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Dimeric 3-Vinylindoles as Potential Antitumor Active Compounds: 1,1,3,4-Tetramethyl-3-(1-methyl-1*H*-indol-3-yl)-1,2,3,4-tetrahydrocyclopenta[*b*]indole and 1,1,3-Trimethyl-4-phenylsulfonyl-3-(1-phenylsulfonyl-1*H*-indol-3-yl)-1,2,3,4-tetrahydrocyclopenta[*b*]indole

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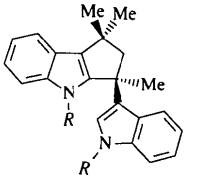
Abstract

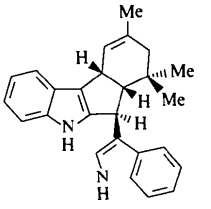
The structures of the title compounds, $C_{24}H_{26}N_2$ and $C_{34}H_{30}N_2O_4S_2$, have been determined from single-crystal X-ray diffraction data. The sterically more demanding phenylsulfonyl group of the second compound gives rise to significant differences, for example, in bond lengths and angles, in comparison with the first compound.

Comment

A variety of [*b*]annellated indoles are of biological interest as antitumor-active substances (Gribble, 1990). We are interested, therefore, in the synthesis and structure of functionalized carbazoles and cyclopentane-annellated indoles (Pindur, 1994; Schollmeyer, Fischer & Pindur, 1993) in order to obtain more information on structure–activity relationships. The compounds reported here, 1,1,3,4-tetramethyl-3-(1-methyl-1*H*-indol-3-yl)-1,2,3,4-tetrahydrocyclopenta-[*b*]indole, (1), and 1,1,3-trimethyl-4-phenylsulfonyl-3-(1-phenylsulfonyl-1*H*-indol-3-yl)-2,2,3,4-tetrahydrocyclopenta[*b*]indole, (2), are closely related to the highly antitumor-active tetrahydroindenoindole alkaloid yuehchuken, (3), the indolocyclopenta[*b*]indole unit of which is responsible for its biological activity (Cheng, Chan, Wong & Lai, 1990). Thus, we suggest that compounds (1) and (2) may reveal similar antitumor activity.



(1) R = Me
(2) $R = SO_2Ph$



(3)

For both compounds, the crystal packing shows no significant stacking interactions or hydrogen bonding. The structures of compounds (1) and (2) are shown in Figs. 1 and 2, respectively. The angle between the normals to the least-squares planes of the indole and cyclopenta[*b*]indole partial structures is 79.9° for compound (1) and 86.4° for (2). In both cases, the cyclopentene ring adopts a twist conformation, with atom C11 (IUPAC numbering: C2) lying on the same side of the cyclopenta[*b*]indole plane as the indole substituent and atom C12 (IUPAC numbering: C1) lying on the opposite side of this plane. The sterically more

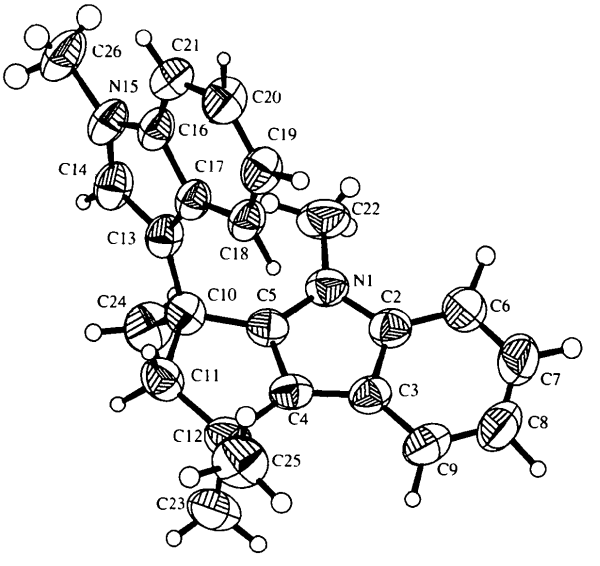


Fig. 1. *ORTEP*II plot (Johnson, 1976) of compound (1) with 50% probability ellipsoids and H atoms as spheres of arbitrary radii.

demanding phenylsulfonyl group of compound (2) leads to some significant differences in bond lengths and angles compared to compound (1). While the N atoms in (1) have an essentially planar environment, they show a small deviation from planarity for (2) (the sum of the angles around N1 is 357.3° and around N15 is 356.5°). The bulky substituents in compound (2) also distort the bond angles at each N atom, so that one *R*—N—C angle is larger than the other, thus relieving the steric crowding, whereas these angles are essentially equal in compound (1). There are some other small but significant differences in corresponding bond lengths and angles in the two structures, particularly in the five-membered rings and the link at atom C10.

