hydrogen bonded to nine adjacent molecules. Along the base vector [100] an infinite one-dimensional chain is formed. Fig. 2 shows the unit cell with two sets of D- and L-enantiomers. Part of the pattern of hydrogen bonds can be discerned in this figure.

In violation of the 'Wallach rule' the density D_x of the racemate is considerably smaller than that of the pure enantiomer (1.510 g cm^{-3}). This could be an artefact of the different temperatures of the measurements or of the overall inferior crystal quality of the investigated D-iditol. The final R value is 0.09 for only 697 reflexions (Azarnia, Jeffrey & Shen, 1972). However, similar deviations from the rule have been observed in the past, indicating some problems of the general applicability of the 'rule' (Jacques, Collet & Wilen, 1981, pp. 28–31).

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Structure of 3-(4-Chlorophenyl)-4-[2-(3,4-dimethoxyphenyl)ethyl]-4,5-dihydro-5-(2-methoxyphenyl)-1-phenyl-1,2,4-triazole

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Abstract. $C_{31}H_{30}ClN_3O_3$, $M_r = 528.06$, monoclinic, $P2_1/c$, $a = 10.793$ (3), $b = 19.317$ (3), $c = 14.038$ (5) Å, $\beta = 110.99$ (3)°, $V = 2733$ (3) Å^3, $Z = 4$, $D_x = 1.283$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.729$ cm^{-1}, $F(000) = 1112$, $T = 295$ K, $R = 0.043$ for 2403 independent observed reflections. The structure features a tetrasubstituted 1,2,4-triazole derivative.

Introduction. The nitrilimine (2) formed from α,4-dichlorobenzaldehyde phenylhydrazone (1) and triethylamine, reacts as a 1,3-dipole and adds to the imine (3) formed from 2-methoxybenzaldehyde and 2-(3,4-dimethoxyphenyl)ethylamine, to give a new crystalline triazole A. As the cycloaddition may occur in two ways, A is either a 1,2,4- or a 1,2,3-triazole. In the first reported example of this type of reaction (Huisgen, Grashey, Knupfer, Kunz & Seidel, 1964), a 1,2,4-triazole structure was reported for the adduct obtained from diphenylnitrilimine and benzalaniline, as the adduct was found to be identi-

cal with the product from (α-anilinobenzal)phenylhydrazine and benzaldehyde (Busch & Ruppenthal, 1910), which was considered to be a 1,2,4-triazole. Since it is conceivable that the reactive (α-anilinobenzal)phenylhydrazine could rearrange *via* a triazetidine and form a 1,2,3-triazole, a more convincing proof seems necessary for triazoles formed from imine–nitrilimine cycloadditions. Furthermore, other imines and nitrilimines could cyclize in different ways. The 1,2,4-triazole structure (4) has now been confirmed for A in this X-ray crystallographic study. To the best of our knowledge no crystallographic structural investigations have been carried out on substituted 4,5-dihydro-1,2,4-triazoles.

(1)   (2)   (3)   (4)
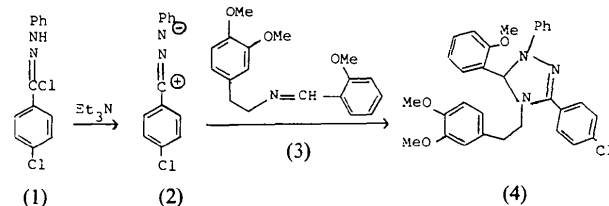
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Experimental. The title compound (4) was obtained in 75% yield from the reaction between (2) and (3) in the presence of triethylamine, and was crystallized from 95% ethanol.

