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N-Methylation of adamantane-substituted oxalamide unit affects its conformational rigidity: A skew conformation of the oxalamide bridge

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Abstract

The synthesis of adamantane functionalized retropeptides, *N,N′*-bis(2-adamantyl-2-carboxylic acid methyl ester)oxalamide (**1**) and *N*-methyl-*N,N′*-bis(2-adamantyl-2-carboxylic acid methyl ester)oxalamide (**2**), as well as the influence of the *N*-methylation on the conformational preferences of oxalamide group are described. In the solid state the oxalamide group of **1** is planar while in the retropeptide **2** adopts a skew-conformation.

Keywords: Adamantane; *N*-Methylation; Oxalamides; Retropeptides; X-ray structure analysis

1. Introduction

The interest in the investigation of retropeptides with an oxalamide unit (—NH—CO—CO—NH—) has been increased over the last two decades due to their potential use as biomimetics and application in crystal engineering [1,2]. A number of compounds of this class have been designed and synthesized in order to investigate the delicate interplay of structure, non-covalent interactions [3], and gelating properties [4–6]. The oxalamide group serves as a synthon to assemble molecules through intermolecular N—H···O hydrogen bonds into one-dimensional α-network, in which the central oxalamide units adopts planar and *trans* configuration. This type of conformation appears to be independent of the type and size of the group attached at the ends of the oxalamide moiety. However, we wanted to examine the influence of bulky adamantane amino acid on the conformation of oxalamide unit. Besides, the introduction of adamantane moiety should change the overall lipophilic properties of synthesized retropeptide [7,8].

On the other hand, it is documented that physico-chemical properties and biological activity of peptide systems are changed by *N*-methylation of amino acids. The *N*-methylation affects pharmacological parameters such as membrane permeability, conformational rigidity, and proteolytic stability [9,10]. Peptides with *N*-methyl amino acids exhibit antibiotic, anticancer, and antiviral activity [11,12]. *N*-Methylation of peptide bond provides a steric modification that can change the conformation of the peptide and aromatic amides, i.e. *cis* preference is general in *N*-methyl

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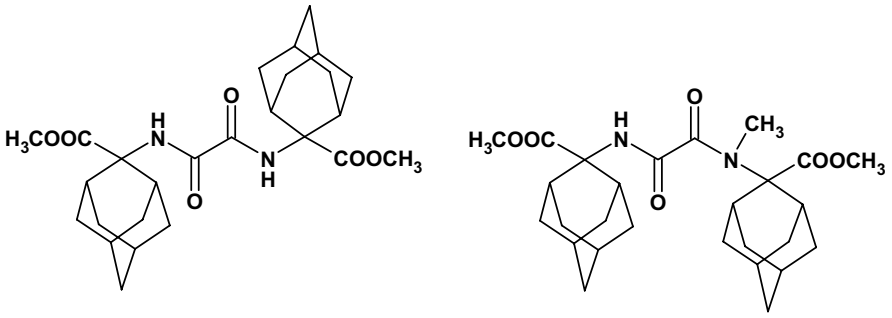


Chart 1.

aromatic amides [13]. Also, the number of donor and acceptor sites is reduced affecting hydrogen bonding.

In this paper, the synthesis and structural characterization of adamantane functionalized oxalamide retropeptides, *N,N'*-bis(2-adamantyl-2-carboxylic acid methyl ester)oxalamide (**1**) and *N*-methyl-*N,N'*-bis(2-adamantyl-2-carboxylic acid methyl ester)oxalamide (**2**), are described (Chart 1). An influence of the *N*-methylation on the oxalamide group conformation in the solid state and in the solution is discussed.

2. Experimental

2.1. General

The one- and two-dimensional homo- and heteronuclear ^{1}H and ^{13}C NMR spectra were recorded with a Bruker AV 600 spectrometer, operating at 600.133 MHz for the ^{1}H nucleus and 150.917 MHz for the ^{13}C nucleus. Samples were measured from $CDCl_3$ solutions at 25 °C in 5 mm NMR tubes. Chemical shifts, in ppm, are referred to TMS as internal standard. IR spectra were recorded on a FT-IR ABB Bomem MB 102 spectrophotometer at room temperature. The diethyl ether solution of diazomethane was prepared from *N*-methyl-*N*-nitroso-*p*-toluensulfonamide following a classical procedure. *Caution*: diazomethane is highly toxic. Hence, this reagent must be handled carefully.