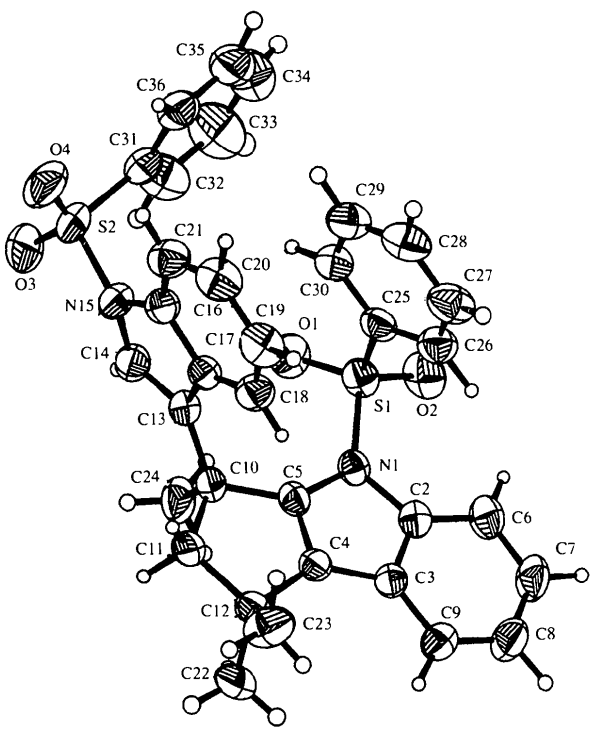


Fig. 2. *ORTEP*II plot (Johnson, 1976) of compound (2) with 50% probability ellipsoids and H atoms as spheres of arbitrary radii.

Experimental

The title compounds are readily available from the ethyl aluminium trichloride-catalyzed dimerization and cyclization of 1-phenylsulfonyl-3-propenylindole and 1-methyl-3-propenylindole (Pindur, 1994; Lutz, 1994). Crystals of (1) were obtained from chloroform, while crystals of (2) were obtained from deuterated chloroform by slow evaporation.

Compound (1)

Crystal data

$C_{24}H_{26}N_2$
M_r = 342.47
Cu $K\alpha$ radiation
λ = 1.54178 Å
Orthorhombic
$Pna2_1$
a = 12.8260 (5) Å
b = 16.9115 (4) Å
c = 9.0052 (3) Å
V = 1953.29 (11) Å^3
Z = 4
D_x = 1.165 Mg m^{-3}
Cell parameters from 75 reflections
θ = 65–70°
μ = 0.516 mm^{-1}
T = 293 (2) K
Needle
1.09 × 0.45 × 0.29 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1973 measured reflections
1973 independent reflections
1914 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 70.12°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 10$
3 standard reflections
frequency: 60 min
intensity decay: 5%

Refinement

Refinement on F^2
$R(F)$ = 0.0494
$wR(F^2)$ = 0.1383
S = 1.031
1973 reflections
253 parameters
H atoms refined as riding with grouped U
$w = 1/[\sigma^2(F_o^2) + (0.1189P)^2 + 0.09P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.205 e Å^{-3}
$\Delta\rho_{min}$ = −0.192 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0050 (9)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = 0.0 (8)

