

refined by full-matrix least squares minimizing the function $\sum w(|F_o| - |F_c|)^2$ with *SHELX76* (Sheldrick, 1976). Final *R* and *wR* values were 0.044 and 0.069, respectively with $w = 0.260/[\sigma^2(F_o) + 0.0094F_o^2]$ and 293 variables. The H-atom positions were found in $\Delta\rho$ map and included in the refinement with a constraint imposed on the C—H bonds. The common isotropic temperature factor *U* for methyl H atoms was 0.151 (9) Å² and for other H atoms was 0.071 (4) Å². The final $\Delta\rho$ showed residual electron density within +0.12 and -0.08 e Å⁻³. Max. value of Δ/σ at convergence was 0.62 (*z*, H142). Scattering factors for C, N and O were from *International Tables for X-ray Crystallography* (1974, Vol. IV), for H from Stewart, Davidson & Simpson (1965). All calculations performed on the VAX 8550 under VMS 5.2 at University Computer Centre, Ljubljana. Additionally, the *GX* (Mallinson & Muir, 1985) package was used for data processing and final interpretation of molecular geometry. The final atomic parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Views of the molecule with the atomic numbering and of the molecular packing are presented in Figs. 1 and 2.

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, selected torsion angles and the results of calculated mean-planes angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53890 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Recently Stanovnik, Svete & Tišler (1987), Stanovnik, Svete, Tišler, Žorž, Hvala & Simonič, (1988) and Svete, Stanovnik, Tišler, Golič & Leban (1989) have described the syntheses of β -heteroaryl amino- α,β -dehydro- α -amino acids and their derivatives from *N*-heteroaryl formamidines and 2-phenyl-5-oxo-1,3-oxazole in which 4-heteroarylaminomethylene-2-phenyl-5(4*H*)-oxazolones are formed as novel intermediates.

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Structure of Methyl (4*aS*,5*R*,7*aR*,11*aR*,12*aS*)-2-Benzyl-2,3,4,4*a*,5,6,7,7*a*,8,10,11*a*,12-dodecahydro-7,7-(ethylenedioxy)-1,9-dioxooxazolo[3',4':1,2]pyrrolo[5,4-*i*]isoquinoline-5-carboxylate Toluene (2/1)

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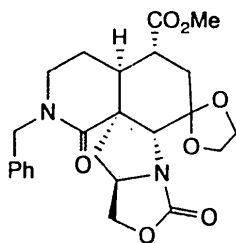
(Received 23 October 1990; accepted 8 January 1991)

Abstract. 2C₂₄H₂₈N₂O₇·C₇H₈, *M_r* = 502.57, triclinic, *P*1, *a* = 11.076 (1), *b* = 11.314 (1), *c* = 12.155 (1) Å, α = 104.41 (1), β = 102.02 (1), γ = 89.37 (1)°, *V* = 1441.6 (3) Å³, *Z* = 1, *D_x* = 1.16 g cm⁻³, $\lambda(\text{Cu K}\alpha)$ = 1.5418 Å, μ = 6.55 cm⁻¹, *F*(000) = 534, room temperature, final *R* = 0.106 for 5273 observed reflections. The asymmetric unit contains two independent molecules, *A* and *B*, of the title compound C₂₄H₂₈-

N₂O₇ plus one toluene molecule. Molecules *A* and *B* show similar conformations except for different orientations of the carboxylate group [O(6)—C(16)—O(7)—C(17)]: torsion angles C(3)—C(4)—C(16)—O(6) are -62 (2)° and +111 (1)° for *A* and *B* respectively. Excluding the carboxylate group least-squares matching of the positions of corresponding non-H atoms of *A* and *B* yields a r.m.s. deviation of 0.20 Å.

The untypical geometry and high U values of the toluene molecule suggest disorder. The disorder could not be modelled and this probably accounts for the high final R factor.

