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Short Note

N,N′-Dipropyloxamide

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Abstract: *N,N′*-Dipropyloxamide (**1**) was synthesised by the reaction between diethyloxalate and *n*-propylamine in ethanol. Compound **1** was fully characterised by both microanalytical (elemental analysis, melting point determination) and spectroscopic means (FT-IR and NMR spectroscopy). Crystals suitable for single crystal X-ray diffraction were isolated by the slow evaporation of a methyl alcohol solution of the compound. The resulting crystal structure shows the prominent role exerted by intermolecular hydrogen bonds in the crystal packing.

Keywords: *N,N′*-dialkyloxamides; oxalamides; oxalic acid derivatives; hydrogen bond; XRD

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1. Introduction

Derivatives of oxamic acid (oxalic acid monoamides) represent a family of compounds with applications in research fields that span from medicine [1,2] to organic synthesis [3,4] and cultural heritage [5–9]. Within this class of compounds, the oxalamide moiety (*N,N′*-diamide of oxalic acid) has been employed for the synthesis of biologically active compounds [10,11], precursors of widely used chemicals such as ethylene glycol [12,13], and ligands in coordination chemistry [14,15]. Moreover, oxalamides are self-complimentary hydrogen bonding molecules capable of donating and receiving two hydrogen bonds [16,17]. This property, combined with their persistent self-assembly behaviour, also allows oxalamide groups to participate in interesting hydrogen bonding (HB) motifs with applications in crystal engineering [18,19], protein engineering [20], organic gelators [21,22], and materials science [23].

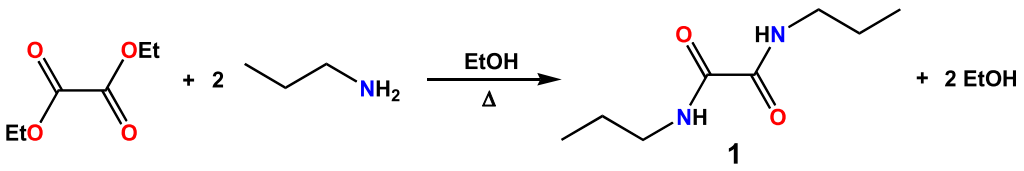
Among *N,N′*-dialkyloxamides, *N,N′*-dipropyloxamide (**1**) has been employed as a precursor for *N,N′*-dialkylureas [24] and primary *N*-nitramines [25], and it has been studied by means of electronic spectroscopy [26,27], vibrational and NMR spectroscopy, thermal analysis, and ab initio calculations [28]. Despite this, the crystal structure of compound **1** was never reported, notwithstanding the importance and applications of the crystal packing interactions described above for this class of compounds, and the advantage of comparing the interactions in differently substituted derivatives.

We report here on the preparation and characterisation of *N,N′*-dipropyloxamide (**1**), and we describe for the first time its single-crystal XRD structural determination.

2. Results and Discussion

The synthesis of *N,N′*-disubstituted oxalamides is traditionally carried out by reacting oxalyl chloride and the desired amine [29,30], but more recently, *N,N′*-dialkyloxamide derivatives were also prepared by different methods, such as the catalytic carbonylation of amines [31]. We have instead synthesised compound **1** starting from diethyloxalate, which was reacted with *n*-propylamine in a 1:2 molar ratio, following the general method

first described by Rice and co-workers in 1953 (Scheme 1) [32]. In particular, a mixture of the reagents in ethyl alcohol was refluxed under stirring overnight, and upon cooling down to room temperature, a white solid precipitated, which was isolated by filtration and recrystallised from methanol.



Scheme 1. Synthetic pathway for the synthesis of compound **1**.

Compound **1** was fully characterised by different means, including elemental analysis, melting point determination, FT-IR, ^{1}H-, and ^{13}C-NMR spectroscopy.

