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Studies on Antifungal Agents. 29. Structure of *N*-[1-(4-Chlorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethylidene]methylamine *N*-Oxide

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Abstract. C₁₁H₁₁ClN₄O, *M_r* = 250.69, monoclinic, *P*2₁/*c*, *a* = 8.999 (2), *b* = 15.171 (1), *c* = 8.933 (1) Å, β = 93.78 (1)°, *V* = 1216.8 Å³, *Z* = 4, *D_x* = 1.368 g cm⁻³, λ(Cu *Kα*) = 1.54184 Å, μ = 27.3 cm⁻¹, *F*(000) = 520, *T* = 297 K, final *R* = 0.044 for 1768 unique observed reflections. The title compound is an important intermediate in the synthesis of 3-aryl-5-(aryloxyalkyl)-3-(1*H*-imidazol-1-ylmethyl)-2-methylisoxazolidines; the results of the X-ray structure determination confirm the *E* configuration of this intermediate.

Experimental. Crystal 0.12 × 0.25 × 0.30 mm, Enraf-Nonius CAD-4 diffractometer with Ni-filtered Cu *Kα* radiation. Lattice parameters from 22 reflections with 48 ≤ 2θ ≤ 65°. 2310 reflections measured using the ω–2θ scan technique within ranges 6 ≤ 2θ ≤ 130°, –10 ≤ *h* ≤ 0, –17 ≤ *k* ≤ 0, –10 ≤ *l* ≤ 10. Intensities of three standard reflections (112̄, 211̄, 102) recorded every 3500 s of X-ray exposure showed no significant decay. 1768 unique observed reflections [*I* > 3σ(*I*), *R*_{int} = 0.017]. Data corrected for Lorentz and polarization effects but not for absorption. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H atoms found from subsequent difference Fourier syntheses. Refinement by full-matrix least squares to minimize ∑w(|*F_o*| – |*F_c*|)² led to *R* = 0.044 and *wR* = 0.062 for 154 variables with *w* = 1/σ²(*F_o*) and *S* = 2.12. Non-H atoms refined anisotropically; H atoms not refined but included as constant contributions to structure factors. Maximum least-squares shift to e.s.d. ratio 0.02 in the final refinement cycle. Largest residual electron densities in the final difference

Fourier synthesis 0.23 and –0.45 e Å⁻³. Atomic scattering factors from Cromer & Waber (1974); *SDP* package (Frenz, 1978).† Positional parameters and equivalent isotropic thermal parameters are given in Table 1.

Related literature. Previously (Mullen, Swift, Maryniak, Allen, Mitchell, Kinsolving & Georgiev, 1988), we described the synthesis of a novel series of systemic antifungal agents, the 3-aryl-5-(aryloxyalkyl)-3-(1*H*-imidazol-1-ylmethyl)-2-methylisoxazolidines (3) via a 1,3-dipolar cycloaddition reaction of α-substituted

† Lists of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51591 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Refined positional parameters

$$B_{\text{eq}} = \frac{1}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}abc\cos\gamma + \beta_{13}acc\cos\beta + \beta_{23}bcc\cos\alpha)$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i> (Å ²)
Cl	0.44963 (6)	0.10235 (4)	0.17459 (6)	5.79 (1)
C(1)	0.3297 (2)	0.1184 (1)	0.0155 (2)	4.08 (4)
C(2)	0.2468 (3)	0.0486 (1)	–0.0434 (3)	4.89 (5)
C(3)	0.1582 (3)	0.0606 (1)	–0.1740 (3)	4.57 (4)
C(4)	0.1520 (2)	0.1420 (1)	–0.2452 (2)	3.40 (3)
C(5)	0.2311 (2)	0.2126 (1)	–0.1797 (2)	3.60 (4)
C(6)	0.3217 (2)	0.2006 (1)	–0.0491 (2)	3.89 (4)
C(7)	0.0611 (2)	0.1572 (1)	–0.3866 (2)	3.40 (3)
C(8)	–0.0571 (2)	0.2276 (1)	–0.3978 (2)	3.83 (4)
C(9)	–0.3142 (2)	0.1755 (2)	–0.4879 (3)	4.86 (5)
C(10)	–0.3817 (2)	0.1307 (2)	–0.2837 (3)	5.26 (5)
C(11)	0.2019 (2)	0.0466 (1)	–0.5190 (3)	4.48 (4)
N(1)	0.0794 (2)	0.11086 (9)	–0.5075 (2)	3.41 (3)
N(2)	–0.2052 (2)	0.1903 (1)	–0.3829 (2)	3.71 (3)
N(3)	–0.4291 (2)	0.1383 (1)	–0.4299 (3)	5.69 (5)
N(4)	–0.2459 (2)	0.1608 (1)	–0.2483 (2)	4.98 (4)
O(1)	–0.0073 (2)	0.11985 (9)	–0.6288 (2)	4.26 (3)

