Chapter 15
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Structural Study of Methyl-2-benzamido-2-{[-2-methoxy-2-oxo-1-phenylethyl]amino}acetate

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ABSTRACT

In the title compound, $C_{19}H_{20}N_2O_5$, the dihedral angle between the phenyl rings is 58.85 (8)°, while that between the planes of the methyl acetate groups is 88.30 (8)°. The molecular conformation is also influenced by the presence of an intramolecular N-H...O hydrogen bond. In the crystal, N-H...O hydrogen bonds link the molecules, forming chains propagating along the a-axis direction.

Keywords: Structural study; carboxylic amino esters; N-alkylation.

1. INTRODUCTION

The synthesis of new α-AAs (α-aminoacetates) and their esters is of international interest because of their extensive applications in enzymology, medicine, pharmacology and industry [1-3], Our strategy used nucleophilic substitution of the *N*-protected methyl α-azido glycinate with 2-amino-2-phenylacetate in methylene chloride, in the presence of triethylamine as a base [4] to produce the title compound in good yield.

In the title molecule (Figs. 1 and 2), the dihedral angles between the phenyl rings and those between the planes of the methyl acetate groups are 58.85 (8) and 88.30 (8)° respectively.

The twisted conformation of the molecule is also influenced by the presence of an intramolecular N-H...O hydrogen bond (Table 1). In the crystal, N-H...O and C-H...O hydrogen bonds (Table 1, Fig. 3) link the molecules into chains propagating along the a-axis direction.

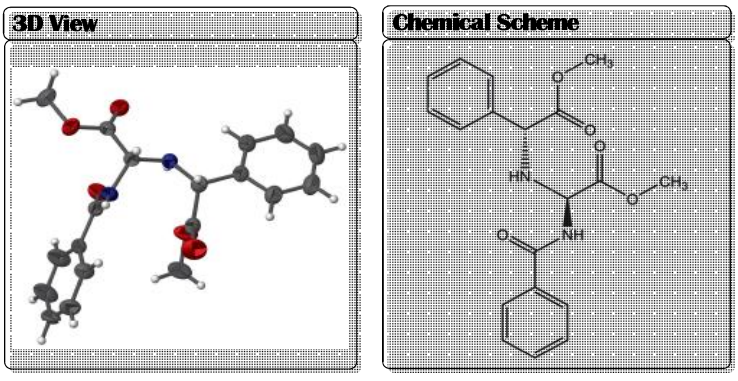

Fig. 1. 3D view and chemical scheme of the title compound

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2. SYNTHESIS AND CRYSTALLIZATION

To a stirred solution of methyl 2-amino-2-phenylacetate (2 mmol) and triethylamine (4 mmol) in 10 mL of dry methylene chloride, *N*-benzoylated methyl α-azidoglycinate (2.6 mmol) was added. The mixture was stirred at 0°C for 1 hour then at room temperature for 16 hours. The resulting solution was washed with citric acid (15%), then with a saturated solution of sodium bicarbonate ($NaHCO_3$). Solvents were removed and colorless single crystals of the title compound were obtained by recrystallization from ether (yield = 86 %; m.p. = 126-128°C).