In the FT-IR spectrum of compound **1**, the peak corresponding to the stretching mode of the N–H bond falls at 3300 cm^{-1}, while the C=O stretching mode can be observed at 1653 cm^{-1}. The peaks corresponding to the stretching modes of the C–H bonds in the aliphatic chain fall in the range 2850–3000 cm^{-1} (Figure S1). In the ^{1}H-NMR spectrum recorded in $CDCl_3$ for compound **1** (Figure S2), the signal corresponding to the N–H protons appears as a broad singlet at 7.50 ppm. On the other hand, the quadruplet corresponding to the -CH_2- groups next to the nitrogen atoms falls at 3.27 ppm, and the signals of the remaining -CH_2- and -CH_3 groups of the alkyl chain are featured at 1.58 and 0.95 ppm, respectively. In the ^{13}C{^{1}H}-NMR spectrum of compound **1**, also recorded in deuterated chloroform, the signal corresponding to the carbonyl groups resonates at around 160 ppm, while those of the remaining carbon atoms in the *n*-propyl chain fall between 11 and 42 ppm (Figure S3).

Compound **1** was recrystallised through the slow evaporation of a concentrated methanol solution of the compound, leading to the formation of colourless needle-shaped crystals suitable for single-crystal X-ray diffraction analysis. The compound crystallises in the triclinic *P*-1 space group (Tables S1–S4), the asymmetric unit containing half of the molecule (Figure 1). The carbonyl moiety showed the common antiperiplanar orientation with an inversion centre located halfway between the sp^2-hybridised carbon atoms, making the oxalamide unit completely planar (Table S4). Previous studies demonstrated theoretically that the antiperiplanar conformation is indeed a minimum in the potential energy surface (PES) of *N*,*N*′-disubstituted oxalamides [9]. An examination of 258 different *N*,*N*′-disubstituted oxamides found in the Cambridge Structural Database (CSD) [33] showed average distances of 1.533, 1.226, and 1.328 Å for the C–C, C=O, and C–N bonds, respectively; very similar values were found in compound **1**, the C4–C4^{i} bond length amounting to 1.5366(18) Å, with a C4–O1 distance of 1.2370(11) Å, and a C4–N1 bond length of 1.3298(11) Å (Table S2).

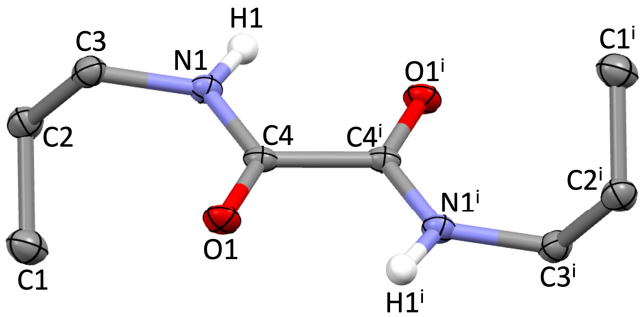
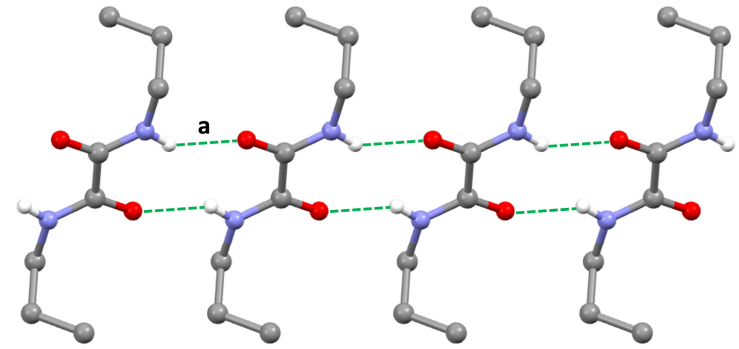


