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The crystal structure of pyrrolidin-1-yl pivalate, $C_9H_{13}NO_4$

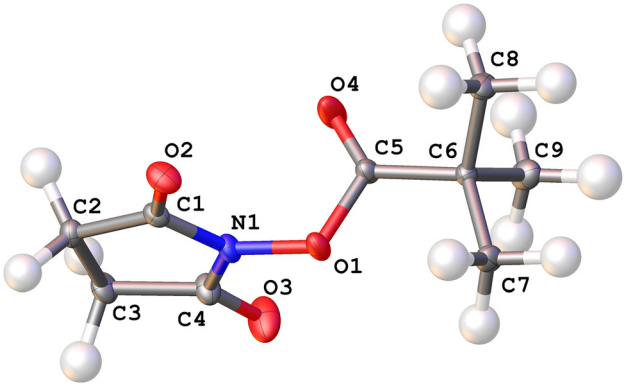

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.40 × 0.11 × 0.02 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.88 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	76.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,980, 2,117, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs}°>°2σ(I_{obs}), 2,101
$N(param)_{refined}$:	180
Programs:	CrysAlis^{PRO},[1] Olex2,[2,5] SHELX[3,4]

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Abstract

$C_9H_{13}NO_4$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.8406(1) Å, b = 7.0840(1) Å, c = 24.3697(3) Å, V = 1,008.29(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0148, $wR_{ref}(F^2)$ = 0.0381, T = 100.0(1) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

The title compound was obtained by an intermolecular arrangement reaction of pivaloyl chloride and 2,5-dioxopyrrolidin-1-yl glycine in acetonitrile (1:1 molar ratio) in the presence of diisopropylethylamine (Hünig's base, DIPEA) as a base. The reaction occurs after 24 h under continuous stirring at room temperature. After completed reaction (TLC control: dichloromethane/methanol, 9:1, R_f = 0.15) the mixture was concentrated under reduced pressure until a pale oil left. The crude material was dissolved in dichloromethane, washed three times with saturated sodium hydrogencarbonate and brine, and dried over sodium sulfate. The organic phase was filtered and evaporated to yield 107.7 mg (70 %) of a brownish oil, which was left to slowly to crystallize at ambient conditions. White crystals suitable for X-ray structure analysis were obtained after recrystallization from dichloromethane and crystallization over a period of several days at 295–297 K. The crystals were analyzed directly from the mother liquor at 100(1) K. The melting point of pyrrolidin-1-yl pivalate is 338.9–341.4 K.

2 Experimental details

A single crystal of the title compound was examined on a Rigaku Supernova diffractometer[1] using CuKα (λ = 1.54184 Å) radiation. The crystal was kept at 100.0(1) K during data collection. Using Olex2,[2] the structure was solved with the ShelXT[3,4] structure solution program using Intrinsic Phasing method and refined with the olex2.refine[5,6] refinement package using Gauss–Newton minimisation. Refinement using NoSpherA2, an implementation of NOn-SPHERical Atom-form-factors in Olex2.[7] NoSpherA2 implementation of

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.46920 (7)	0.38586 (6)	0.380074 (18)	0.01749 (10)
O2	0.31426 (7)	0.49476 (6)	0.275772 (19)	0.02198 (10)
O3	0.40633 (10)	0.00003 (7)	0.39319 (2)	0.03435 (13)
O4	0.81127 (8)	0.30810 (8)	0.34404 (2)	0.02958 (12)
N1	0.37577 (9)	0.26785 (6)	0.34127 (2)	0.01538 (11)
C1	0.29848 (9)	0.33288 (8)	0.29114 (2)	0.01413 (11)
C2	0.19979 (10)	0.16399 (8)	0.26216 (2)	0.01607 (12)
H2A	0.3006 (16)	0.1392 (14)	0.2254 (4)	0.038 (2)*
H2B	0.0235 (16)	0.1940 (13)	0.2501 (4)	0.033 (2)*
C3	0.21366 (10)	0.00071 (8)	0.30327 (2)	0.01858 (12)
H3A	0.3052 (17)	−0.1217 (13)	0.2867 (4)	0.035 (2)*
H3B	0.0379 (18)	−0.0393 (15)	0.3184 (4)	0.039 (2)*
C4	0.34294 (10)	0.07723 (8)	0.35195 (2)	0.01844 (12)
C5	0.70599 (9)	0.39101 (8)	0.37838 (2)	0.01450 (11)
C6	0.80311 (9)	0.51332 (7)	0.42386 (2)	0.01283 (11)
C7	0.61930 (10)	0.60241 (8)	0.46030 (2)	0.01792 (12)
H7A	0.5163 (17)	0.4940 (15)	0.4821 (4)	0.038 (2)*
H7B	0.7019 (17)	0.6833 (14)	0.4922 (4)	0.038 (2)*
H7C	0.5074 (18)	0.6978 (15)	0.4370 (4)	0.040 (3)*
C8	0.94332 (11)	0.66861 (9)	0.39574 (2)	0.01998 (13)
H8A	1.0700 (17)	0.6081 (14)	0.3680 (4)	0.043 (2)*
H8B	0.8315 (19)	0.7604 (15)	0.3716 (4)	0.044 (3)*
H8C	1.0291 (18)	0.7551 (14)	0.4275 (4)	0.038 (2)*
C9	0.95807 (11)	0.38520 (9)	0.45845 (3)	0.02120 (13)
H9A	0.859 (2)	0.2683 (14)	0.4770 (4)	0.042 (3)*
H9B	1.093 (2)	0.3224 (16)	0.4342 (4)	0.049 (3)*
H9C	1.0332 (19)	0.4688 (16)	0.4923 (4)	0.047 (3)*

HAR makes use of tailor-made aspherical atomic form factors calculated on-the-fly from a Hirshfeld-partitioned electron density (ED) – not from spherical-atom form factors. The ED is calculated from a Gaussian basis set single determinant SCF wavefunction – either Hartree–Fock or DFT using selected functionals – for a fragment of the crystal. This fragment can be embedded in an electrostatic crystal field by employing cluster charges or modelled using implicit solvatation models, depending on the software used. Displacement ellipsoids are drawn at the 50 % probability level. Hydrogen atoms were taken into account using a riding model. There is one molecule in the asymmetric unit. All bond lengths and angles are in the expected ranges. [8–11]

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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