$C_{50}H_{71}N_9O_9.C_2H_3N.2H_2O$

$S = 2.18$
4162 reflections
658 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F_o)$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Hydrogen bonds* (Å, °)

Type*	D—H	A	D···A	N···O=C'
Intramolecular				
C7	N5	O3	3.045 (8)	86 (1)
C10	N7	O4	3.102 (7)	126 (1)
C10	N8	O5	3.042 (7)	114 (1)
C13	N4	O9	2.998 (6)	147 (1)
C17	N9	O4	3.086 (6)	133 (1)
Intermolecular	N6	O8^i	3.07 (1)	120 (1)
Peptide solvent	N3	N1s^{ii}	2.96 (4)	
	N1s	O9^{ii}	2.91 (3)	140 (2)
	O1w	O6	2.96 (1)	115 (1)
	O1w	O2^{iii}	2.78 (5)	136 (3)
	O2w	O5^{iii}	3.78 (4)	111 (1)

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

* Benedetti (1982).

Direct application of the phase determination procedures failed to give sensible interpretations of the E map. The phase problem of CLAIB was overcome by applying the molecular replacement technique using a molecular fragment of known structure. In fact, the coordinates of the Pro-Pro-Phe-Phe fragment, as determined previously in the structure of CLAIB grown from methanol/water, were used as a starting model for the vector search procedure on the set of diffraction data collected with the Patterson search program *PATSEE* (Egert, 1983). The initial model, containing 34 atoms, was correctly orientated and positioned with respect to the origin of the new unit cell. Then the best solution of this procedure was used in the *SIR*92 (Altomare, Cascarano, Giacovazzo, Guagliardi, Polidori, Burla & Camalli, 1994) program with the *PARTIAL* procedure. The direct phase-expansion procedure led to a molecular fragment containing all non-H atoms. Subsequent difference Fourier analysis revealed the two water and acetonitrile molecules. The H atoms were introduced in their stereochemically expected positions with an isotropic temperature factor equal to the B_{eq} of the heavy atom to which they are bonded. Their parameters were kept fixed. The structure was refined using *SHELX*76 (Sheldrick, 1976) on a MicroVAX 3100 computer.

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Two Steroidal Epimers, (5β,17α)-Pregna-20-yno[3,4-c][1,2,5]oxadiazol-17-ol (the 5β-Epimer) and (5α,17α)-Pregna-20-yno[3,4-c][1,2,5]oxadiazol-17-ol (the 5α-Epimer)

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Abstract

The crystal and molecular structures of the steroidal oxadiazoles (5β,17α)-pregna-20-yno[3,4-c][1,2,5]oxadiazol-17-ol (the 5β-epimer) and (5α,17α)-pregna-20-yno[3,4-c][1,2,5]oxadiazol-17-ol (the 5α-epimer), $C_{21}H_{28}N_2O_2$, have been determined. In both structures rings *A* are similar, having conformations intermediate between a strained sofa, a half-chair and an envelope. Rings *B* and *C* are also similar, adopting

chair conformations. In the 5β-epimer, ring D has a 13β-envelope conformation, while in the 5α-epimer, ring D has an envelope/intermediate half-chair conformation.

Comment

The two title compounds were prepared during work on the synthesis of 3,4-condensed oxadiazole derivatives of pregnanes, in order to test their anti-fertility properties and binding affinity to progesterone receptors (Singh, Yadav & Jindal, 1987; Yadav, Jindal & Singh, 1988). The structures of the title compounds have been determined in order to distinguish between the 5α and 5β configurations and to study the influence of the 3,4-condensed oxadiazole system on the steroid skeleton with a *cis* or *trans* junction between rings A and B. This work continues the investigation into a series of steroidal oxadiazoles which is currently being undertaken by the authors.

The average C—C single-bond length within the steroid nucleus, excluding C3—C4 for both the 5β- and 5α-epimers, has a normal value. The C3—C4 bond length, as with other steroids in the series (El Shora, Palmer, Singh & Paul, 1984), is shortened as a result of π delocalization in the oxadiazole ring, similar to that observed in other heterocyclic oxadiazoles (Sagebarth & Cox, 1965; Calleri, Chiari, Chiesi Villa, Gaetani, Guastini & Viterbo, 1975; Viterbo & Serafino, 1978; El Shora *et al.*, 1984).