Experimental. A brick-shaped crystal of compound (I) (approximate dimensions $0.86 \times 0.50 \times 0.45$ mm) was used for data collection on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation and θ - 2θ scan. A total of 5454 unique reflections was measured within the range $-13 \leq h \leq 13$, $0 \leq k \leq 13$, $-14 \leq l \leq 14$. Of these, 5273 were above the significance level of $2.5\sigma(I)$. The maximum value of $(\sin\theta)/\lambda$ was 0.61 \AA^{-1} . Two standard reflections ($\bar{2}13$, $1\bar{3}2$) were measured hourly, no significant decrease was observed during the 51 h collecting time. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with $80 < 2\theta < 89^\circ$. Corrections for Lorentz and polarization effects were applied. The structure was solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) which revealed about half of the structure: a large part of one molecule and a small part of the other molecule. These fragments were used as input for the program *AUTOFOUR* (Kinneking & de Graaff, 1984). Using an iterative procedure consisting of a $2F_{\text{obs}} - F_{\text{calc}}$ Fourier synthesis and an R_2 factor based peak selection, the program completed both fragments and revealed the solvent molecule.



The positions of the H atoms were calculated. Anisotropic block-diagonal least-squares refinement on F , isotropic for the solvent molecule, keeping the H atoms fixed at their calculated positions with $U = 0.07 \text{ \AA}^2$, converged to $R = 0.106$, $wR = 0.142$, $(\Delta/\sigma)_{\text{max}} = 0.57$, origin defined by fixing C(1A), $w = (1.5 + F_{\text{obs}} + 0.02F_{\text{obs}}^2)^{-1}$. An empirical absorption correction was applied, with corrections in the range 0.80–1.54 (Walker & Stuart, 1983). The isotropic secondary-extinction coefficient refined to 0.20 (2) (Zachariasen, 1968). A final difference Fourier map revealed a residual electron density between -0.4 and 1.2 e \AA^{-3} . Scattering factors were taken from Cromer & Mann (1968). Anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). The absolute

Table 1. Fractional coordinates of the non-H atoms and equivalent isotropic thermal parameters