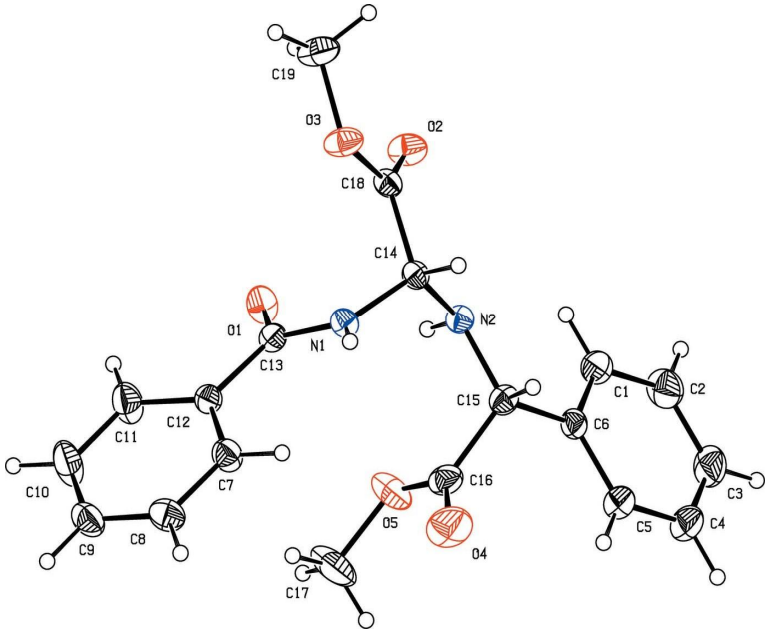

Fig. 2. ORTEP view of the title compound

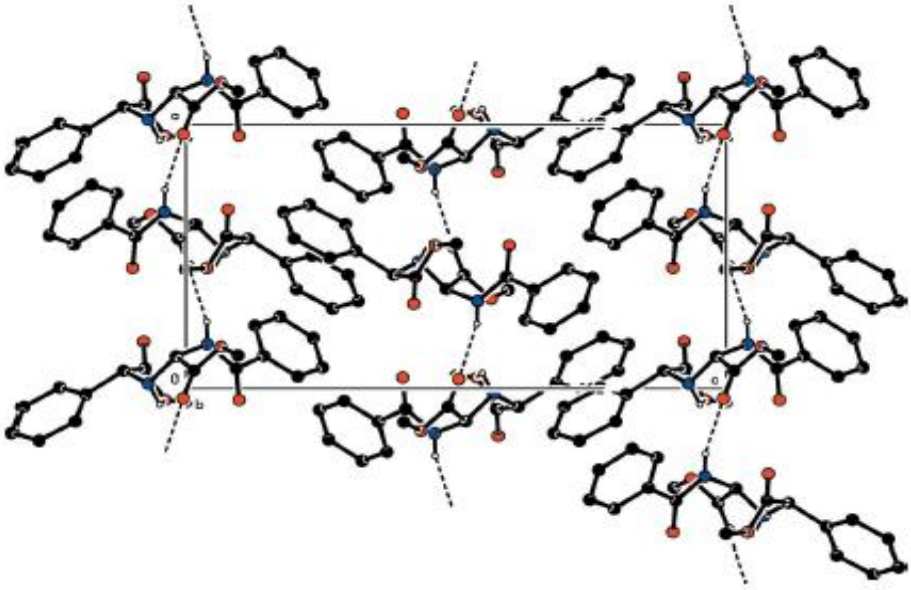

Fig. 3. A view of the crystal packing of the title compound along the b axis

δ_H(ppm, $CDCl_3$, 300.13 MHz): 3.3 (1H, NH-CH-Ph, e); 3.51 (3H, OCH_3, s); 3.78 (3H, OCH_3, s); 4.65 (1H, NH-CH-Ph, e); 5.52 (1H, N-CH-N, d, *J* = 8.4 Hz); 6.75 (1H, NHBz, d, *J* = 8.4 Hz); 7.28-7.82 ($10H_{arom}$, m). δ_C(ppm, $CDCl_3$, 75.47 MHz): 52.44 (1C, OCH_3); 52.88 (1C, OCH_3); 61.97 (1C, NH-CH-Ph); 63.62 (1C, N-CH-N); 127.13-137.48 (10C, C_{arom}); 167.14; 170.14 and 173.63 (3C, 3 x C=O). MS-EI: $[M]^+$ = 356.49. Elemental analysis: Calcd.: C, 64.04; H, 5.66; N, 7.86; Found: C 63.84, H 5.67, N 7.89.