Excluding bond angles C2—C3—C4 and C3—C4—C5, the average bond angle within the
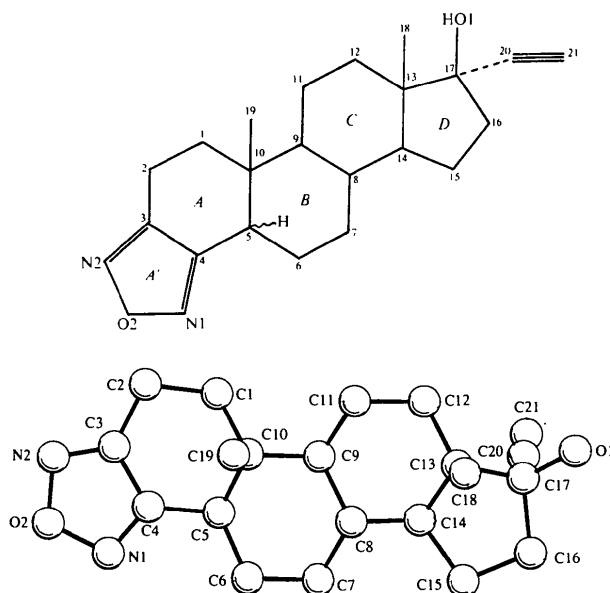

Fig. 1. Structural formula and numbering scheme for both molecules. The numbers refer to C atoms unless otherwise indicated.

steroid skeleton is 110.2 (1) and 110.0 (1)° for the 5β- and 5α-epimer, respectively. The bond angles C2—C3—C4 and C3—C4—C5 for both epimers are somewhat larger than the average and the differences are associated with fusion of the oxadiazole rings. As with similar molecules (El Shora *et al.*, 1984; Maes, Wyns, Lisgarten, Lisgarten & Palmer, 1992), there is a variation in C—C—C bond angles in the steroid nucleus depending on their environments; bond angles around quaternary, tertiary and secondary C atoms have the average values 109.4 (1), 111.4 (1) and 109.0 (1)°, respectively, for the 5β-epimer, and 109.4 (1), 111.8 (1) and 109.1 (1)°, respectively, for the 5α-epimer.

Conformational features of the molecules are described in terms of torsion angles (Table 2) and asymmetry parameters (Table 3). The magnitudes of the asymmetry parameters ΔC_s and ΔC_2 (Duax & Norton, 1975) have been calculated to indicate the deviation (about bond directions and bond bisectors) from ideal mirror ($\Delta C_s = 0$) and ideal twofold ($\Delta C_2 = 0$) symmetry. In ring A the values of $\Delta C_2(3,4)$ and $\Delta C_s(3)$ show that the conformation of ring A for both molecules is intermediate between 10-sofa and 1,10-half-chair/envelope, similar to that found in other steroidal oxadiazoles (El Shora *et al.*, 1984). For both molecules, rings B and C have low ΔC_2 and ΔC_s values and are described as having chair conformations. The five-membered D ring in the 5β-epimer has a 13β-envelope conformation, while in the 5α-epimer the D ring conformation is intermediate between 13β-envelope and 13β,14α-half-chair.

The ring-junction connections together with their absolute endocyclic torsion angles for each molecule are: A/B *cis*, T1 = 50.8 + 54.6 = 105.4°, B/C *trans*, T2 = 56.0 + 53.8 = 109.8° and C/D quasi *trans*, T3 = 60.7 + 45.5 = 106.3° for the 5β-epimer, and A/B *trans*, T1 = 53.9 + 58.5 = 112.4°, B/C *trans*, T2 = 51.7 + 57.2 = 108.9° and C/D quasi *trans*, T3 = 58.7 + 48.0 = 106.7° for the 5α-epimer. The junction between the oxadiazole ring and ring A is quasi *cis* fused in both molecules. The dihedral angles made by the planes A and B, and A and ABCD are 77.8 and 70.7°, respectively, for the 5β-epimer, showing the typical configuration for a 5β(A/B cis) steroid. The corresponding dihedral angles (2.7 and 1.5°) for the 5α-epimer indicate an almost planar molecular profile (Fig. 2).