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}}(\text{\AA}^2)$
Molecule A				
C(1A)	0.0	0.0	0.0	0.055 (8)
C(2A)	0.023 (1)	-0.134 (1)	-0.017 (1)	0.044 (6)
C(3A)	-0.0543 (9)	-0.197 (1)	0.0395 (9)	0.034 (5)
C(4A)	-0.021 (1)	-0.151 (1)	0.1733 (9)	0.039 (6)
C(5A)	-0.102 (1)	-0.217 (1)	0.229 (1)	0.044 (6)
C(6A)	-0.234 (1)	-0.184 (1)	0.1944 (9)	0.038 (6)
C(7A)	-0.2840 (9)	-0.2158 (9)	0.061 (1)	0.038 (6)
C(8A)	-0.1931 (9)	-0.1861 (9)	-0.0121 (8)	0.033 (5)
C(9A)	-0.227 (1)	-0.060 (1)	-0.033 (1)	0.043 (6)
C(10A)	-0.231 (1)	-0.287 (1)	-0.127 (1)	0.044 (6)
C(11A)	-0.246 (1)	-0.398 (1)	-0.080 (1)	0.045 (6)
C(12A)	-0.333 (1)	-0.503 (1)	-0.159 (1)	0.068 (9)
C(13A)	-0.436 (1)	-0.384 (1)	-0.031 (1)	0.055 (8)
C(14A)	-0.344 (2)	-0.034 (2)	0.290 (2)	0.11 (1)
C(15A)	-0.391 (2)	-0.155 (2)	0.291 (2)	0.13 (2)
C(16A)	0.115 (1)	-0.176 (1)	0.216 (1)	0.053 (7)
C(17A)	0.310 (2)	-0.095 (2)	0.308 (2)	0.14 (2)
C(18A)	-0.164 (2)	0.140 (1)	-0.052 (1)	0.069 (9)
C(19A)	-0.105 (1)	0.245 (1)	0.048 (1)	0.052 (7)
C(20A)	-0.102 (2)	0.248 (1)	0.162 (1)	0.08 (1)
C(21A)	-0.047 (2)	0.345 (2)	0.253 (1)	0.09 (1)
C(22A)	0.007 (2)	0.446 (1)	0.220 (2)	0.10 (1)
C(23A)	0.002 (2)	0.440 (1)	0.103 (1)	0.08 (1)
C(24A)	-0.048 (1)	0.343 (1)	0.024 (1)	0.061 (8)
N(1A)	-0.134 (1)	0.0198 (9)	-0.0276 (9)	0.050 (6)
N(2A)	-0.3139 (8)	-0.3508 (9)	0.0112 (9)	0.046 (5)
O(1A)	-0.3363 (8)	-0.0368 (8)	-0.0608 (8)	0.059 (6)
O(2A)	-0.4512 (9)	-0.4787 (9)	-0.1264 (9)	0.065 (6)
O(3A)	-0.5242 (8)	-0.341 (1)	0.0085 (9)	0.071 (6)
O(4A)	-0.2412 (8)	-0.0525 (8)	0.2337 (8)	0.056 (5)
O(5A)	0.3091 (9)	0.236 (1)	0.2514 (8)	0.065 (6)
O(6A)	0.1542 (9)	-0.276 (1)	0.203 (1)	0.079 (7)
O(7A)	0.1801 (9)	-0.074 (1)	0.269 (1)	0.087 (7)
Molecule B				
C(1B)	0.451 (1)	0.071 (1)	0.761 (1)	0.056 (8)
C(2B)	0.481 (1)	0.207 (1)	0.774 (1)	0.048 (7)
C(3B)	0.3686 (9)	0.2639 (9)	0.7099 (9)	0.037 (5)
C(4B)	0.338 (1)	0.208 (1)	0.581 (1)	0.040 (6)
C(5B)	0.224 (1)	0.267 (1)	0.5242 (9)	0.043 (6)
C(6B)	0.115 (1)	0.240 (1)	0.5678 (9)	0.037 (5)
C(7B)	0.1338 (9)	0.2855 (9)	0.7015 (9)	0.033 (5)
C(8B)	0.2602 (9)	0.2568 (9)	0.7719 (9)	0.035 (5)
C(9B)	0.244 (1)	0.136 (1)	0.804 (1)	0.044 (6)
C(10B)	0.278 (1)	0.365 (1)	0.8841 (9)	0.041 (6)
C(11B)	0.237 (1)	0.472 (1)	0.832 (1)	0.044 (6)
C(12B)	0.185 (1)	0.582 (1)	0.908 (1)	0.062 (8)
C(13B)	0.023 (1)	0.461 (1)	0.780 (1)	0.049 (7)
C(14B)	-0.026 (2)	0.083 (2)	0.484 (2)	0.11 (1)
C(15B)	-0.090 (1)	0.198 (2)	0.487 (2)	0.08 (1)
C(16B)	0.448 (1)	0.218 (1)	0.530 (1)	0.056 (8)
C(17B)	0.588 (2)	0.358 (2)	0.493 (2)	0.09 (1)
C(18B)	0.321 (2)	-0.062 (1)	0.826 (1)	0.07 (1)
C(19B)	0.313 (1)	-0.170 (1)	0.718 (1)	0.061 (8)
C(20B)	0.244 (2)	-0.169 (1)	0.607 (1)	0.08 (1)
C(21B)	0.248 (3)	-0.272 (2)	0.517 (2)	0.13 (2)
C(22B)	0.304 (2)	-0.371 (2)	0.523 (2)	0.11 (2)
C(23B)	0.375 (2)	-0.374 (1)	0.632 (2)	0.10 (1)
C(24B)	0.381 (2)	-0.271 (1)	0.731 (1)	0.07 (1)
N(1B)	0.335 (1)	0.0571 (9)	0.7992 (9)	0.050 (6)
N(2B)	0.1258 (8)	0.4205 (8)	0.7404 (8)	0.038 (5)
O(1B)	0.1497 (8)	0.1156 (8)	0.8352 (8)	0.055 (5)
O(2B)	0.0518 (8)	0.5616 (8)	0.8726 (8)	0.059 (5)
O(3B)	-0.0839 (8)	0.4224 (9)	0.7438 (8)	0.063 (6)
O(4B)	0.0935 (8)	0.1100 (7)	0.5430 (7)	0.052 (5)
O(5B)	0.0085 (7)	0.2856 (8)	0.5108 (7)	0.054 (5)
O(6B)	0.506 (1)	0.134 (1)	0.492 (1)	0.10 (1)
O(7B)	0.4803 (9)	0.3356 (9)	0.5377 (9)	0.069 (7)
Molecule S				
C(1S)	0.803 (2)	0.698 (2)	0.555 (2)	0.110 (7)*
C(2S)	0.886 (2)	0.794 (2)	0.611 (2)	0.121 (8)*
C(3S)	0.843 (3)	0.888 (3)	0.645 (3)	0.16 (1)*
C(4S)	0.724 (2)	0.913 (2)	0.639 (2)	0.131 (8)*
C(5S)	0.654 (2)	0.825 (2)	0.601 (2)	0.115 (7)*
C(6S)	0.701 (2)	0.718 (2)	0.556 (2)	0.117 (7)*
C(7S)	0.853 (3)	0.596 (3)	0.524 (3)	0.18 (1)*

* Isotropic thermal parameters.