Table 1. Hydrogen-bond geometry (Å, °)

D-H··· A	D-H	H··· A	D··· A	D—H··· A
N1H1N··· $O2^{i}$	0.85 (1)	2.00 (2)	2.9147 (15)	162 (1)
N2H2N··· O5	0.85 (1)	2.52 (2)	2.8334 (15)	103 (1)

Symmetry codes: (i) x+1/2, –y+1/2, –z+2

Table 2. Experimental details

Crystal Data	
Chemical formula	$C_{19}H_{20}N_2O_5$
Mr	356.37
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	173
a, b, c (Å)	9.3432 (6), 10.4314 (8),18.0901 (14)
V ($Å^3$)	1763.1 (2)
Z	4
Radiation type	Mo Kα
μ (mm^{-1})	0.10
Crystal size (mm)	0.35 ~ 0.16 ~ 0.14
Data Collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Bruker, 2005)
No. of measured, independent and observed [I > 2σ(I)] reflections	0.967, 0.986
R_{int}	28362, 3455, 3246
(sin &/h)max ($Å^{-1}$)	0.617
Refinement	
R[F^2 > 2 σ (F^2)], wR(F^2), S	0.027, 0.065, 1.04
No. of reflections	3455
No. of parameters	246
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ max, Δρ min (e $Å^{-3}$)	0.14, -0.13
Absolute structure	Flack (1983), 1822 Friedel pairs
Absolute structure parameter	-0.3 (8)

3. SPECIAL DETAILS

3.1 Geometry

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes [5-9].

3.2 Refinement

Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors (gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Table 3. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C14	0.12279 (14)	0.23661 (11)	0.98202 (7)	0.0305 (3)
H14	0.2102	0.2580	0.9526	0.037*
N1	0.16831 (11)	0.14782 (10)	1.03988 (6)	0.0315 (2)
O3	0.15911 (11)	0.40644 (9)	1.06499 (6)	0.0464 (3)
O1	−0.04242 (10)	0.16104 (10)	1.09856 (6)	0.0456 (3)
O2	−0.03638 (11)	0.41900 (9)	0.99493 (6)	0.0475 (3)
C15	0.06822 (15)	0.08188 (12)	0.88463 (7)	0.0359 (3)
H15	0.1676	0.1036	0.8684	0.043*
N2	0.01902 (12)	0.18778 (10)	0.93039 (6)	0.0321 (2)
C12	0.12463 (14)	0.00585 (12)	1.14380 (7)	0.0330 (3)
C18	0.06847 (13)	0.36261 (11)	1.01468 (7)	0.0323 (3)
C13	0.07640 (13)	0.11150 (12)	1.09329 (7)	0.0322 (3)
C1	−0.12382 (16)	0.15859 (14)	0.79537 (8)	0.0452 (3)
H1	−0.1444	0.2276	0.8279	0.054*
C6	−0.02209 (14)	0.06888 (13)	0.81452 (7)	0.0353 (3)
C9	0.19247 (18)	−0.19864 (15)	1.23588 (9)	0.0493 (4)
H9	0.2146	−0.2693	1.2669	0.059*
O5	−0.04305 (14)	−0.07267 (10)	0.96120 (7)	0.0620 (3)
C8	0.27280 (17)	−0.17514 (15)	1.17398 (9)	0.0479 (4)
H8	0.3519	−0.2288	1.1627	0.057*
C19	0.1260 (2)	0.53040 (15)	1.09662 (10)	0.0572 (4)
H19A	0.1460	0.5977	1.0602	0.086*
H19B	0.1851	0.5443	1.1407	0.086*
H19C	0.0246	0.5332	1.1104	0.086*
C7	0.23931 (16)	−0.07355 (13)	1.12783 (8)	0.0423 (3)
H7	0.2954	−0.0582	1.0849	0.051*
C4	−0.0684 (2)	−0.04241 (16)	0.70022 (9)	0.0571 (4)
H4	−0.0492	−0.1120	0.6678	0.069*
C5	0.00429 (18)	−0.03273 (15)	0.76647 (8)	0.0474 (4)
H5	0.0730	−0.0960	0.7794	0.057*
C11	0.04614 (19)	−0.01734 (17)	1.20718 (9)	0.0560 (4)
H11	−0.0316	0.0373	1.2196	0.067*
C16	0.07709 (18)	−0.04722 (14)	0.92455 (8)	0.0471 (4)
O4	0.17846 (16)	−0.11697 (13)	0.92196 (8)	0.0816 (4)
C3	−0.1684 (2)	0.04823 (18)	0.68102 (9)	0.0619 (5)