2.2. Synthesis of retropeptides *1* and *2*

A solution of diazomethane in diethyl ether was added cautiously dropwise to a stirred (0 °C) solution of 2-aminoadamantyl-2-carboxylic acid (**3**, 0.22 g, 1.1 mmol) in methanol (5 mL) in a stoppered flask protected from the light. The resulting mixture was stirred under an inert atmosphere (N_2) for about 30 min and then treated with anhydrous $CaCl_2$ to destroy excess of diazomethane. The solution was filtered and concentrated to dryness. The mixture of products, 2-aminoadamantyl-2-carboxylic acid methyl ester (**4**), *N*-methyl-2-aminoadamantyl-2-carboxylic acid methyl ester (**5**) and *N,N'*-dimethyl-2-aminoadamantyl-2-carboxylic acid methyl ester (**6**), was thereby obtained as yellowish oil and was used in the next reaction without further purification.

Subsequently, to the mixture of products in CH_2Cl_2 (5 mL) and triethylamine (170 μL, 1.22 mmol), oxalyl chloride (51 μL, 0.60 mmol) was added at 0 °C. After the addition was completed, the mixture was allowed to achieve room temperature and stirring was continued overnight. The reaction was then quenched by addition of water (5 mL) and stirred for 10 min. The organic layer was washed successively with water, saturated ammonium chloride, and water again, dried over anhydrous $MgSO_4$ and filtered. Evaporation of the filtrate followed by flash chromatography (EtOAc/hexane 1:2) gave the retropeptides **1** (59 mg, 22%) and **2** (42 mg, 16%) as a white solids, as well as nonreacted **6** (21 mg, 8%).

2.2.1. *N,N'-bis(2-adamantyl-2-carboxylic acid methyl ester)oxalamide (1)*

M.p. 218–219 °C; ^{1}H NMR ($CDCl_3$) δ/ppm: 7.68 (s, 2H, —NH), 3.74 (s, 6H, —OCH_3), 2.57 (s, 4H, H-1 and H-3), 1.68–2.03 (m, 24H); ^{13}C NMR ($CDCl_3$) δ/ppm: 26.1 and 26.4 (C-5 and C-7), 32.2 (C-1 and C-3), 32.5 and 33.8 (C-4 and C-8 + C-9 and C-10), 37.4 (C-6), 52.0 (O—CH_3), 63.6 (C-2), 158.1 (C=O), 171.6 (COO); IR (KBr): $\nu = 3394$, 2922, 1746, 1678, 1497; IR ($CDCl_3$, CaF_2): $\nu = 3388$, 2926, 1742, 1685, 1494. Anal. Calcd for $C_{26}H_{36}N_2O_6$: C, 66.08; H, 7.68; N, 5.93%. Found: C, 66.15; H, 7.71; N 5.95%.

2.2.2. *N-Methyl-N,N'-bis(2-adamantyl-2-carboxylic acid methyl ester)oxalamide (2)*

M.p. 158–159 °C; ^{1}H NMR ($CDCl_3$) δ/ppm: 7.15 (s, 1H, —NH), 3.77 and 3.73 (2s, 6H, 2× —OCH_3), 3.08 (s, 3H, N—CH_3), 2.54 (m, 4H, H-1 and H-3), 1.72–1.98 (m, 24H); ^{13}C NMR ($CDCl_3$) δ/ppm: 26.0, 26.1, 26.3, 26.6 (C-5 and C-7), 31.7 and 32.3 (C-1 and C-3), 32.8 (N—CH_3) 32.8 and 33.8 (C-4 and C-8), 32.8, 33.2, 34.4, 34.7 (C-9 and C-10), 37.0 and 37.4 (C-6), 51.9 (O—CH_3), 63.6 and 70.2 (C-2), 160.8 and 166.2 (C=O), 171.3 and 172.2 (COO). IR (KBr): $\nu = 3394$, 3287, 2907, 1734, 1718, 1681, 1665, 1551, 1453 cm^{-1}; IR ($CDCl_3$, CaF_2): $\nu = 3394$, 2920, 1741, 1722, 1690, 1658, 1507, 1458 cm^{-1}. Anal. Calcd for

$C_{27}H_{38}N_2O_6$: C, 66.64; H, 7.87; N, 5.76%. Found: C, 66.60; H, 7.81; N, 5.69%.