A yellow crystal of dimensions $0.2 \times 0.15 \times 0.3$ mm was selected for data collection using an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, and scintillation counter. Cell dimensions by least squares from the setting angles of 25 reflections ($11 < 2\theta < 15°$) measured on the diffractometer. The intensities of 7347 reflections ($2\theta_{max} = 48°$), in the range $0 \le h \le 12$, $-22 \le k \le 22$, $-16 \le l \le 16$ were measured, using the ω–2θ scan, ω scan angle $(0.65 + 0.344\tan\theta)°$ at 0.82 to $5.49°$ min^{-1}, extended 25% on each side for background measurement. Three standard reflections measured every 2 h showed no decay. Systematic absences indicated $P2_1/c$ and this was confirmed in the structure solution. The intensity data were corrected for Lp effects and empirical absorption (minimum correction factor 0.9843, maximum correction factor 0.9986). After equivalent reflections were averaged, the 7347 measured reflections gave 3632 independent reflections, of which 2408 with $I > 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2)$, $S = $ scan, B_1 and $B_2 = $ background counts, were considered observed. $R_{int} = 0.016$ for observed reflections and 0.021 for all reflections. The structure was determined by direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares (on F) with atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), using the *SDP* programs (Enraf–Nonius, 1985) on a MicroVAXII computer. The C, Cl, N and O atoms were refined anisotropically; H atoms

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the molecule (4) with numbering of atoms.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl	0.4430 (1)	0.18564 (6)	0.84840 (7)	7.74 (3)
O1	0.7008 (2)	-0.0167 (1)	1.3999 (2)	5.16 (5)
O2	0.7880 (2)	0.0935 (1)	1.3417 (2)	5.44 (5)
O3	0.0506 (2)	-0.0647 (1)	1.3085 (1)	5.11 (5)
N1	0.2082 (2)	-0.0255 (1)	1.1263 (2)	3.52 (5)
N2	0.1323 (2)	-0.1313 (1)	1.0599 (2)	4.25 (6)
N3	0.1877 (2)	-0.0979 (1)	0.9946 (2)	4.25 (6)
C1	0.6635 (4)	-0.0708 (2)	1.4514 (3)	6.5 (1)
C2	0.8406 (4)	0.1530 (2)	1.3085 (4)	9.0 (1)
C3	0.0095 (3)	-0.0468 (2)	1.3914 (2)	6.13 (9)
C4	0.2371 (3)	0.0663 (2)	1.2592 (2)	4.39 (7)
C5	0.1672 (3)	0.0434 (2)	1.1477 (2)	4.15 (7)
C6	0.1334 (3)	-0.0850 (2)	1.1430 (2)	3.88 (7)
C7	0.2273 (3)	-0.0385 (1)	1.0345 (2)	3.73 (7)
C8	0.2006 (3)	-0.1192 (1)	1.2465 (2)	3.70 (7)
C9	0.3081 (3)	-0.1618 (2)	1.2624 (2)	4.63 (8)
C10	0.3746 (3)	-0.1918 (2)	1.3567 (3)	5.69 (9)
C11	0.3321 (4)	-0.1789 (2)	1.4357 (3)	5.93 (9)
C12	0.2247 (3)	-0.1374 (2)	1.4219 (2)	5.23 (8)
C13	0.1583 (3)	-0.1070 (2)	1.3283 (2)	4.02 (7)
C14	0.0281 (3)	-0.1773 (1)	1.0140 (2)	4.05 (7)
C15	-0.0600 (3)	-0.1939 (2)	1.0617 (3)	5.15 (8)
C16	-0.1651 (4)	-0.2397 (2)	1.0129 (3)	6.4 (1)
C17	-0.1805 (4)	-0.2678 (2)	0.9199 (3)	6.9 (1)
C18	-0.0903 (4)	-0.2522 (2)	0.8763 (3)	6.4 (1)
C19	0.0133 (3)	-0.2074 (2)	0.9207 (2)	5.21 (8)
C20	0.2902 (3)	0.0119 (2)	0.9886 (2)	3.71 (7)
C21	0.2388 (3)	0.0227 (2)	0.8841 (2)	4.55 (7)
C22	0.2882 (3)	0.0741 (2)	0.8394 (2)	5.31 (8)
C23	0.3902 (3)	0.1150 (2)	0.9008 (2)	4.66 (7)
C24	0.4482 (3)	0.1030 (2)	1.0039 (2)	4.67 (8)
C25	0.3982 (3)	0.0511 (2)	1.0471 (2)	4.29 (7)
C26	0.3836 (3)	0.0773 (2)	1.2870 (2)	3.86 (7)
C27	0.4324 (3)	0.1378 (2)	1.2626 (2)	4.66 (8)
C28	0.5674 (3)	0.1452 (2)	1.2795 (2)	4.95 (8)
C29	0.6538 (3)	0.0921 (2)	1.3227 (2)	4.14 (7)
C30	0.6068 (3)	0.0319 (2)	1.3516 (2)	3.80 (7)
C31	0.4733 (3)	0.0245 (2)	1.3331 (2)	3.97 (7)