The substituents at C17 are pseudo-equatorial (OH) and pseudo-axial (alkyne) in both molecules. The pseudo-torsion angle C19—C10—C13—C18, which measures the twist along the molecule, has values of 1.1 and 4.4° for the 5β- and 5α-epimer, respectively. Hydrogen bonding in the 5β-epimer is indicated by interactions involving the hydroxyl O atoms, O1···O1′($-x + y$, $1 - x$, $z + \frac{1}{3}$): O1···O1′ = 2.993 (2), O1—H1 = 0.801 (6), H1···O1′ =
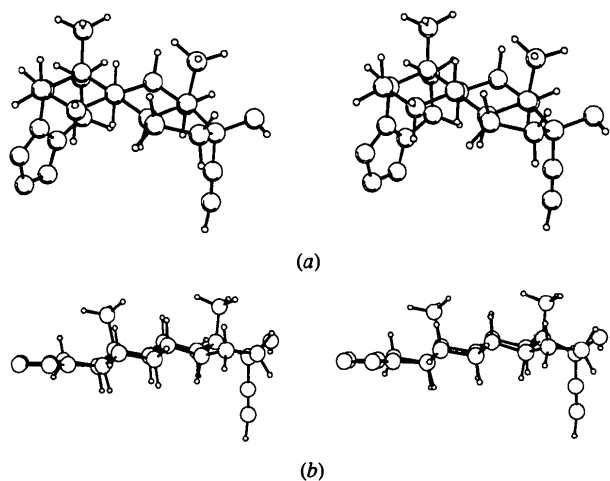
(a)

(b)

Fig. 2. Stereoview of the molecular conformations for (a) the 5β-epimer and (b) the 5α-epimer.
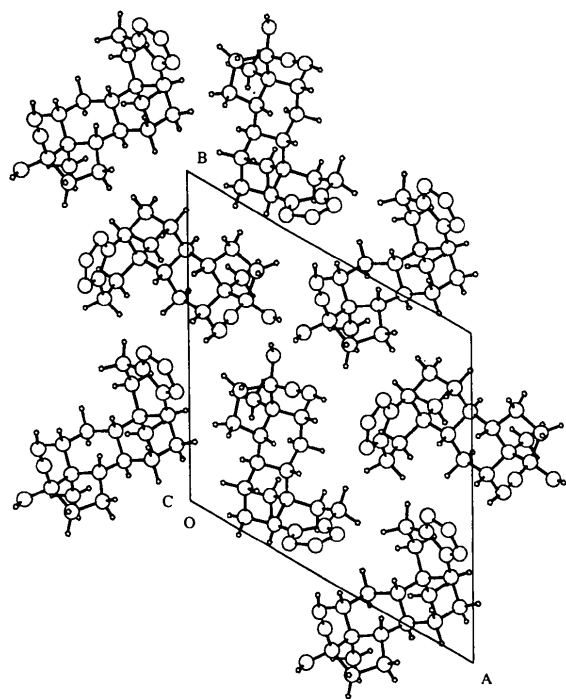

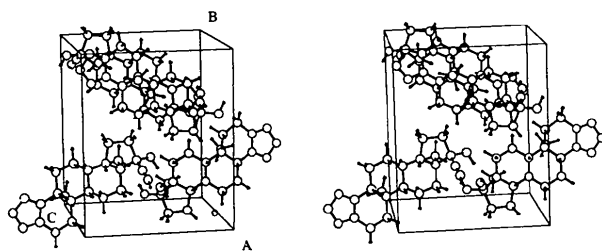Fig. 3. Crystal packing for the 5β-epimer viewed along the c axis.

Fig. 4. Stereoview of the crystal packing of the 5α-epimer.

2.195 (6) Å, O1—H1···O1' = 173.3 (1)°. In the 5α-epimer, hydrogen bonding is indicated by the (x, y, $1 - z$)O1···N1' interaction: O1···N1' = 3.040 (2), O1—H1 = 0.95 (3), N1'···H1 = 2.107 (4) Å, O1—H1···N1' = 165.8 (1)°. The hydrogen-bonding patterns are similar to those observed in a related epimeric pair of steroidal oxadiazoles (El Shora *et al.*, 1984). Packing coefficients were calculated for both molecules using the *OPEC* program (Gavezotti, 1982, 1983) and are 0.73 and 0.74 for the 5β-epimer and the 5α-epimer, respectively. These values are somewhat larger than the average for organic compounds containing N and O atoms.