In order to obtain symmetrical, *N*,*N*'-dimethylated oxalamide derivative, we separated monomethylated amino acid **5** from other two derivatives **4** and **6** by flash chromatography (CH_2Cl_2/methanol 100:1), and subjected to the reaction with oxalyl chloride under the same condition. However, ^{1}H NMR spectrum did not indicate the formation of desired *N*,*N*'-dimethylated oxalamide derivative.

2.2.3. 2-Aminoadamantyl-2-carboxylic acid methyl ester (*4*)

M.p. 57 °C (lit. [14] 58 °C); ^{1}H NMR ($CDCl_3$, 600 MHz) δ/ppm: 3.72 (s, 3H, —OCH_3), 1.56–2.24 (m, 16H, Ada-H + NH_2); ^{13}C NMR ($CDCl_3$, 150 MHz) δ/ppm: 27.0 and 27.3 (C-5 and C-7), 34.3 (C-1 and C-3), 32.0 and 35.3 (C-4 and C-8 + C-9 and C-10), 37.9 (C-6), 51.8 (O—CH_3), 61.9 (C-2), 177.1 (COO).

2.2.4. N-Methyl-2-aminoadamantyl-2-carboxylic acid methyl ester (*5*)

Colourless oil; ^{1}H NMR ($CDCl_3$, 600 MHz) δ/ppm: 3.71 (s, 3H, —OCH_3), 2.20 (s, 3H, N—CH_3), 1.49–2.19 (m, 15H, Ada-H + NH); ^{13}C NMR ($CDCl_3$, 150 MHz) δ/ppm: 27.2 and 27.5 (C-5 and C-7), 29.0 (C-1 and C-3), 32.0 (N-CH_3), 32.0 and 35.1 (C-4 and C-8 + C-9 and C-10), 38.0 (C-6), 51.2 (O—CH_3), 66.4 (C-2), 175.2 (COO).

2.2.5. N,N'-Dimethyl-2-aminoadamantyl-2-carboxylic acid methyl ester (*6*)

Colourless oil which partially solidified; ^{1}H NMR ($CDCl_3$, 600 MHz) δ/ppm: 3.70 (s, 3H, —OCH_3), 2.16 (s, 6H, N—CH_3), 1.48–2.34 (m, 14H, Ada-H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ/ppm: 26.7 and 27.0 (C-5 and C-7), 30.3 (C-1 and C-3), 31.8 and 35.3 (C-4 and C-8 + C-9 and C-10), 37.8 (C-6), 37.9 (N—CH_3), 50.1 (O—CH_3), 69.2 (C-2), 170.6 (COO).

2.3. X-ray crystallographic analysis of *1* and *2*

Single crystals of **1** and **2** were grown by slow evaporation from an ethanol solution. Intensities were measured on an Enraf Nonius CAD4 diffractometer, with graphite monochromated CuK_α radiation, wavelength 1.54180 Å, using the $\omega/2\theta$ scan-technique. The crystallographic data, and details of data collections and refinements are listed in Table 1. During data collections there were no significant variations in intensities of the three control reflections, which were measured every 120 min. The data were corrected for Lorentz and polarization effects [15]. The absorption correction was based on a ψ-scan of seven reflections. Structures were solved using WinGX package [16] and refined by the package SHELXL97 [17]. Molecular geometry calculations and illustrations of the crystal packing were prepared by PLATON98 [18]. The molecule of **1** involves the crystallographic inversion symmetry and an asymmetric unit comprises a half of molecule ($Z = 2$).

Table 1
Crystal data and summary of data collection and refinement for compounds **1** and **2**

	1	**2**
Formula	$C_{26}H_{36}N_2O_6$	$C_{27}H_{38}N_2O_6$
Formula weight	472.57	486.59
Crystal size (mm)	0.2 × 0.1 × 0.1	0.2 × 0.3 × 0.3
Crystal colour	Colourless	Colourless
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/a$
a (Å)	13.1184(4)	9.7714(18)
b (Å)	6.7477(2)	10.421(13)
c (Å)	13.9360(4)	24.36(3)
β (°)	98.320(2)	95.05(7)
V (Å^3)	1220.62(6)	2471(4)
Z	2	4
D (g cm^{-3})	1.286	1.308
F (000)	508	1048
Index range for data collection	−16:16; 0:8; 0:17	0:12; 0:13; −30:30
Collected reflections	2689	5496
Independent reflections/(R_{int})	2575(0.014)	5171(0.036)
Data/restrains/parameters	2,575, 214	5,171, 320
Weighting parameters	1/[$\sigma^2(F_o^2) + (0.0634P)^2 + 0.3331P$] where $P = (F_o^2+2F_c^2)/3$	1/[$\sigma^2(F_o^2) + (0.1009P)^2 + 0.8155P$] where $P = (F_o^2+2F_c^2)/3$
Goodness-of-fit on F^2	1.14	1.04
R	0.0471	0.0640
wR	0.1339	0.1933
Min/max residual electron density	−0.28, 0.23	−0.40, 0.47