Table 2. *Interatomic distances (Å) and bond angles (°)*

	Molecule A	Molecule B
C(1)—C(2)	1.51 (1)	1.54 (2)
C(1)—N(1)	1.48 (1)	1.48 (2)
C(2)—C(3)	1.49 (2)	1.54 (2)
C(3)—C(4)	1.54 (1)	1.51 (1)
C(3)—C(8)	1.55 (1)	1.56 (2)
C(4)—C(5)	1.53 (2)	1.53 (2)
C(4)—C(16)	1.54 (2)	1.49 (2)
C(5)—C(6)	1.50 (2)	1.48 (2)
C(6)—C(7)	1.55 (1)	1.55 (1)
C(6)—O(4)	1.45 (1)	1.43 (1)
C(6)—O(5)	1.40 (2)	1.41 (1)
C(7)—C(8)	1.56 (2)	1.56 (1)
C(7)—N(2)	1.51 (1)	1.49 (1)
C(8)—C(9)	1.54 (2)	1.54 (2)
C(8)—C(10)	1.55 (1)	1.57 (1)
C(9)—N(1)	1.35 (2)	1.34 (2)
C(9)—O(1)	1.23 (1)	1.22 (2)
C(10)—C(11)	1.53 (2)	1.53 (2)
C(11)—C(12)	1.53 (2)	1.54 (2)
C(11)—N(2)	1.45 (2)	1.49 (1)
C(12)—O(2)	1.45 (2)	1.46 (2)
C(13)—N(2)	1.36 (1)	1.36 (2)
C(13)—O(2)	1.35 (2)	1.37 (1)
C(13)—O(3)	1.22 (2)	1.22 (1)
C(14)—C(15)	1.48 (3)	1.47 (3)
C(14)—O(4)	1.43 (2)	1.36 (2)
C(15)—O(5)	1.36 (3)	1.42 (2)
C(16)—O(6)	1.20 (2)	1.19 (2)
C(16)—O(7)	1.31 (2)	1.36 (2)
C(17)—O(7)	1.45 (2)	1.45 (3)
C(18)—C(19)	1.51 (2)	1.54 (2)
C(18)—N(1)	1.48 (2)	1.48 (2)
C(19)—C(20)	1.37 (2)	1.41 (2)
C(19)—C(24)	1.39 (2)	1.39 (2)
C(20)—C(21)	1.39 (2)	1.39 (3)
C(21)—C(22)	1.47 (3)	1.29 (3)
C(22)—C(23)	1.40 (3)	1.40 (3)
C(23)—C(24)	1.31 (2)	1.44 (2)

Molecule S	
C(1S)—C(2S)	1.37 (3)
C(1S)—C(6S)	1.16 (3)
C(1S)—C(7S)	1.28 (4)
C(2S)—C(3S)	1.18 (4)
C(3S)—C(4S)	1.33 (4)
C(4S)—C(5S)	1.20 (3)
C(5S)—C(6S)	1.34 (3)