Experimental

5β-Epimer

Crystal data

$C_{21}H_{28}N_2O_2$	Cu $K\alpha$ radiation
$M_r = 340.46$	$\lambda = 1.5418$ Å
Trigonal	Cell parameters from 25
$P3_2$	reflections
$a = 15.034$ (2) Å	$\theta = 0-25°$
$c = 7.187$ (2) Å	$\mu = 0.58$ mm^{-1}
$V = 1406.8$ (5) Å^3	$T = 296$ K
$Z = 3$	Needle/prism
$D_x = 1.206$ Mg m^{-3}	$0.32 \times 0.11 \times 0.10$ mm
	Clear, colourless

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.021$
diffractometer	$\theta_{max} = 70°$
ω–θ scans	$h = -18 \rightarrow 9$
Absorption correction:	$k = 0 \rightarrow 18$
none	$l = -8 \rightarrow 6$
7378 measured reflections	3 standard reflections
3503 independent reflections	monitored every 100
3454 observed reflections	reflections
$[I > 3\sigma(I)]$	intensity decay: $<5\%$

Refinement

Refinement on F	$\Delta\rho_{max} = 0.31$ e Å^{-3}
$R = 0.048$	$\Delta\rho_{min} = -0.05$ e Å^{-3}
$wR = 0.052$	Extinction correction: none
3503 reflections	Atomic scattering factors
250 parameters	from *International Tables*
$w = 1/[\sigma^2(F) + 0.016534F^2]$	*for X-ray Crystallography*
$(\Delta/\sigma)_{max} = 0.682$	(1974, Vol. IV) and
	SHELX76 (Sheldrick, 1976)

5α-Epimer

Crystal data

$C_{21}H_{28}N_2O_2$	Cu $K\alpha$ radiation
$M_r = 340.46$	$\lambda = 1.5418$ Å
Orthorhombic	Cell parameters from 25
$P2_12_12_1$	reflections
$a = 11.949$ (1) Å	$\theta = 0-25°$
$b = 13.284$ (1) Å	$\mu = 0.59$ mm^{-1}
$c = 11.504$ (1) Å	$T = 296$ K

$V = 1826.0$ (2) Å^3
$Z = 4$
$D_x = 1.238$ Mg m^{-3}

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scans
Absorption correction: none
7632 measured reflections
3430 independent reflections
3257 observed reflections
$[I > 3\sigma(I)]$

Prism
$0.22 \times 0.20 \times 0.18$ mm
Yellow

$R_{int} = 0.042$
$\theta_{max} = 70°$
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 100 reflections
intensity decay: $<5\%$

Refinement

Refinement on F
$R = 0.042$
$wR = 0.050$
3257 reflections
260 parameters
$w = 1/[\sigma^2(F) + 0.005596F^2]$
$(\Delta/\sigma)_{max} = 0.579$

$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) and *SHELX76* (Sheldrick, 1976)