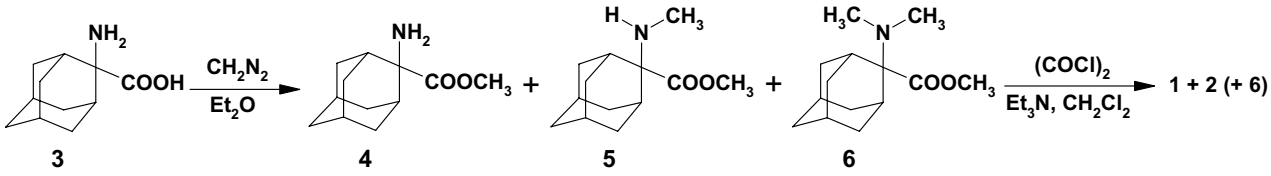
Atomic scattering factors were those included in SHELXL97.

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers CCDC-611811 and CCDC-611812. Copies of data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 (0)1223 336033 or e-mail: deposit@ccdc.cam.ac.uk).

3. Results and discussion

The new oxalamide derivatives were synthesized using 2-aminoadamantyl-2-carboxylic acid **3** prepared as described previously [19,20]. The direct *N*-methylation of **3**, performed with a large excess of diazomethane, provided mixture of three products, amino acid methyl ester **4**, and the corresponding mono- and dimethylated derivatives **5** and **6** in the ratio of 4.5:2:1, respectively. The mixture of methyl esters **4**, **5** and **6** was then subjected to the reaction with oxalyl chloride in the presence of triethylamine and retropeptides **1** and **2** as well as the nonreacted dimethylated derivative **6** were obtained (Scheme 1).

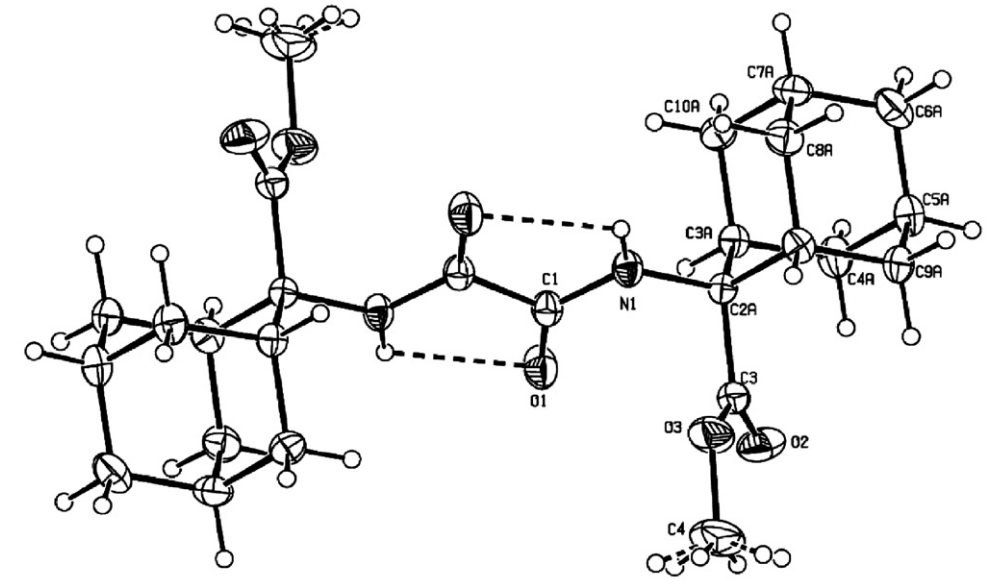
The structures of retropeptides **1** and **2** were confirmed by one- and two-dimensional homo- and heteronuclear ^{1}H and



