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Original Scientific Paper

On the Hydrogen Bonding of Succinimide Derivatives: Crystal Structure of 3(4-Pyridil-methyl)amino-pyrrolidine-2,5-dione*

*Gyula Argay, László Fábián, and Alajos Kálmán***

Institute of Chemistry, Chemical Research Center, Hungarian Academy of Sciences, H-1525 Budapest 114, P.O.Box 17, Hungary

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The structure of 3(4-pyridil-methyl)amino-pyrrolidine-2,5-dione has been established by X-ray crystallography from diffractometer data with an F^2 refinement to $R = 0.0526$ for 1999 observed reflections. The close packing of orthorhombic crystals [space group *Pbca* (No. 61) with $a = 11.595(4)$, $b = 8.325(1)$, $c = 20.452(4)$ Å, $V = 1974.2(8)$ Å^3, $Z = 8$, $D_c = 1.381$ Mg/m^3, $\mu = 0.824$ mm^{-1}] is built of herringbone-like molecular layers folded along the c axis and fused together by infinite chains of NH ··· O and NH ··· N hydrogen bonds perpendicular to one another. This prompted us to review and characterize the hydrogen bond networks formed by succinimides possessing an unsubstituted >NH group by the use of the Cambridge Structural Database (CSD), and our own earlier structure determinations. The common motifs of the hydrogen bond systems are analyzed using graph descriptions suggested by M. C. Etter. The influence of the hydrogen bonds on molecular close packing is also discussed.

Key words: X-ray structure, hydrogen bonds, succinimides.

INTRODUCTION

As revealed by a search in CSD[1] (March 1998 release), the crystal structures of several succinimide (pyrrolidine-2,5-dione) derivatives have been

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* Dedicated to Professor Boris Kamenar on the occasion of his 70th birthday.

** Author to whom correspondence should be addressed. (E-mail: akalman@cric.chemres.hu)

reported with respect to their biological properties, *e.g.* antiepileptic (MRMPYR),[2] anticonvulsive (MRMPYR[2], VAYTUK[3], VAYVAS[3], SORCYL[4], WISFAF[5]), fungicidal (GIDPAK, GIDPEO, GIDPOY)[6] and other pharmacological (KEKXIH[7]) activities. In each of these compounds, the ring nitrogen is substituted either by methyl group (SORCYL), or differently substituted phenyl (GIDPAK, GIDPEO, GIDPOY), and pyridine rings (KEKXIH, VAYTUK, VAYVAS, WISFAF) or morpholinomethyl (MRMPYR) moiety. Only in HEMVIE[8] (aldose reductase inhibitor) and REWWOF[9] (cytotoxic), there is an >NH group which participates in the formation of intermolecular hydrogen bond. The present work, starting from the study of the structure and packing of the title compound, aims to investigate those succinimide derivatives (16 entries*** from the 96 archived structures) in which the ring nitrogen is not substituted, and therefore it may form hydrogen bond(s) either with one of the vicinal carbonyl groups or other acceptors.

EXPERIMENTAL DETAILS

Preparation

This part of the work, in particular the synthesis of 1-morpholinomethyl-3-phenylpyrrolidine-2,5-dione,[2] registered under the names »perlepsyn« (in Hungary) and »morpholep«[10] as an efficacious antiepileptic drug, has been described in detail in Ref. 2a.

Structure Solution and Refinement

The X-ray data collection was performed on an Enraf-Nonius CAD4 diffractometer using graphite monochromated Cu-Kα radiation (λ = 1.54180 Å) at room temperature. Table I lists general and crystallographic data and details of data collection and refinement. The intensities of three standard reflections were monitored every 60 min. and a correction for 1% decay was applied. Semi-empirical absorption correction (psi-scans) was also performed. The structure solved by the direct method was refined by full-matrix least-squares on F^2 for all non-hydrogen atoms in anisotropic mode. Hydrogen positions H2a and H3a were located in difference maps, while the others were calculated from assumed geometries. All hydrogen atoms were included in structure factor calculations but their positions (except for N2–H2a and N3–H3a distances) were not refined. Isotropic displacement parameters of the hydrogen atoms were approximated by the U_{eq} value of the corresponding heavy atoms.

*** Of which succinimide complexes with inorganic (HIBDOL) and organic salts (WEZVOM, ZZZROG01) and bulky host molecules like melanine (REGKOD) are deliberately omitted from the analysis.