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

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

	x	y	z	U_{eq}
5β-Epimer				
C1	0.4566 (2)	0.2023 (1)	0.1628 (3)	0.0627 (4)
C2	0.5327 (2)	0.2340 (2)	0.3229 (4)	0.0693 (4)
C3	0.4819 (2)	0.1630 (2)	0.4825 (3)	0.0640 (3)
C4	0.3747 (1)	0.0923 (1)	0.4911 (3)	0.0526 (3)
C5	0.2995 (1)	0.0770 (1)	0.3413 (3)	0.0514 (3)
C6	0.1937 (1)	0.0463 (1)	0.4195 (3)	0.0550 (3)
C7	0.1946 (1)	0.1346 (1)	0.5269 (3)	0.0467 (2)
C8	0.2393 (1)	0.2337 (1)	0.4132 (3)	0.0390 (2)
C9	0.3465 (1)	0.2630 (1)	0.3377 (3)	0.0387 (2)
C10	0.3461 (1)	0.1760 (1)	0.2203 (3)	0.0470 (3)
C11	0.3974 (1)	0.3669 (1)	0.2370 (3)	0.0485 (3)
C12	0.4018 (1)	0.4534 (1)	0.3558 (3)	0.0440 (2)
C13	0.2953 (1)	0.4246 (1)	0.4258 (1)	0.0381 (2)
C14	0.2505 (1)	0.3227 (1)	0.5331 (2)	0.0389 (2)
C15	0.1556 (1)	0.3127 (1)	0.6308 (3)	0.0530 (3)
C16	0.1810 (1)	0.4245 (1)	0.6647 (3)	0.0560 (3)
C17	0.2894 (1)	0.4946 (1)	0.5800 (2)	0.0430 (2)
C18	0.2274 (1)	0.4198 (1)	0.2609 (3)	0.0526 (3)
C19	0.2845 (2)	0.1547 (2)	0.0408 (3)	0.0665 (4)
C20	0.3686 (1)	0.5195 (1)	0.7230 (3)	0.0470 (3)
C21	0.4297 (2)	0.5390 (2)	0.8425 (3)	0.0600 (3)
N1	0.3543 (2)	0.0453 (1)	0.6509 (3)	0.0655 (3)
N2	0.5267 (2)	0.1586 (2)	0.6349 (3)	0.0825 (4)
O1	0.3009 (1)	0.5876 (1)	0.5028 (2)	0.0563 (2)
O2	0.4482 (1)	0.0851 (2)	0.7418 (3)	0.0805 (3)
5α-Epimer				
C1	0.3799 (2)	0.4357 (1)	-0.0675 (1)	0.0498 (3)
C2	0.3493 (2)	0.4951 (1)	-0.1781 (1)	0.0601 (3)
C3	0.3694 (2)	0.4307 (1)	-0.2820 (1)	0.0535 (3)
C4	0.3902 (1)	0.3253 (1)	-0.2760 (1)	0.0450 (3)
C5	0.4041 (1)	0.2696 (1)	-0.1642 (1)	0.0405 (2)
C6	0.3781 (1)	0.1572 (1)	-0.1704 (1)	0.0486 (3)
C7	0.4079 (2)	0.1083 (1)	-0.0547 (1)	0.0509 (3)
C8	0.3507 (1)	0.1590 (1)	0.0489 (1)	0.0405 (2)
C9	0.3693 (1)	0.2746 (1)	0.0496 (1)	0.0365 (2)
C10	0.3390 (1)	0.3251 (1)	-0.0673 (1)	0.0366 (2)
C11	0.3115 (1)	0.3220 (1)	0.1555 (1)	0.0461 (3)
C12	0.3521 (1)	0.2768 (1)	0.2707 (1)	0.0469 (3)
C13	0.3380 (1)	0.1625 (1)	0.2715 (1)	0.0447 (2)
C14	0.3959 (1)	0.1182 (1)	0.1640 (1)	0.0431 (2)
C15	0.3947 (2)	0.0040 (1)	0.1860 (2)	0.0604 (3)
C16	0.4083 (2)	-0.0040 (1)	0.3199 (2)	0.0637 (3)
C17	0.4021 (1)	0.1046 (1)	0.3697 (1)	0.0489 (3)
C18	0.2140 (2)	0.1344 (2)	0.2787 (2)	0.0635 (4)
C19	0.2123 (1)	0.3218 (1)	-0.0903 (1)	0.0460 (3)
C20	0.5140 (1)	0.1462 (1)	0.3881 (1)	0.0518 (3)
C21	0.6024 (2)	0.1830 (2)	0.4105 (2)	0.0668 (4)
N1	0.3970 (2)	0.2892 (1)	-0.3803 (1)	0.0592 (3)
N2	0.3635 (2)	0.4580 (1)	-0.3909 (1)	0.0641 (3)
O1	0.3451 (1)	0.1013 (1)	0.4788 (1)	0.0659 (3)
O2	0.3813 (1)	0.3708 (1)	-0.4545 (1)	0.0676 (3)

Table 2. *Bond lengths* (Å), *bond angles and torsion angles* (°) *for the 5β- and 5α-epimers*