	Molecule A	Molecule B
C(2)—C(1)—N(1)	110.7 (6)	110 (1)
C(1)—C(2)—C(3)	112.9 (9)	110.0 (9)
C(2)—C(3)—C(4)	112.0 (8)	112.8 (9)
C(2)—C(3)—C(8)	109.9 (9)	107 (1)
C(4)—C(3)—C(8)	112.7 (9)	114.5 (8)
C(3)—C(4)—C(5)	110.9 (8)	109.5 (9)
C(3)—C(4)—C(16)	108.5 (9)	110.1 (9)
C(5)—C(4)—C(16)	109 (1)	113 (1)
C(4)—C(5)—C(6)	109 (1)	110 (1)
C(5)—C(6)—C(7)	113.6 (9)	112.7 (8)
C(5)—C(6)—O(4)	108.4 (8)	109.9 (9)
C(5)—C(6)—O(5)	110 (1)	111 (1)
C(7)—C(6)—O(4)	105.5 (9)	105.2 (9)
C(7)—C(6)—O(5)	112.2 (8)	111.5 (8)
O(4)—C(6)—O(5)	106.6 (9)	106.6 (8)
C(6)—C(7)—C(8)	115.7 (8)	115.3 (9)
C(6)—C(7)—N(2)	112 (1)	112.4 (9)
C(8)—C(7)—N(2)	103.4 (8)	105.0 (7)
C(3)—C(8)—C(7)	115.0 (9)	112.6 (9)
C(3)—C(8)—C(9)	114.3 (9)	115.6 (9)
C(3)—C(8)—C(10)	108.5 (8)	108.8 (8)
C(7)—C(8)—C(9)	106.0 (9)	108.1 (8)
C(7)—C(8)—C(10)	102.0 (8)	101.5 (8)
C(9)—C(8)—C(10)	110.3 (9)	109.3 (9)
C(8)—C(9)—N(1)	118 (1)	119 (1)
C(8)—C(9)—O(1)	119 (1)	119 (1)
N(1)—C(9)—O(1)	122 (1)	122 (1)
C(8)—C(10)—C(11)	100.5 (9)	101.1 (8)
C(10)—C(11)—C(12)	117 (1)	118 (1)
C(10)—C(11)—N(2)	102.9 (9)	103.1 (8)
C(12)—C(11)—N(2)	102 (1)	102.2 (9)
C(11)—C(12)—O(2)	104 (1)	104.4 (9)
N(2)—C(13)—O(2)	111 (1)	110.6 (9)
N(2)—C(13)—O(3)	128 (1)	130 (1)
O(2)—C(13)—O(3)	121 (1)	120 (1)
C(15)—C(14)—O(4)	108 (2)	109 (1)

Table 2 (cont.)

	Molecule A	Molecule B
C(14)—C(15)—O(5)	105 (2)	102 (1)
C(4)—C(16)—O(6)	123 (1)	125 (1)
C(4)—C(16)—O(7)	111 (1)	112 (1)
O(6)—C(16)—O(7)	125 (1)	122 (1)
C(19)—C(18)—N(1)	113 (1)	112 (1)
C(18)—C(19)—C(20)	123 (1)	124 (1)
C(18)—C(19)—C(24)	119 (1)	118 (1)
C(20)—C(19)—C(24)	118 (1)	118 (1)
C(19)—C(20)—C(21)	122 (2)	118 (2)
C(20)—C(21)—C(22)	116 (2)	128 (2)
C(21)—C(22)—C(23)	120 (1)	116 (2)
C(22)—C(23)—C(24)	120 (2)	121 (2)
C(19)—C(24)—C(23)	124 (2)	119 (2)
C(1)—N(1)—C(9)	126.6 (9)	127 (1)
C(1)—N(1)—C(18)	114 (1)	113 (1)
C(9)—N(1)—C(18)	119 (1)	120 (1)
C(7)—N(2)—C(11)	110 (1)	109.5 (8)
C(7)—N(2)—C(13)	115.7 (9)	116.2 (9)
C(11)—N(2)—C(13)	108.8 (9)	108.8 (9)
C(12)—O(2)—C(13)	109.0 (9)	110 (1)
C(6)—O(4)—C(14)	106 (1)	108 (1)
C(6)—O(5)—C(15)	111 (2)	108 (1)
C(16)—O(7)—C(17)	113 (1)	118 (1)

Molecule S	
C(2S)—C(1S)—C(6S)	115 (2)
C(2S)—C(1S)—C(7S)	114 (3)
C(6S)—C(1S)—C(7S)	130 (3)
C(1S)—C(2S)—C(3S)	116 (3)
C(2S)—C(3S)—C(4S)	128 (3)
C(3S)—C(4S)—C(5S)	115 (3)
C(4S)—C(5S)—C(6S)	117 (2)
C(1S)—C(6S)—C(5S)	128 (2)

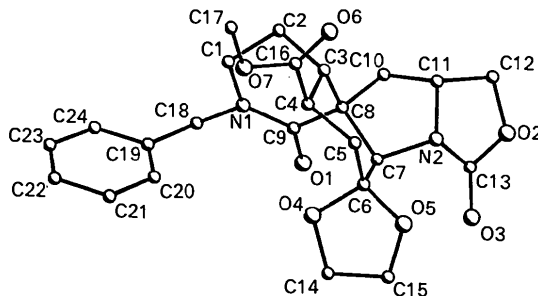


Fig. 1. The molecular structure of the title compound (molecule A), showing the numbering scheme.

configuration, known from the synthesis in which only optically pure reagents were used, could not be verified by least-squares refinement. All calculations were performed with the *XTAL* system of crystallographic programs (Hall & Stewart, 1989) unless stated otherwise.