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ketonitrone (1) and 1-alkenyl phenyl ethers (2). The resulting products represented *cis/trans* mixtures of the corresponding diastereomers of (3). While cyclic nitrones do not present configurational problems, acyclic ketonitrone are known to exist in either *E* or *Z* geometric form (Tufariello, 1984). Based on NMR spectral evidence, an *E* configuration was assigned for the α -substituted ketonitrone (1). An X-ray crystal structure determination of the triazole analog *N*-[1-(4-chlorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethylidene]-methylamine *N*-oxide (1*a*) (Fig. 1) was undertaken in order to firmly establish the configuration of (1) and allow for consistent differentiation between the *E* and *Z* isomers of (1) by NMR spectral interpretation.

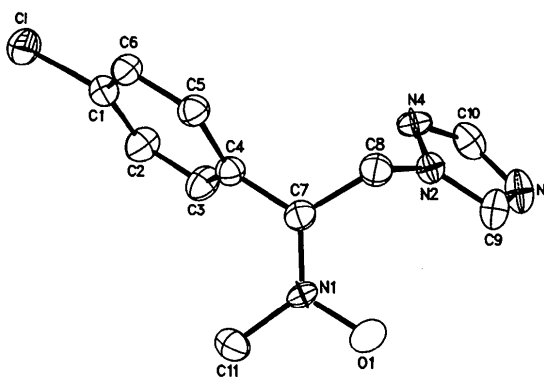
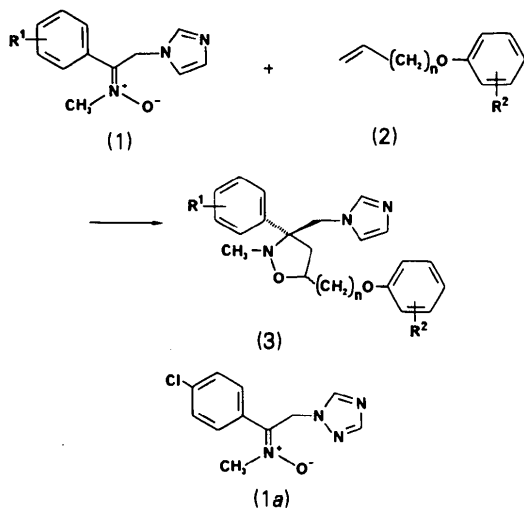


Fig. 1. Drawing of the molecule of (1*a*) showing the atom-numbering scheme.



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Structure of L-Arginyl-L-aspartic Acid Monohydrate

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Abstract. C₁₀H₁₉N₅O₅·H₂O, *M_r* = 309·32, monoclinic, *P*2₁, *a* = 8·396 (2), *b* = 8·811 (2), *c* = 9·940 (2) Å, β = 100·71 (2)°, *V* = 723 (1) Å³, *Z* = 2, *D_m* = 1·45, *D_x* = 1·42 g cm⁻³, Mo *K*α, λ = 0·71073 Å, μ = 1·1 cm⁻¹, *F*(000) = 328, *T* = 293 K. The final *R* value for 1289 observed reflections [*I_o* ≥ 3σ(*I_o*)] is 0·032. The dipeptide exists as a zwitterion. The peptide bond is planar and the backbone assumes an extended conformation. The two terminal N atoms of the guanidino group are hydrogen bonded to the carbonyl

O atom and to one of the terminal carboxyl O atoms of the peptide backbone, a mode of interaction not found in earlier related structures.

Experimental. The dipeptide was purchased from Sigma Chemicals and used without further purification. Prismatic crystals of the peptide were grown by diffusing acetone into a solution of 50% water/50% dioxane. The size of the crystal used for X-ray data collection was 0·2 × 0·2 × 0·2 mm. Density was