TABLE I

Crystallographic data for the title compound

Empirical formula	C_{10} H_{11} N_3 O_2
Molecular weight, M_r	205.22
Temperature / K	293(2)
Radiation and wavelength / Å	Cu-Kα; λ = 1.54180
Crystal system and space group	orthorhombic, *Pbca* (No. 61)
a / Å	11.595(4)
b / Å	8.325(1)
c / Å	20.452(4)
V / Å^3	1974.2(8)
Z	8
Density (calculated), D_c / Mg m^{-3}	1.381
μ / mm^{-1}	0.824
F(000)	864
Crystal size / mm^3	0.30 × 0.18 × 0.04
θ range for data collection / °	4.32 ≤ θ ≤ 73.62
Index ranges	–2 ≤ h ≤ 14; –2 ≤ k ≤ 10; –25 ≤ l ≤ 10
Reflections collected/observed [I>2σ(I)]	2092/1080
Independent reflections	1999 [R(int) = 0.0044]
Completeness to 2θ	100%
Max. and min. transmission	0.9678 and 0.7902
Data / restraints / parameters	1999 / 0 / 138
Goodness-of-fit on F^2	0.938
Final R indices [I>2σ(I)]	R1 = 0.0526, wR2 = 0.1309
R indices (all data)	R1 = 0.1061, wR2 = 0.1477
Max. and mean shift/esd	0.000, 0.000
Largest diff. peak and hole / e Å^{-3}	0.248 and –0.240

Weighting scheme used in refine_ls_weighting_details:
$w = 1/[\sigma^2 (F_o^2) + (0.0814P)^2 + 0.0000P]$ where $P = (F_o^2 + 2F_c^2)/3$.

Neutral atomic scattering factors were taken from the *International Tables for Crystallography* Vol C.[11] Computer programs used are: XCAD4,[12] DATCOR,[13] SHELXS-97,[14] SHELXL-97[15] and ORTEPIII.[16] Fractional atomic coordinates, bond lengths and bond angles as well as the atomic displacement parameters have been deposited at the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 106920. Copies of data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+ 44)1223–336–033; e-mail deposit@ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Fractional atomic coordinates of the non hydrogen atoms obtained for the title compound are listed in Table II. The molecule (Figure 1) is folded around the $–CH_2–NH–$ bond with two antiperiplanar torsion angles C5–C6––N2–C7 = 172.7(2)° and C6–N2–C7–C8 = 171.6(2)°, respectively. The LSQs

TABLE II

Atomic coordinates (x, y, $z \times 10^4$) and equivalent isotropic displacement parameters ($U_{eq} \times 10^3$ / Å^2) for the title compound

	x	y	z	U_{eq}
C1	4802(2)	3413(3)	653(1)	48(1)
C2	4807(2)	2443(4)	108(1)	52(1)
N1	3968(2)	2438(3)	–336(1)	50(1)
C3	3088(2)	3433(4)	–235(1)	54(1)
C4	3016(2)	4443(3)	299(1)	51(1)
C5	3889(2)	4460(3)	753(1)	42(1)
C6	3870(3)	5573(3)	1333(1)	54(1)
N2	3579(2)	4691(2)	1931(1)	42(1)
C7	3701(2)	5705(3)	2515(1)	42(1)
C8	3207(2)	4767(3)	3093(1)	44(1)
N3	4087(2)	4458(3)	3516(1)	46(1)
C9	4949(2)	5983(3)	2692(1)	49(1)
C10	5134(2)	5041(3)	3305(1)	46(1)
O1	2226(2)	4316(3)	3168(1)	65(1)
O2	6038(2)	4801(3)	3591(1)	71(1)

plane of the ring bridging atoms C5,C6,N2,C7,C8 is almost perpendicular [98.8(1)°] to that of the pyridine ring while it makes an angle of 111.8(2)° with the best plane of the slightly puckered [Q = 0.070(2) Å][17] succinimide moiety. The dihedral angle between the succinimide and pyridine rings is 104.20(7)°, with an intramolecular N1 ··· N3 distance of 8.056(3) Å. These corrugated molecules, related by the glide plane c in space group *Pbca,* form infinite hydrogen bond chains of N ··· N type with the terminal N3/N1 atoms in donor/acceptor function. In addition, the herringbone-like molecular ribbons are crosslinked by NH ··· O hydrogen bonds generated by glide plane a (Table III). The herringbone-like close packing of the corrugated molecular