In the tables, the two molecules are referred to as *A* and *B*, and the solvent molecule as *S*. Final positional parameters for the non-H atoms are listed in Table 1, bond lengths and bond angles are in Table 2.* A *PLUTO* (Motherwell & Clegg, 1978) drawing of molecule *A* is given in Fig. 1 and a packing diagram is in Fig. 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53908 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

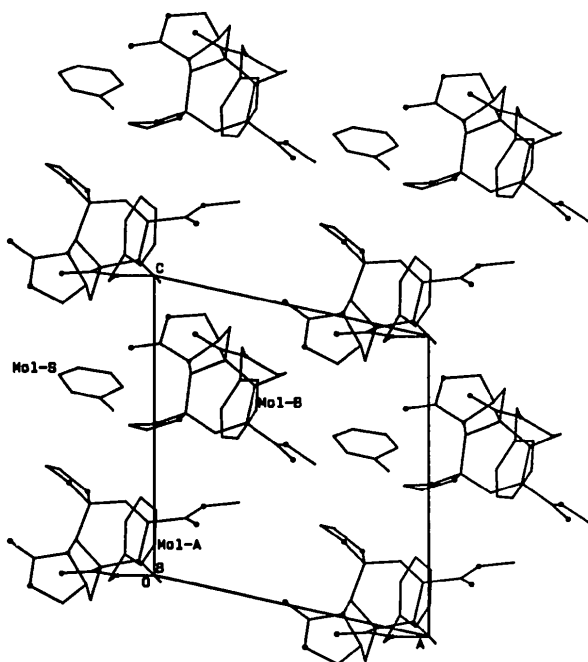


Fig. 2. Packing diagram looking along the *b* axis.

Related literature. The compound (+)-manzamine-*A*, which can be isolated from the sponge *Haliclona*, is an anti-tumour alkaloid. The structure here presented is that of an intermediate in the synthesis of (+)-manzamine-*A* as described by Brands (1990).

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Structure de l'Acide (*tert*-Butylamino-4 oxo-5 triazol-1,2,4 yle-1)acétique

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Abstract. (4-*tert*-Butylamino-5-oxo-1,2,4-triazol-1-yl)acetic acid, $C_8H_{14}N_4O_3$, $M_r = 214.2$, monoclinic, $P2_1/c$, $a = 8.275(2)$, $b = 10.624(3)$, $c = 12.086(3)$ Å, $\beta = 97.89(2)^\circ$, $V = 1052.4(1)$ Å³, $Z = 4$, $D_x = 1.352$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.792$ mm⁻¹, $F(000) = 456$, $T = 294(1)$ K, $R = 0.058$ for 1272 independent reflections. The triazolone ring deviates slightly from planarity [maximum deviation $-0.016(3)$ Å] with a twist conformation defined by the Cremer and Pople puckering parameters $Q = 0.026(3)$ Å, $\varphi = 14(6)^\circ$. C(2) and N(4) lie out of this mean plane [by $0.396(3)$ and $0.067(3)$ Å, respectively]. All O atoms participate in hydrogen bonding: N—H \cdots O(=C) with N \cdots O $2.970(4)$, N—H $0.99(4)$,

O \cdots H $1.99(4)$ Å, N—H \cdots O $167(2)^\circ$, and (C—)O—H \cdots O (triazolone) with O \cdots O $2.639(3)$, O—H $1.05(4)$, O \cdots H $1.59(3)$ Å, O—H \cdots O $174(2)^\circ$.

Partie expérimentale. Cristal du composé (1) incolore $0,2 \times 0,2 \times 0,5$ mm obtenu par cristallisation dans l'eau. Diffractomètre Siemens.