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

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

	x	y	z	U_{eq}
N1	−0.3098 (2)	0.01786 (12)	0.5743 (3)	0.0543 (6)
C2	−0.3520 (2)	−0.01776 (14)	0.6986 (3)	0.0552 (7)
C3	−0.3610 (2)	0.03949 (14)	0.8129 (3)	0.0538 (7)
C4	−0.3214 (2)	0.11099 (13)	0.7513 (3)	0.0510 (6)
C5	−0.2922 (2)	0.09598 (13)	0.6096 (3)	0.0491 (6)
C6	−0.3804 (2)	−0.0967 (2)	0.7188 (5)	0.0728 (10)
C7	−0.4178 (3)	−0.1177 (2)	0.8581 (6)	0.0882 (13)
C8	−0.4284 (2)	−0.0624 (2)	0.9713 (5)	0.0831 (11)
C9	−0.4011 (2)	0.0153 (2)	0.9513 (3)	0.0675 (9)
C10	−0.2463 (2)	0.16477 (13)	0.5263 (3)	0.0536 (6)
C11	−0.2366 (2)	0.22504 (14)	0.6594 (3)	0.0618 (8)
C12	−0.3045 (2)	0.1960 (2)	0.7928 (3)	0.0613 (8)
C13	−0.1414 (2)	0.14714 (12)	0.4583 (3)	0.0509 (6)
C14	−0.1079 (2)	0.1651 (2)	0.3172 (3)	0.0591 (7)
N15	−0.0064 (2)	0.14234 (13)	0.2973 (2)	0.0595 (6)
C16	0.0287 (2)	0.10789 (13)	0.4255 (3)	0.0514 (6)
C17	−0.0543 (2)	0.10946 (12)	0.5293 (3)	0.0465 (6)
C18	−0.0379 (2)	0.07636 (14)	0.6712 (3)	0.0514 (6)
C19	0.0572 (2)	0.0433 (2)	0.7030 (3)	0.0608 (7)
C20	0.1378 (2)	0.0422 (2)	0.5979 (4)	0.0666 (9)
C21	0.1246 (2)	0.0742 (2)	0.4586 (3)	0.0617 (8)
C22	−0.2954 (3)	−0.0188 (2)	0.4304 (4)	0.0777 (10)
C23	−0.4087 (3)	0.2404 (2)	0.8025 (5)	0.0824 (11)
C24	−0.3214 (3)	0.1973 (2)	0.4079 (4)	0.0729 (10)
C25	−0.2480 (3)	0.2057 (2)	0.9404 (4)	0.0860 (12)
C26	0.0517 (3)	0.1476 (2)	0.1590 (4)	0.0817 (11)

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

N1—C2	1.382 (3)	C10—C13	1.508 (3)
N1—C5	1.377 (3)	C13—C14	1.376 (4)
C2—C3	1.417 (4)	C13—C17	1.436 (3)
C3—C4	1.424 (3)	C14—N15	1.369 (3)
C4—C5	1.353 (3)	N15—C16	1.370 (3)
C5—C10	1.504 (3)	C16—C17	1.416 (3)
C5—N1—C2	107.2 (2)	C11—C12—C4	101.0 (2)
C22—N1—C2	126.0 (2)	C23—C12—C4	110.9 (2)
C22—N1—C5	126.6 (2)	C23—C12—C11	112.0 (2)
C3—C2—N1	108.8 (2)	C25—C12—C4	112.9 (2)
C4—C3—C2	105.5 (2)	C25—C12—C11	111.8 (2)
C5—C4—C3	107.9 (2)	C25—C12—C23	108.1 (3)
C12—C4—C5	112.0 (2)	C14—C13—C10	127.6 (2)
C4—C5—N1	110.6 (2)	C17—C13—C10	127.0 (2)
C10—C5—N1	133.7 (2)	C17—C13—C14	105.4 (2)
C10—C5—C4	115.7 (2)	N15—C14—C13	110.8 (2)
C11—C10—C5	98.7 (2)	C16—N15—C14	108.8 (2)
C13—C10—C5	113.5 (2)	C26—N15—C16	125.4 (2)
C13—C10—C11	111.4 (2)	C17—C16—N15	107.5 (2)
C24—C10—C5	112.1 (2)	C21—C16—C17	122.1 (2)
C24—C10—C11	110.2 (2)	C16—C17—C13	107.4 (2)
C24—C10—C13	110.3 (2)	C18—C17—C16	118.6 (2)
C12—C11—C10	109.6 (2)		
C22—N1—C2—C3	−175.0 (2)	C3—C4—C12—C25	50.3 (4)
C22—N1—C5—C10	−6.4 (3)	C11—C10—C13—C14	−114.9 (3)
N1—C5—C10—C24	74.4 (3)	C10—C13—C14—N15	177.8 (2)
N1—C5—C10—C13	−51.5 (3)	C13—C14—N15—C26	175.8 (3)
C3—C4—C12—C23	−71.3 (3)	C12—C4—C5—C10	2.2 (2)

Compound (2)

Crystal data

$C_{34}H_{30}N_2O_4S_2$
M_r = 594.72
Monoclinic
$P2_1/c$
a = 12.3400 (10) Å
b = 13.766 (2) Å
c = 17.1648 (9) Å
β = 92.258 (9)°
V = 2913.6 (5) Å^3
Z = 4
D_x = 1.356 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 75 reflections
θ = 25–29°
μ = 0.226 mm^{-1}
T = 295 (2) K
Plate
0.55 × 0.45 × 0.18 mm
Pale orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
8818 measured reflections
8463 independent reflections
4656 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0180
θ_{max} = 29.96°
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 19$
$l = -24 \rightarrow 24$
3 standard reflections
frequency: 60 min
intensity decay: 5%