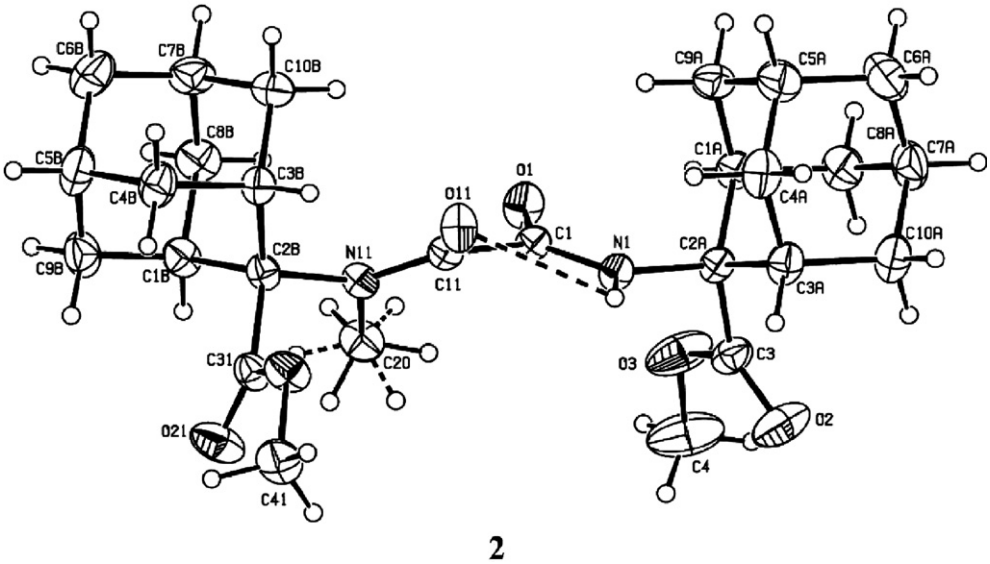
Scheme 1. Synthesis of retropeptides **1** and **2**.

^{13}C NMR measurements. The adamantane-proton resonance appeared as a multiplet at 1.6–2.6 ppm in both ^{1}H NMR spectra of **1** and **2**. The amide proton of **2** is shifted upfield by about 0.5 ppm relative to their counterpart in **1**.

The role of the oxalamide functions in the self-assembling of **1** and **2** could be probed through the NMR and IR measurements. Temperature-variable ^{1}H NMR measurements of **1** and **2** in the $CDCl_3$ (5 mM) within the range



1



2

Fig. 1. Molecular structures and atom labelling of **1** and **2**. The molecular symmetry of **1** is C_i whereas **2** reveals a pseudo twofold axis perpendicular to the bond C1—C11. The methyl groups in **1** and *N*-methyl group in **2** are disordered over two positions. Displacement ellipsoids are drawn at the 30% probability level.

of 298–325 K show a relatively low temperature dependence for the chemical shifts of the amide proton ($\Delta\delta/\Delta T = -1.4$ ppb/K for **1** and -2.7 ppb/K for **2**, respectively). Furthermore, ^{1}H NMR spectra recorded upon adding up to 20% DMSO-d_6 to solutions of **1** and **2** in $CDCl_3$ show that the signal of the NH proton exhibits no change for **1** and the small change for **2** ($\Delta\delta = 0.19$ ppm) with increasing amounts of DMSO-d_6. These results suggest that no intermolecular hydrogen bonds are formed in chloroform solution. Further support for the absence of intermolecular hydrogen bonds between oxalamide units in solution came from the lack of any concentration dependence (30–5 mM) of the IR spectra of **1** and **2** in chloroform solution; the IR spectra showed a weak band at 3388 cm^{-1} (for **1**) and 3394 cm^{-1} (for **2**), respectively.

In solid state (KBr matrix), the IR spectrum of retropeptide **1** showed a single broad band appearing at 3394 cm^{-1}. There are also the amide I band at 1678 cm^{-1} and the NH bending peak at 1497 cm^{-1} (amide II) indicating the intramolecular hydrogen bonding [5,6]. However, the IR spectrum of *N*-methylated derivative **2**, showed a weak band at 3394 cm^{-1} and a strong peak at 3287 cm^{-1} indicating both intramolecular as well as intermolecular hydrogen bonding between oxalamide units in solid state. Further support for the existence of intramolecular and intermolecular hydrogen bonding are two amide I bands at 1658 and 1681 cm^{-1} and the NH bending peaks at 1453 and 1551 cm^{-1} (amide II). Due to a disruption of the intermolecular hydrogen bonds between the oxalamide groups of the adamantane molecules, in the IR spectrum of the $CDCl_3$ solutions of **2**, one of the amide I band is shifted to higher wave number (1690 cm^{-1}). On the other hand, the NH bending peak is shifted to lower wave number (1507 cm^{-1}).