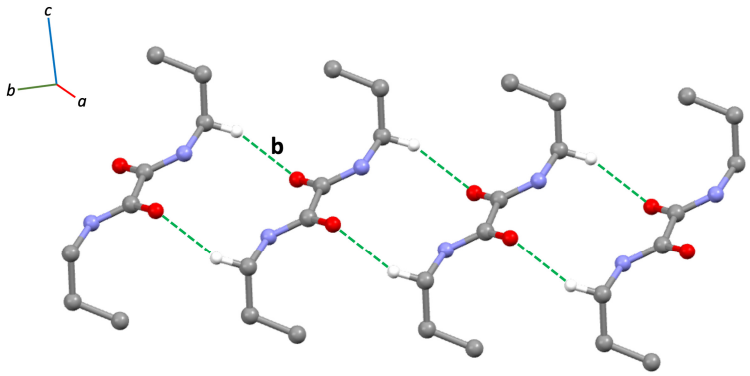
Figure 1. Molecular structure and atom labelling scheme for compound **1**. Thermal ellipsoids are drawn at 50% probability level; hydrogen atoms other than those of the N–H groups were omitted for clarity; ^{i} = 2–x, 1–y, 1–z.

The two *n*-propyl chains at the nitrogen atoms are on opposite sides of the oxamide unit and feature N1–C3–C2–C1 torsion angles of 68.91(11)° (Table S4).

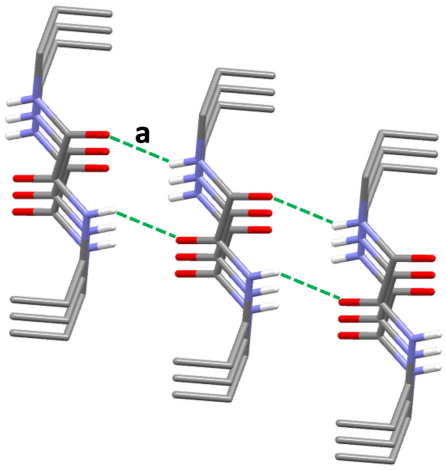
As expected, the crystal packing of compound **1** is governed by N–H···O HB interactions. In particular, neighbouring molecules whose oxamide units lie on the same plane interact through $R_2^2(10)$ HB motifs [34,35] running along the *b*-axis (Figure 2A, Table 1). These interactions are similar to those observed for other oxalamides featuring alkyl [17,28,36,37] or aryl [38–40] substituents.



(**A**)



(**B**)



(**C**)

Figure 2. (**A**) View of the crystal packing of compound **1** along the *a*-axis, showing the intermolecular N–H···O hydrogen bonding (HB) interactions (**a**, Table 1); (**B**) partial view of the crystal packing of compound **1** showing intermolecular C–H···O interactions (**b**, Table 1); (**C**) view of the crystal packing of compound **1** along the *b*-axis, showing the stacking of ribbons formed through HB interactions; only hydrogen atoms involved in intermolecular interactions are shown for clarity.

Table 1. Intermolecular interactions of compound **1**.

Interaction		$d_{D–H}$ (Å)	$d_{H\cdots A}$ (Å)	$d_{D\cdots A}$ (Å)	$\alpha_{D–H\cdots A}$ (°)
a	O1···N1^{ii}–H1^{ii}	0.87(2)	2.11(2)	2.882(9)	146.4(13)
b	O1···C3^{iii}–H3^{iii}	0.99	2.601	3.500(1)	151.0

Symmetry operations: ^{ii} = x, −1 + y, z; ^{iii} = 1 + x, −1 + y, z.

The ribbons formed by the described HBs along the *b*-axis are joined by C–H···O intermolecular interactions connecting the oxygen atoms at the carbonyl groups from one ribbon to the -CH_2- groups next to the nitrogen atoms of another ribbon (Figure 2B, Table 1), leading to a stacking perpendicular to the *b*-axis (Figure 2C).

The described crystal packing is very similar to that recently described for the analogous compound *N*,*N*′-dibutyloxamide [41].

3. Materials and Methods

3.1. General Methods

Solvents and reagents were obtained from TCI, FluoroChem, and Merck and were employed without further purification. Deuterated solvents were obtained from Eurisotop and stored over molecular sieves prior to use. FT-IR measurements were recorded at room temperature on a Thermo-Nicolet 5700 spectrometer on KBr pellets by using a KBr beam splitter and KBr windows (4000−400 cm^{-1}, resolution 4 cm^{-1}). ^{1}H- and ^{13}C-NMR spectra were recorded in $CDCl_3$ at room temperature on a Bruker Avance III HD 600 spectrometer. Chemical shifts are reported in ppm (δ). The residual ^{1}H and ^{13}C peaks of the deuterated solvent ($CDCl_3$) were used for chemical shift calibration. Elemental analysis was performed with a CHNS/O PE 2400 series II elemental analyser (T = 925 °C). Uncorrected melting points were determined in capillaries on a FALC mod. C melting point apparatus.