Refinement

Refinement on F^2
$R(F)$ = 0.0502
$wR(F^2)$ = 0.1446
S = 1.008
8462 reflections
403 parameters
H atoms refined as riding with grouped U
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.9431P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.001

$\Delta\rho_{max}$ = 0.309 e Å^{-3}
$\Delta\rho_{min}$ = −0.301 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0034 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (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}
S1	0.36604 (5)	0.38282 (4)	0.80546 (3)	0.0445 (2)
S2	0.28681 (6)	0.81534 (4)	0.82162 (4)	0.0559 (2)
O1	0.41921 (14)	0.47438 (13)	0.80464 (10)	0.0584 (5)
O2	0.41763 (14)	0.30094 (14)	0.84209 (10)	0.0601 (5)
O3	0.3845 (2)	0.85284 (14)	0.79159 (12)	0.0776 (6)
O4	0.1959 (2)	0.87651 (13)	0.83301 (12)	0.0775 (6)
N1	0.33986 (14)	0.35053 (13)	0.71341 (9)	0.0366 (4)
C2	0.3213 (2)	0.2529 (2)	0.68724 (12)	0.0376 (5)
C3	0.2733 (2)	0.25758 (15)	0.61149 (12)	0.0375 (5)
C4	0.2601 (2)	0.35898 (15)	0.59289 (11)	0.0362 (4)
C5	0.2987 (2)	0.41247 (15)	0.65296 (11)	0.0354 (4)
C6	0.3462 (2)	0.1643 (2)	0.72233 (15)	0.0501 (6)
C7	0.3242 (2)	0.0811 (2)	0.6800 (2)	0.0576 (7)
C8	0.2782 (2)	0.0844 (2)	0.6051 (2)	0.0579 (7)
C9	0.2523 (2)	0.1713 (2)	0.57018 (14)	0.0482 (5)
C10	0.3137 (2)	0.51925 (15)	0.63430 (12)	0.0409 (5)
C11	0.2473 (2)	0.5223 (2)	0.55408 (12)	0.0534 (6)
C12	0.2303 (2)	0.4173 (2)	0.52152 (12)	0.0478 (6)
C13	0.2666 (2)	0.59259 (15)	0.68923 (12)	0.0385 (5)
C14	0.3175 (2)	0.6725 (2)	0.71765 (13)	0.0434 (5)
N15	0.2451 (2)	0.72986 (13)	0.75922 (11)	0.0456 (5)
C16	0.1450 (2)	0.6808 (2)	0.75917 (12)	0.0418 (5)
C17	0.1562 (2)	0.5961 (2)	0.71472 (12)	0.0383 (5)
C18	0.0661 (2)	0.5346 (2)	0.70412 (13)	0.0488 (6)
C19	−0.0289 (2)	0.5587 (2)	0.73787 (15)	0.0573 (7)
C20	−0.0375 (2)	0.6431 (2)	0.7818 (2)	0.0593 (7)
C21	0.0489 (2)	0.7055 (2)	0.79314 (14)	0.0531 (6)
C22	0.3075 (3)	0.3947 (2)	0.45565 (15)	0.0736 (9)
C23	0.1129 (2)	0.4001 (2)	0.4930 (2)	0.0712 (9)
C24	0.4352 (2)	0.5392 (2)	0.6235 (2)	0.0596 (7)
C25	0.2371 (2)	0.3990 (2)	0.84472 (11)	0.0431 (5)
C26	0.1616 (2)	0.3253 (2)	0.83867 (14)	0.0523 (6)
C27	0.0613 (2)	0.3390 (2)	0.86963 (15)	0.0639 (7)
C28	0.0375 (2)	0.4254 (2)	0.90619 (15)	0.0667 (8)
C29	0.1145 (3)	0.4981 (2)	0.91333 (15)	0.0650 (7)
C30	0.2150 (2)	0.4856 (2)	0.88216 (13)	0.0542 (6)
C31	0.3178 (2)	0.7538 (2)	0.90915 (14)	0.0505 (6)
C32	0.4195 (2)	0.7149 (3)	0.9219 (2)	0.0748 (9)
C33	0.4448 (3)	0.6686 (3)	0.9916 (2)	0.0908 (11)
C34	0.3688 (3)	0.6614 (3)	1.0474 (2)	0.0828 (10)
C35	0.2680 (3)	0.6988 (2)	1.0345 (2)	0.0715 (8)
C36	0.2405 (2)	0.7457 (2)	0.96518 (15)	0.0583 (7)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

