1,2-*l*]phenanthrene.

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