3.2. X-ray Diffraction Analysis

X-ray diffraction data for compound **1** were collected at 100(2) K on a Rigaku FRE+ diffractometer, equipped with VHF Varimax confocal mirrors, an AFC12 goniometer, and a HyPix 6000 detector. The structure was solved with the ShelXT [42] solution program using dual methods, and the model was refined with ShelXL 2014/7 [43] using full matrix least squares minimisation on F^2. Olex2 1.5 [44] was employed as the graphical interface.

3.3. Synthesis of N,N′-Dipropyloxamide (**1**)

Compound **1** was synthesised by reacting 1.22 g of *n*-propylamine (20.6 mmol) dissolved in 5 mL of ethanol with 1.50 g of diethyloxalate (10.3 mmol) diluted in 5 mL of the same solvent. The resulting mixture was refluxed under stirring overnight and then allowed to cool down to room temperature, resulting in the precipitation of a white solid. The solid was isolated by filtration on a Gooch funnel, washed with *n*-hexane, and air dried. The purity of the isolated product was confirmed by TLC (stationary phase: silica gel; eluent: dichloromethane/ethyl acetate 2:1). Recrystallisation by slow evaporation of a methanol solution of the product afforded colourless needle-shaped crystals suitable for XRD analysis (1.26 g; 7.3 mmol; Y = 71%). M. p. = 161.8 °C (Lit.: 162.5–163.5 °C [45]). Elemental analysis calcd (%) for $C_8H_{16}N_2O_2$: C 55.79, H 9.36, N 16.27. Found: C 55.26, H 9.32, N 16.35. FT-IR (KBr, 4000–400 cm^{-1}): 3300 s, 3059 w, 2964 m, 2931 m, 2873 m, 1649 vs, 1529 m, 1460 w, 1439 w, 1383 w, 1363 vw, 1344 w, 1308 w, 1281 vw, 1255 w, 1227 m, 1146 m, 764 s, 550 s, 459 m cm^{-1}. ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 7.50 (s, br, 2H, NH), 3.27 (q, 4H, $CH_2CH_2CH_3$), 1.58 (q, 4H, $CH_2CH_2CH_3$), 0.95 (t, 6H, $CH_2CH_2CH_3$) ppm. ^{13}C{^{1}H}-NMR (151 MHz, $CDCl_3$) δ: 160.1, 41.5, 22.7, 11.4 ppm.

4. Conclusions

N,*N*′-Dipropyloxamide (**1**) was synthesised starting from diethyloxalate with *n*-propylamine and structurally characterised by single crystal X-ray diffraction.

The crystal structure of compound **1** shows the antiperiplanar conformation peculiar to planar oxalamide core with the *n*-propyl alkyl chains protruding on opposite sides.

In the crystal packing, several intermolecular interactions can be observed, the main ones consisting of N–H···O HBs between adjacent molecules featuring oxalamides core lying on the same plane.

These HB interactions are typical of *N*,*N*′-dialkyloxamides, and the resulting self-assembly behaviour can have applications in different fields of crystal engineering.

Supplementary Materials: The following supporting information can be downloaded online. Figure S1: FT-IR spectrum; Figure S2: ^{1}H-NMR spectrum; Figure S3: ^{13}C{^{1}H}-NMR spectrum; Table S1: crystal data and refinement parameters; Table S2: bond Lengths; Table S3: bond Angles; Table S4: torsion Angles.

Author Contributions: Conceptualisation: A.P.; Methodology: A.P., M.A. and M.C.A.; Investigation: A.P., E.D. and E.P.; Data Curation: A.P., E.D., E.P. and S.J.C.; Writing—original draft preparation: A.P.; Writing—review and editing: A.P., E.D., E.P., M.A., M.C.A., V.L. and S.J.C. All authors have read and agreed to the published version of the manuscript.

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