N1—C5	1.421 (2)	C10—C13	1.513 (3)
N1—C2	1.432 (3)	C13—C14	1.349 (3)
C2—C3	1.409 (3)	C13—C17	1.448 (3)
C3—C4	1.440 (3)	C14—N15	1.407 (3)
C4—C5	1.339 (3)	N15—C16	1.408 (3)
C5—C10	1.517 (3)	C16—C17	1.404 (3)
C5—N1—C2	106.6 (2)	C4—C12—C11	100.3 (2)
C5—N1—S1	125.8 (1)	C23—C12—C11	111.7 (2)
C2—N1—S1	124.9 (1)	C22—C12—C11	111.8 (2)
C3—C2—N1	107.6 (2)	C14—C13—C17	107.1 (2)
C2—C3—C4	106.8 (2)	C14—C13—C10	125.8 (2)
C5—C4—C12	113.8 (2)	C17—C13—C10	126.9 (2)
C3—C4—C12	136.3 (2)	C13—C14—N15	110.1 (2)
C4—C5—N1	109.8 (2)	C14—N15—C16	107.8 (2)
C4—C5—C10	114.4 (2)	C14—N15—S2	122.5 (2)

N1—C5—C10	133.8 (2)	C16—N15—S2	126.2 (2)
C13—C10—C5	117.5 (2)	C21—C16—C17	122.5 (2)
C13—C10—C24	110.8 (2)	C17—C16—N15	107.1 (2)
C5—C10—C24	109.0 (2)	C18—C17—C16	118.5 (2)
C13—C10—C11	109.1 (2)	C16—C17—C13	107.8 (2)
C5—C10—C11	98.5 (2)	C20—C21—C16	117.3 (2)
C24—C10—C11	111.4 (2)	C26—C25—S1	119.6 (2)
C12—C11—C10	110.2 (2)	C30—C25—S1	118.8 (2)
C4—C12—C23	112.2 (2)	C32—C31—C36	120.5 (3)
C4—C12—C22	110.7 (2)	C36—C31—S2	120.1 (2)
C23—C12—C22	109.9 (2)		
O1—S1—N1—C2	158.0 (2)	C12—C4—C5—C10	−5.2 (2)
O2—S1—N1—C2	28.0 (2)	C5—C10—C11—C12	−16.8 (2)
C25—S1—N1—C2	−87.0 (2)	C3—C4—C12—C22	−56.6 (3)
N1—S1—C25—C26	53.6 (2)	C3—C4—C12—C23	66.6 (3)
O3—S2—N15—C16	−171.5 (2)	C11—C10—C13—C14	−114.8 (2)
O4—S2—N15—C16	−41.5 (2)	C10—C13—C14—N15	172.9 (2)
C31—S2—N15—C16	73.3 (2)	N1—C5—C10—C24	59.0 (2)
N15—S2—C31—C32	87.0 (2)	N1—C5—C10—C11	175.2 (2)

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CELSIUS* (Svenson, 1974); data reduction: *CORINC* (Dräger & Gattow, 1971; Wiehl & Schollmeyer, 1994). Program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990) for (1); *SIR92* (Altomare *et al.*, 1994) for (2). For both compounds, program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1009). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Thyrotropin-Releasing Hormone Analogue: pGlu-Phe-D-Pro-Ψ [CN_4]-NMe at 293 and 107 K

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Abstract

Data have been measured at two temperatures, 293 K and 107 K, for a crystal of a thyrotropin-releasing hormone analogue, pGlu-Phe-D-Pro-Ψ[CN_4]-NMe, $C_{20}H_{25}N_7O_3$, and the structures solved and refined. The tripeptide contains a tetrazole ring which mimics a *cis*-peptide bond at the C terminus. An intermolecular hydrogen bond exists between two molecules related by the twofold screw axis, resulting in infinite chains of hydrogen-bonded peptide molecules. Because of the folding and packing of the molecules, there are no intermolecular contacts of less than 4 Å to the N atom of the phenylalanine residue.

Comment

The title compound was prepared by solution methods in an ongoing evaluation of the 1,5-disubstituted tetrazole ring as a surrogate for *cis*-amide bonds (Marshall, Humblet, Van Opdenbosch & Zabrocki, 1981;