	5β	5α
C1—C2	1.521 (2)	1.541 (2)
C1—C10	1.559 (3)	1.548 (2)
C2—C3	1.490 (3)	1.488 (2)
C3—C4	1.420 (3)	1.424 (2)
C4—C5	1.494 (2)	1.493 (2)
C5—C6	1.525 (3)	1.526 (2)
C5—C10	1.555 (2)	1.547 (2)
C6—C7	1.531 (2)	1.523 (2)
C7—C8	1.529 (2)	1.530 (2)
C8—C9	1.543 (2)	1.552 (2)
C8—C14	1.529 (2)	1.530 (2)
C9—C10	1.554 (2)	1.545 (2)
C9—C11	1.535 (2)	1.535 (2)
C10—C19	1.526 (2)	1.538 (2)
C11—C12	1.529 (2)	1.533 (2)
C12—C13	1.520 (2)	1.527 (2)
C13—C14	1.537 (2)	1.535 (2)
C13—C17	1.561 (2)	1.566 (2)
C13—C18	1.543 (2)	1.532 (2)
C14—C15	1.529 (2)	1.538 (2)
C15—C16	1.544 (2)	1.552 (2)
C16—C17	1.556 (2)	1.554 (2)
C17—C20	1.473 (2)	1.461 (2)
C20—C21	1.183 (3)	1.192 (3)
C17—O1	1.432 (2)	1.429 (2)
C3—N2	1.305 (3)	1.306 (2)
C4—N1	1.302 (2)	1.294 (2)
N2—O2	1.378 (3)	1.386 (2)
N1—O2	1.390 (3)	1.392 (2)
C1—C2—C3	108.9 (2)	109.3 (1)
C2—C3—C4	123.5 (2)	123.7 (1)
C3—C4—C5	124.9 (2)	123.3 (1)
C4—C5—C10	108.1 (1)	109.2 (1)
C5—C10—C1	107.8 (1)	107.0 (1)
C10—C1—C2	114.6 (1)	114.3 (1)
C5—C6—C7	111.6 (1)	109.2 (1)
C6—C7—C8	112.6 (1)	112.9 (1)
C7—C8—C9	110.7 (1)	112.1 (1)
C8—C9—C10	112.8 (1)	113.1 (1)
C9—C10—C5	108.2 (1)	107.6 (1)
C10—C5—C6	113.5 (1)	113.4 (1)
C8—C9—C11	112.0 (1)	110.2 (1)
C9—C11—C12	113.0 (1)	112.5 (1)
C11—C12—C13	110.6 (1)	111.1 (1)
C12—C13—C14	109.0 (1)	109.0 (1)
C13—C14—C8	113.0 (1)	113.7 (1)
C14—C8—C9	107.8 (1)	107.2 (1)
C13—C14—C15	104.6 (1)	104.0 (1)
C14—C15—C16	104.7 (1)	103.3 (1)
C15—C16—C17	106.3 (1)	107.3 (1)
C16—C17—C13	102.4 (1)	102.3 (1)
C17—C13—C14	99.8 (1)	99.9 (1)