in calculated positions were not refined. Convergence for 343 variables by least squares with $w = 4F_o^2/\sigma^2(F_o^2)$, where $\sigma(F_o^2) = [\sigma^2(I) + (0.055F_o^2)^2]^{1/2}$ and reflections with $F_o^2 < 1.5\sigma(F_o^2)$ given negative weights and omitted in the refinement, was reached at $R = 0.043$ and $wR = 0.060$ and $S = 1.620$ for 2408 reflections. $(\Delta/\sigma)_{max} = 0.05$. A final difference Fourier map was featureless, with maximum positive and negative peaks of 0.29 and -0.35 e Å^{-3}.

Discussion. An *ORTEP*II drawing (Johnson, 1976) of the molecule with atomic numbering scheme is shown in Fig. 1. The atomic parameters of the C, Cl, N and O atoms are listed in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2.

In the 1,2,4-triazole ring, the C—N bonds involving C6 are comparable [N1—C6 = 1.472 (4) and N2—C6 = 1.466 (4) Å] while those involving C7

$C_{31}H_{30}ClN_3O_3$

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

Cl—C23	1.740 (3)	C10—C11	1.367 (6)
O1—C1	1.409 (5)	C11—C12	1.365 (6)
O1—C30	1.371 (3)	C12—C13	1.383 (4)
O2—C2	1.431 (5)	C14—C15	1.384 (5)
O2—C29	1.377 (4)	C14—C19	1.388 (5)
O3—C3	1.429 (4)	C15—C16	1.408 (4)
O3—C13	1.367 (4)	C16—C17	1.368 (5)
N1—C5	1.466 (4)	C17—C18	1.356 (6)
N1—C6	1.472 (4)	C18—C19	1.374 (5)
N1—C7	1.398 (4)	C20—C21	1.386 (4)
N2—N3	1.417 (4)	C20—C25	1.386 (4)
N2—C6	1.466 (4)	C21—C22	1.379 (5)
N2—C14	1.396 (3)	C22—C23	1.379 (5)
N3—C7	1.281 (4)	C23—C24	1.376 (4)
C4—C5	1.539 (4)	C24—C25	1.377 (5)
C4—C26	1.502 (5)	C26—C27	1.376 (4)
C6—C8	1.524 (3)	C26—C31	1.395 (4)
C7—C20	1.463 (4)	C27—C28	1.397 (5)
C8—C9	1.374 (4)	C28—C29	1.372 (5)
C8—C13	1.398 (4)	C29—C30	1.388 (4)
C9—C10	1.387 (4)	C30—C31	1.378 (4)