Table 4. Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C14	0.0284 (6)	0.0311 (6)	0.0321 (6)	−0.0018 (5)	−0.0005 (5)	0.0026 (5)
N1	0.0270 (5)	0.0325 (5)	0.0352 (5)	0.0043 (4)	−0.0002 (5)	0.0039 (4)
O3	0.0447 (5)	0.0364 (5)	0.0581 (6)	0.0044 (4)	−0.0181 (5)	−0.0123 (5)
O1	0.0363 (5)	0.0549 (6)	0.0454 (5)	0.0140 (5)	0.0072 (4)	0.0097 (5)
O2	0.0412 (6)	0.0406 (5)	0.0607 (6)	0.0089 (4)	−0.0159 (5)	−0.0052 (5)
C15	0.0341 (6)	0.0374 (7)	0.0362 (7)	−0.0007 (6)	0.0017 (5)	−0.0037 (5)
N2	0.0307 (5)	0.0324 (5)	0.0332 (5)	−0.0013 (4)	−0.0016 (5)	−0.0005 (4)
C12	0.0340 (6)	0.0337 (6)	0.0314 (6)	−0.0010 (5)	−0.0040 (5)	0.0003 (5)
C18	0.0311 (6)	0.0305 (6)	0.0351 (6)	−0.0020 (5)	−0.0040 (5)	0.0042 (5)
C13	0.0322 (6)	0.0333 (6)	0.0310 (6)	0.0022 (5)	−0.0010 (5)	−0.0018 (5)
C1	0.0494 (8)	0.0441 (8)	0.0421 (7)	−0.0024 (7)	−0.0045 (7)	−0.0009 (6)
C6	0.0383 (7)	0.0365 (7)	0.0310 (6)	−0.0076 (5)	0.0033 (5)	0.0012 (5)
C9	0.0577 (9)	0.0423 (8)	0.0479 (8)	−0.0024 (7)	−0.0112 (7)	0.0134 (7)
O5	0.0676 (8)	0.0449 (6)	0.0736 (8)	−0.0110 (6)	−0.0148 (6)	0.0246 (6)
C8	0.0477 (8)	0.0397 (7)	0.0562 (9)	0.0076 (7)	−0.0043 (7)	0.0055 (7)
C19	0.0653 (10)	0.0380 (8)	0.0681 (10)	0.0046 (7)	−0.0147 (9)	−0.0170 (7)
C7	0.0429 (8)	0.0412 (7)	0.0427 (7)	0.0053 (6)	0.0041 (6)	0.0062 (6)
C4	0.0746 (11)	0.0543 (9)	0.0426 (8)	−0.0127 (9)	−0.0008 (8)	−0.0113 (7)
C5	0.0570 (9)	0.0446 (8)	0.0407 (8)	−0.0036 (7)	0.0012 (7)	−0.0050 (7)
C11	0.0566 (10)	0.0626 (10)	0.0487 (8)	0.0169 (8)	0.0150 (8)	0.0159 (8)
C16	0.0599 (9)	0.0377 (7)	0.0436 (8)	0.0078 (7)	−0.0176 (7)	−0.0074 (6)
O4	0.0964 (10)	0.0652 (8)	0.0831 (9)	0.0450 (8)	−0.0142 (8)	−0.0072 (7)
C3	0.0760 (12)	0.0700 (11)	0.0397 (8)	−0.0191 (10)	−0.0167 (8)	0.0003 (8)
C10	0.0731 (12)	0.0746 (12)	0.0494 (9)	0.0129 (10)	0.0160 (9)	0.0265 (9)
C2	0.0636 (10)	0.0595 (10)	0.0546 (10)	−0.0007 (9)	−0.0191 (8)	0.0069 (8)
C17	0.139 (2)	0.0537 (11)	0.0873 (14)	−0.0394 (13)	−0.0489 (15)	0.0349 (10)
C14	0.0284 (6)	0.0311 (6)	0.0321 (6)	−0.0018 (5)	−0.0005 (5)	0.0026 (5)
N1	0.0270 (5)	0.0325 (5)	0.0352 (5)	0.0043 (4)	−0.0002 (5)	0.0039 (4)
O3	0.0447 (5)	0.0364 (5)	0.0581 (6)	0.0044 (4)	−0.0181 (5)	−0.0123 (5)
O1	0.0363 (5)	0.0549 (6)	0.0454 (5)	0.0140 (5)	0.0072 (4)	0.0097 (5)