C3—C4—N1	108.4 (2)	109.3 (2)
C4—N1—O2	105.6 (2)	105.7 (1)
N1—O2—N2	111.2 (1)	110.4 (1)
O2—N2—C3	104.6 (2)	105.4 (2)
N2—C3—C4	123.5 (2)	109.2 (2)
C2—C3—N2	126.2 (2)	127.0 (2)
N1—C4—C5	126.7 (2)	127.4 (2)
C4—C5—C6	112.1 (1)	115.0 (1)
C1—C10—C9	112.0 (1)	109.8 (1)
C19—C10—C1	106.6 (2)	109.7 (1)
C19—C10—C5	109.6 (1)	110.9 (1)
C19—C10—C9	112.5 (1)	111.6 (1)
C7—C8—C14	110.8 (1)	111.1 (1)
C10—C9—C11	113.1 (1)	114.0 (1)
C12—C13—C17	116.8 (1)	116.1 (1)
C18—C13—C12	109.8 (1)	110.5 (1)
C18—C13—C14	112.8 (2)	112.7 (1)
C18—C13—C17	108.5 (1)	108.3 (1)
C15—C14—C8	118.9 (1)	119.2 (1)
C13—C17—C20	110.8 (1)	111.5 (1)
C16—C17—C20	109.9 (1)	111.2 (2)
C17—C20—C21	177.7 (2)	175.2 (2)
O1—C17—C13	111.2 (1)	114.5 (1)
O1—C17—C16	112.8 (1)	108.5 (1)
O1—C17—C20	109.5 (1)	108.7 (1)
C1—C2—C3—C4	−11.6 (1)	12.2 (2)
C2—C3—C4—C5	2.0 (2)	−5.6 (2)
C3—C4—C5—C10	−22.8 (1)	27.2 (2)
C4—C5—C10—C1	50.8 (1)	−53.9 (1)
C5—C10—C1—C2	−66.1 (1)	66.1 (2)
C10—C1—C2—C3	44.0 (1)	−43.0 (2)
C5—C6—C7—C8	52.9 (1)	54.6 (2)
C6—C7—C8—C9	−53.6 (1)	−51.6 (2)
C7—C8—C9—C10	56.0 (1)	51.6 (2)
C8—C9—C10—C5	−55.3 (1)	−53.5 (1)
C9—C10—C5—C6	54.6 (1)	58.5 (1)
C10—C5—C6—C7	−54.2 (1)	−59.5 (1)
C8—C9—C11—C12	53.7 (1)	57.2 (1)
C9—C11—C12—C13	−54.5 (1)	−55.3 (1)
C11—C12—C13—C14	56.3 (1)	53.8 (1)
C12—C13—C14—C8	−60.7 (1)	−58.7 (1)
C13—C14—C8—C9	58.6 (1)	59.9 (2)
C14—C8—C9—C11	−53.8 (1)	−57.2 (1)
C13—C14—C15—C16	−29.4 (1)	−34.1 (2)
C14—C15—C16—C17	1.3 (1)	6.7 (2)
C15—C16—C17—C13	26.6 (1)	22.2 (2)
C16—C17—C13—C14	−43.6 (1)	−42.3 (1)
C17—C13—C14—C15	45.6 (1)	47.9 (1)

Table 3. *Asymmetry parameters (°) for the 5β and 5α epimers*

	5β	5α		5β	5α
Ring A					
$\Delta C_s(1)$	28.8	30.2	$\Delta C_2(1,2)$	52.2	51.0
$\Delta C_s(2)$	44.8	42.0	$\Delta C_2(2,3)$	43.0	38.1
$\Delta C_s(3)$	16.3	12.1	$\Delta C_2(3,4)$	9.2	13.1
Ring B					
$\Delta C_s(9)$	1.0	5.0	$\Delta C_2(6,7)$	1.1	7.4
$\Delta C_s(10)$	1.2	5.7	$\Delta C_2(5,10)$	2.4	4.8
$\Delta C_s(8)$	2.0	0.9	$\Delta C_2(5,6)$	1.7	3.0
Ring C					
$\Delta C_s(12)$	5.0	2.0	$\Delta C_2(11,12)$	5.2	2.6
$\Delta C_s(11)$	1.9	2.3	$\Delta C_2(11,9)$	1.7	4.4
$\Delta C_s(9)$	3.5	3.8	$\Delta C_2(9,8)$	5.7	3.0
Ring D					
$\Delta C_s(13)$	2.5	9.4	$\Delta C_2(16)$	20.5	12.3
$\Delta C_s(14)$	32.0	26.9			

*SHELX*76 (Sheldrick, 1976) was used for structure refinement. Torsion angles were calculated using the *XANADU* program (Roberts & Sheldrick, 1975) and molecular diagrams were produced using *SNPI* (Karaulov, 1993).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HR1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ein anorganischer Spirocyclus aus Cyclotriphosphazen und Phosphor(V)-Hydrazin-Heterocyclus mit Twist-Konformation

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Abstract

4,4,6,6-Tetrachloro-1',2',4',5'-tetramethyl-6'-phenoxy-6'-thioxocyclotriphosphazene-2-spiro-3'-cyclodi[λ^5-phosphadiazane], $C_{10}H_{17}Cl_4N_7OP_4S$, was synthesized in