C1—O1—C30	117.7 (3)	N2—C14—C19	120.3 (4)
C2—O2—C29	117.7 (2)	C15—C14—C19	119.5 (3)
C3—O3—C13	118.0 (2)	C14—C15—C16	119.0 (3)
C5—N1—C6	116.9 (2)	C15—C16—C17	121.0 (4)
C5—N1—C7	120.5 (2)	C16—C17—C18	118.8 (3)
C6—N1—C7	106.1 (3)	C17—C18—C19	122.2 (4)
N3—N2—C6	110.6 (2)	C14—C19—C18	119.5 (4)
N3—N2—C14	116.6 (2)	C7—C20—C21	119.6 (2)
C6—N2—C14	122.0 (3)	C7—C20—C25	121.8 (2)
N2—N3—C7	106.1 (2)	C21—C20—C25	118.5 (3)
C5—C4—C26	113.1 (3)	C20—C21—C22	121.1 (3)
N1—C5—C4	113.3 (3)	C21—C22—C23	118.7 (3)
N1—C6—N2	101.4 (2)	Cl—C23—C22	119.7 (2)
N1—C6—C8	113.0 (2)	Cl—C23—C24	118.9 (2)
N2—C6—C8	111.2 (2)	C22—C23—C24	121.4 (4)
N1—C7—N3	115.4 (3)	C23—C24—C25	119.0 (3)
N1—C7—C20	122.1 (2)	C20—C25—C24	121.1 (3)
N3—C7—C20	122.5 (3)	C4—C26—C27	121.1 (2)
C6—C8—C9	120.0 (3)	C4—C26—C31	120.6 (3)
C6—C8—C13	121.8 (3)	C27—C26—C31	118.2 (3)
C9—C8—C13	118.2 (2)	C26—C27—C28	120.9 (3)
C8—C9—C10	121.6 (3)	C27—C28—C29	120.1 (3)
C9—C10—C11	119.3 (3)	O2—C29—C28	124.9 (3)
C10—C11—C12	120.4 (3)	O2—C29—C30	115.5 (2)
C11—C12—C13	120.7 (3)	C28—C29—C30	119.6 (3)
O3—C13—C8	116.1 (2)	O1—C30—C29	115.9 (3)
O3—C13—C12	124.2 (3)	O1—C30—C31	124.1 (3)
C8—C13—C12	119.9 (3)	C29—C30—C31	120.0 (3)
N2—C14—C15	120.3 (3)	C26—C31—C30	121.1 (3)

C1—O1—C30—C29	165.5 (3)	C5—N1—C7—C20	39.8 (4)
C1—O1—C30—C31	−13.2 (4)	C6—N1—C7—N3	−5.4 (3)

Table 2 (*cont.*)

C2—O2—C29—C28	−4.0 (4)	C6—N1—C7—C20	175.6 (3)
C2—O2—C29—C30	176.7 (3)	C6—N2—N3—C7	3.5 (3)
C3—O3—C13—C8	−175.6 (3)	C14—N2—N3—C7	148.8 (3)
C3—O3—C13—C12	4.5 (4)	N3—N2—C6—N1	−6.4 (3)
C6—N1—C5—C4	88.6 (3)	N3—N2—C6—C8	−126.7 (2)
C7—N1—C5—C4	140.1 (3)	C14—N2—C6—N1	−149.4 (2)
C5—N1—C6—N2	144.3 (2)	C14—N2—C6—C8	90.3 (3)
C5—N1—C6—C8	−96.7 (3)	N2—N3—C7—N1	1.3 (3)
C7—N1—C6—N2	6.7 (3)	N2—N3—C7—C20	−179.7 (3)
C7—N1—C6—C8	125.7 (3)	C26—C4—C5—N1	66.2 (3)
C5—N1—C7—N3	−141.2 (3)		

are different [N1—C7 = 1.398 (4) and N3—C7 = 1.281 (4) Å], indicating the presence of a C7—N3 double bond. Although the triazole ring is planar within experimental error, the best least-squares plane is through the group N1—C7=N3—N2 with C6 deviating 0.107 (2) Å from it. The values of 1.466 (4) Å for N1—C5 and 1.396 (3) Å for N2—C14 can be compared with values of 1.452 (2) and 1.417 (2) Å for similar bonds in 5,5-dimethyl-4-(2-methylallyl)-2-tolyl-1,2,4-triazolidine-3-thione (Schulze, Richter & Faure, 1988).

All intermolecular contacts correspond to van der Waals interactions.

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Structure of (1*R*,2*S*)-(−)-2-(Benzylamino)-1-(*tert*-butyldimethylsiloxy)-1-phenyl-propane Hydrochloride

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Abstract. $C_{22}H_{34}NOSi^+.Cl^-$, $M_r = 381.98$, orthorhombic, $P2_12_12_1$, $a = 8.3358$ (6), $b = 14.3567$ (5), $c = 19.8068$ (7) Å, $V = 2370.4$ (3) Å^3, $Z = 4$, $D_x = 1.07$ g cm^{-3}, λ(Mo $K\alpha$) = 0.710730 Å, $\mu = 2.17$ cm^{-1}, $F(000) = 848$, $T = 293$ K, final $R = 0.0372$ for 1105 significant reflections. The asymmetric unit

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