Table 5. Geometric parameters (Å, °)

C14—N2	1.4395 (16)	**C9—H9**	0.9500
C14—N1	1.4610 (16)	**O5—C16**	1.331 (2)
C14—C18	1.5277 (17)	**O5—C17**	1.4527 (19)
C14—H14	1.0000	**C8—C7**	1.385 (2)
N1—C13	1.3469 (17)	**C8—H8**	0.9500
N1—H1N	0.855 (13)	**C19—H19A**	0.9800
O3—C18	1.3246 (15)	**C19—H19B**	0.9800
O3—C19	1.4474 (17)	**C19—H19C**	0.9800
O1—C13	1.2282 (15)	**C7—H7**	0.9500
O2—C18	1.1973 (15)	**C4—C3**	1.374 (3)
C15—N2	1.4549 (16)	**C4—C5**	1.381 (2)
C15—C6	1.5294 (18)	**C4—H4**	0.9500
C15—C16	1.530 (2)	**C5—H5**	0.9500
C15—H15	1.0000	**C11—C10**	1.385 (2)
N2—H2N	0.850 (12)	**C11—H11**	0.9500
C12—C11	1.382 (2)	**C16—O4**	1.1952 (19)
C12—C7	1.3848 (19)	**C3—C2**	1.378 (3)
C12—C13	1.5009 (17)	**C3—H3**	0.9500
C1—C6	1.378 (2)	**C10—H10**	0.9500
C1—C2	1.392 (2)	**C2—H2**	0.9500
C1—H1	0.9500	**C17—H17A**	0.9800