Table 2
Selected torsional angles

	1	2
O1—C1—C11—O11	180.0	−132.7(2)
C2A—N1—C1—C1^i(C11)	−179.38(12)	−172.4(2)
C2B—N11—C11—C1	–	−165.85(19)
C1—N1—C2A—C3	−50.43(17)	−86.6(3)
C11—N11—C2A—C31	–	−92.3(2)
N1—C2A—C1A—C8A	−59.81(15)	−61.5(3)
N11—C2B—C1B—C8B	–	−63.9(3)
N1—C2A—C3—O2	131.88(17)	−107.0(3)
N11—C2B—C31—O21	–	−85.9(3)

Single crystals of **1** and **2** suitable for X-ray structure analysis were successfully prepared from ethanol solutions. The results of structure analysis of **1** and **2** (Fig. 1) are in agreement with spectroscopic evidences obtained by NMR and FT-IR measurements.

The overall molecular conformations of **1** and **2** are different (Fig. 1 and Table 2). The orientation of the bulky adamantane moieties with respect to the oxalamide group is *anti* in **1** whereas in *N*-methylated analogue **2** is *syn*. The central oxalamide unit in the molecule **1** is strictly planar due to the symmetry (the crystallographic and molecular symmetry C_i), whereas in the compound **2** it deviates substantially from the planarity (Fig. 2) [torsional angle O1—C1—C11—O11 is −132.7(2)°, Table 2].

The non-planarity of the central unit in **2** is induced by sterical hindrances of the methyl group attached to N11. The results of crystal packing analysis of **1** and **2** are consistent with the spectroscopic data. In the solid state and in the solution analyses reveal that there are no intermolecular hydrogen bonds in **1** whereas in **2** shows a presence of the intermolecular N—H···O [d(H···A) = 2.08 Å, d(D–A) = 2.95 Å and α(D–H···A) = 162.8°] hydrogen bonds.

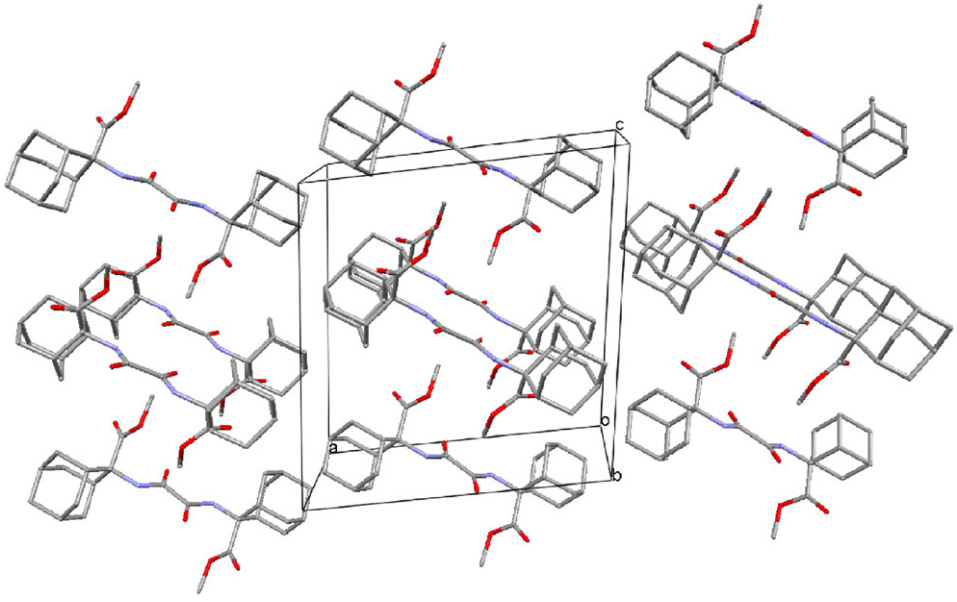


Fig. 2. Crystal packing of **1** dominated by van der Waals contacts. Hydrogen atoms are omitted.