C6—C5	1.393 (2)	**C17—H17B**	0.9800
C9—C8	1.370 (2)	**C17—H17C**	0.9800
C9—C10	1.369 (2)		
N2—C14—N1	115.89 (10)	**C9—C8—H8**	119.8
N2—C14—C18	109.37 (10)	**C7—C8—H8**	119.8
N1—C14—C18	111.41 (10)	**O3—C19—H19A**	109.5
N2—C14—H14	106.5	**O3—C19—H19B**	109.5
N1—C14—H14	106.5	**H19A—C19—H19B**	109.5
C18—C14—H14	106.5	**O3—C19—H19C**	109.5
C13—N1—C14	120.44 (11)	**H19A—C19—H19C**	109.5
C13—N1—H1N	121.3 (10)	**H19B—C19—H19C**	109.5
C14—N1—H1N	118.2 (10)	**C12—C7—C8**	120.44 (14)
C18—O3—C19	116.33 (11)	**C12—C7—H7**	119.8
N2—C15—C6	111.39 (11)	**C8—C7—H7**	119.8
N2—C15—C16	114.64 (11)	**C3—C4—C5**	120.22 (16)
C6—C15—C16	110.07 (11)	**C3—C4—H4**	119.9
N2—C15—H15	106.8	**C5—C4—H4**	119.9
C6—C15—H15	106.8	**C4—C5—C6**	120.67 (15)
C16—C15—H15	106.8	**C4—C5—H5**	119.7
C14—N2—C15	115.15 (10)	**C6—C5—H5**	119.7
C14—N2—H2N	111.8 (9)	**C12—C11—C10**	120.44 (15)
C15—N2—H2N	110.0 (10)	**C12—C11—H11**	119.8
C11—C12—C7	118.61 (13)	**C10—C11—H11**	119.8
		O4—C16—O5	124.52 (15)
C7—C12—C13	122.99 (12)	**O4—C16—C15**	124.07 (16)
O2—C18—O3	123.96 (12)	**O5—C16—C15**	111.41 (12)
O2—C18—C14	125.39 12)	**C4—C3—C2**	119.61 (15)
O3—C18—C14	110.51 (10)	**C4—C3—H3**	120.2
O1—C13—N1	120.90 (12)	**C2—C3—H3**	120.2
O1—C13—C12	122.21 (12)	**C9—C10—C11**	120.49 (15)
N1—C13—C12	116.86 (11)	**C9—C10—H10**	119.8
C6—C1—C2	120.23 (15)	**C11—C10—H10**	119.8
C6—C1—H1	119.9	**C3—C2—C1**	120.43 (16)
C2—C1—H1	119.9	**C3—C2—H2**	119.8
C1—C6—C5	118.82 (13)	**C1—C2—H2**	119.8
C1—C6—C15	121.92 (12)	**O5—C17—H17A**	109.5
C5—C6—C15	119.17 (12)	**O5—C17—H17B**	109.5
C8—C9—C10	119.61 (14)	**H17A—C17—H17B**	109.5
C8—C9—H9	120.2	**O5—C17—H17C**	109.5
C10—C9—H9	120.2	**H17A—C17—H17C**	109.5
C16—O5—C17	116.64 (16)	**H17B—C17—H17C**	109.5
C9—C8—C7	120.38 (15)		
N2—C14—N1—C13	−68.46 (15)	**N2—C15—C6—C5**	−175.99 (12)
C18—C14—N1—C13	57.43 (15)	**C16—C15—C6—C5**	−47.69 (17)
N1—C14—N2—C15	−64.44 (15)	**C10—C9—C8—C7**	−1.2 (2)
C18—C14—N2—C15	168.63 (10)	**C11—C12—C7—C8**	1.0 (2)
C6—C15—N2—C14	−158.86 (11)	**C13—C12—C7—C8**	−175.46 (13)
C16—C15—N2—C14	75.32 (15)	**C9—C8—C7—C12**	0.3 (2)
C19—O3—C18—O2	−0.1 (2)	**C3—C4—C5—C6**	−0.3 (2)
C19—O3—C18—C14	175.61 (13)	**C1—C6—C5—C4**	1.1 (2)
N2—C14—C18—O2	−7.24 (17)	**C15—C6—C5—C4**	−175.50 (14)
N1—C14—C18—O2	−136.67 (13)	**C7—C12—C11—C10**	−1.4 (2)
N2—C14—C18—O3	177.07 (11)	**C13—C12—C11—C10**	175.25 (16)
N1—C14—C18—O3	47.65 (14)	**C17—O5—C16—O4**	0.2 (2)
C14—N1—C13—O1	−6.90 (19)	**C17—O5—C16—C15**	179.10 (13)
C14—N1—C13—C12	171.27 (10)	**N2—C15—C16—O4**	−131.74 (15)
C11—C12—C13—O1	−13.9 (2)	**C6—C15—C16—O4**	101.76 (17)

C7—C12—C13—O1	162.54 (13)	**N2—C15—C16—O5**	49.39 (16)
C11—C12—C13—N1	167.94 (13)	**C6—C15—C16—O5**	−77.11 (14)
C7—C12—C13—N1	−15.60 (19)	**C5—C4—C3—C2**	−0.4 (3)
C2—C1—C6—C5	−1.2 (2)	**C8—C9—C10—C11**	0.8 (3)
C2—C1—C6—C15	175.29 (14)	**C12—C11—C10—C9**	0.5 (3)
N2—C15—C6—C1	7.55 (18)	**C4—C3—C2—C1**	0.3 (3)
C16—C15—C6—C1	135.86 (14)	**C6—C1—C2—C3**	0.5 (2)

4. CONCLUSION

In summary, the synthesis of methyl (2R)-2-benzamido-2-{[(1R)-2-methoxy-2-oxo-1-phenylethyl]-amino}acetate was performed via *N*-alkylation reaction. The structural study and elemental data are in perfect agreement with the proposed structure of the obtained product.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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