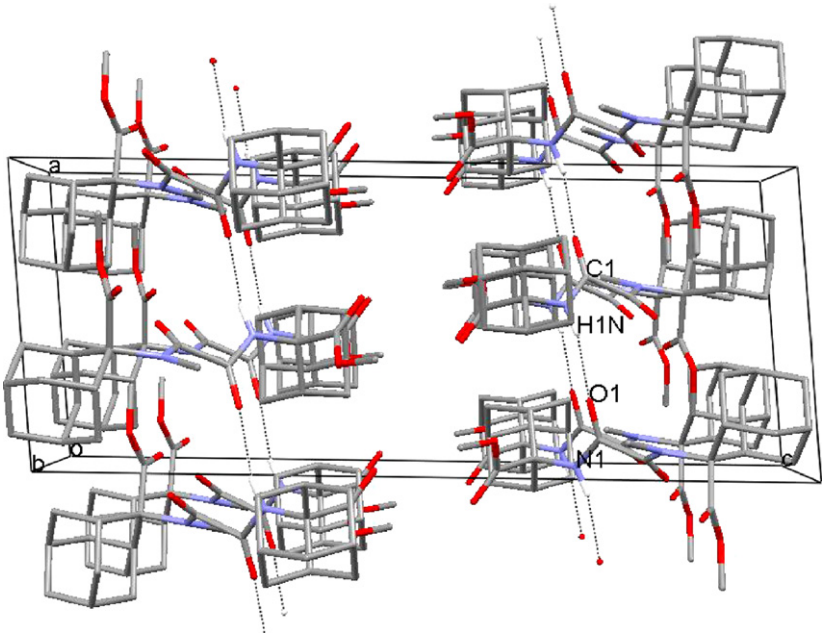


Fig. 3. Crystal packing of **2**, with hydrogen bonded chains along the *a* axis (dashed lines).

The substitution of bulky adamantane cages on both terminal sides of the molecules prevents the intermolecular hydrogen bond between central oxalamide units. Although the oxalamide units are in proper orientation, i.e. they are coplanar; the separation distance is too large. The characteristic distance for oxalamide units in the presence of N—H···O hydrogen bonds is about 5 Å [5,6,21]. In the compound **1**, this distance is exactly the length of the crystallographic *b* axis, i.e. 6.75 Å (Fig. 2). However, two symmetrically and chemically identical intramolecular N—H···O [d(H···A) = 2.25 Å, d(D–A) = 2.69 Å and α(D–H···A) = 110.7°] hydrogen bonds within oxalamide bridge of a pseudo-C5 type are present stabilizing the planarity of the oxalamide group.

In the crystal structure of the compound **2** the neighbouring molecules are separated by $a/2 = 4.88$ Å, which is less than the characteristic distance in oxalamides connected by two parallel hydrogen bonds [22]. Thus, the close contacts between oxalamide donor and acceptor sites are not obstructed by the bulky adamantane cages (Fig. 3) and the non-planar oxalamide units can be packed closely. The non-methylated nitrogen atom N1 forms intermolecular hydrogen bond with O1 atom of the oxalamide unit of the molecule operated by the glide plane in the *a* direction [N1···O1 of 2.951(4) Å].

Ab initio HF/6-31G(d) calculations of **2** predicted a stable conformer with geometry very close to that obtained by X-ray analysis; torsional angle O1—C1—C11—O11 is −144.5°[−132.7(2)°, Table 2]. The constrained optimization with torsional angle O1—C1—C11—O11 fixed to 180° gave conformer which is by 9.51 kcal mol^{-1} less stable than fully optimized molecule **2**. The results of these gas-phase calculations indicate that crystal packing forces are not the major driving force for deviation from planarity. The methyl substitution increases steric hindrance leading to non-planar conformation of oxalamide unit.

4. Conclusions

Two new retropeptides, conformationally restricted adamantane-substituted oxalamide derivatives **1** and **2** were synthesized in a moderate yields. Both ^{1}H NMR and FT-IR studies in the solution are in agreement with results obtained in the solid state by X-ray structure analysis. The oxalamide group of **1** is planar while in the monomethylated derivative **2** the oxalyl-carbonyls are in a skew-conformation. The conformational preferences of other methylated oxalamide derivatives are under current investigation.

Acknowledgments

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