TABLE III

Hydrogen bonds and density for the structures discussed

D–H ··· A	H···A/Å	D···A/Å	D–H···A/°	D_c/Mg m^{-3}
Title compound				1.381
N2–H2a ··· O2[*x*–1/2, *y*, –*z*+1/2]	2.28	3.135(3)	145.48	
N3–H3a ··· N1[*x*, –*y*+1/2, *z*+1/2]	2.03	2.833(3)	164.5	
SUCCIN01				1.401
N1–H1 ··· O2[–*x*, –*y*, –*z*]	2.02	2.875	176.9	
SUCCIN02				1.440
N1–H1 ··· O2[–*x*, –*y*, –*z*]	2.02	2.875	177.3	
PHAMPD[19]				1.377
N2–H10 ··· O2[–*x*, –*y*, –*z*]	1.92	2.901	176.7	
PHPYRO[20]				1.281
N1–H93 ··· O1[–*x*–2, –*y*, –*z*+1/2]	2.04	2.859	172.1	
TANPUT[23]				1.517
N1–H8 ··· O6[*x*, *y*, *z*]	2.04	2.902	171.1	
N3–H16 ··· O2[*x*, *y*+1, *z*]	1.89	2.826	175.3	
BEPSCM[24]				1.626
N1–H8 ··· O2[–*x*+1, –*y*+1/2, *z*+1/2]	2.12	2.823	172.9	
BZAPYR[25]				1.314
O3–H21 ··· O1[*x*, –*y*+1/2, *z*+1/2]	2.12	2.920	152.2	
N1–H31 ··· O3[*x*, *y*, *z*]	1.79	2.880	173.2	
N2–H32 ··· O3[–*x*, *y*–1/2, –*z*–3/2]	1.90	2.808	174.9	
CUBRUM[27]				1.489
N14–H14 ··· O17[–*x*+1, *y*+1/2, –*z*+1/2]	2.49	3.297	156.0	
N14–H14 ··· O13[*x*, –*y*+1/2, *z*+1/2]	2.54	2.987	113.3	
N18–H18 ··· N1[*x*+1, –*y*+1/2, *z*–1/2]	1.83	2.902	165.7	
N6–H61 ··· O19[*x*–1, –*y*+1/2, *z*+1/2]	2.04	2.955	172.3	
N6–H62 ··· N3[–*x*, *y*–1/2, –*z*+1/2]	2.29	3.065	169.9	
HEMVEA[8]				1.699
N2–H5 ··· O1[–*x*+2, *y*–1/2, –*z*–1/2]	1.79	2.937	155.4	
HEMVIE[8]				1.750
N2–H4 ··· O1[–*x*–1, *y*–1/2, –*z*+1/2]	2.24	3.016	139.9	
REWWOF[9]				1.295
N1–H1 ··· O2[*x*+1/2, –*y*+3/2, –*z*+1]	1.94	2.826	167.0	
O2–H5 ··· O1[–*x*+3, *y*–1/2, –*z*+1/2]	1.79	2.937	155.4	

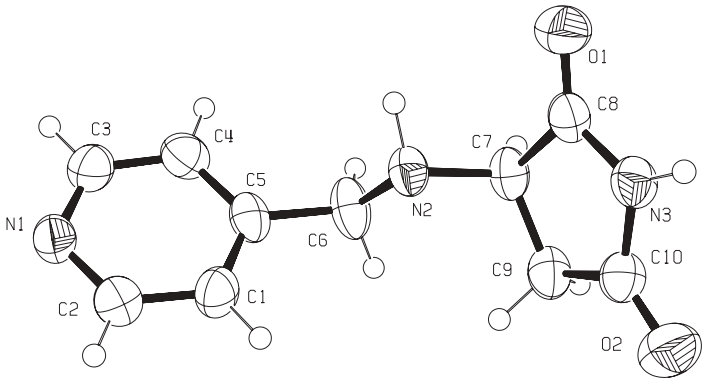
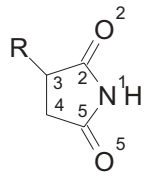


Figure 1. An ORTEP diagram of the title compound with non H-atom labels. The atoms are represented by their 50% of probability ellipsoids for thermal motion.

sheets depicted in Figure 2 prompted us to compare it to the hydrogen bond networks generated primarily by the ring NH group in other succinimide derivatives.

The crystal structure of the parent molecule (*i.e.* succinimide or pyrrolidine-2,5-dione) has been archived three times in CSD as SUCCIN,[18] SUCCIN01 and SUCCIN02 (the last two entries are private communications). In the orthorhombic unit cell (space group *Pbca*), there are eight molecules forming four dimers. These dimers are built up around crystallographic centres of inversion by NH $\cdots$ O pairs (their parameters are listed in Table III), resulting in a density of about 1.40 Mg m^{-3}. Similarly, in PHAMPD[19] there are four dimers of 3-(*N*-phenyl)aminopyrrolidine-2,5-dione formed with the same space group *Pbca* (Figure 3). The H $\cdots$ O distance of 1.92 Å is rather short and accompanied by a high D–H $\cdots$ A angle of 177°. Here, in contrast to the parent structure, the acceptor oxygen O^5 (Scheme 1) can be distinguished from the inactive one owing to the ring substituent. The latter oxy-



Scheme 1.

gen O^2 is engaged in an intramolecular hydrogen bond donated by the secondary amine group from the substituent R.

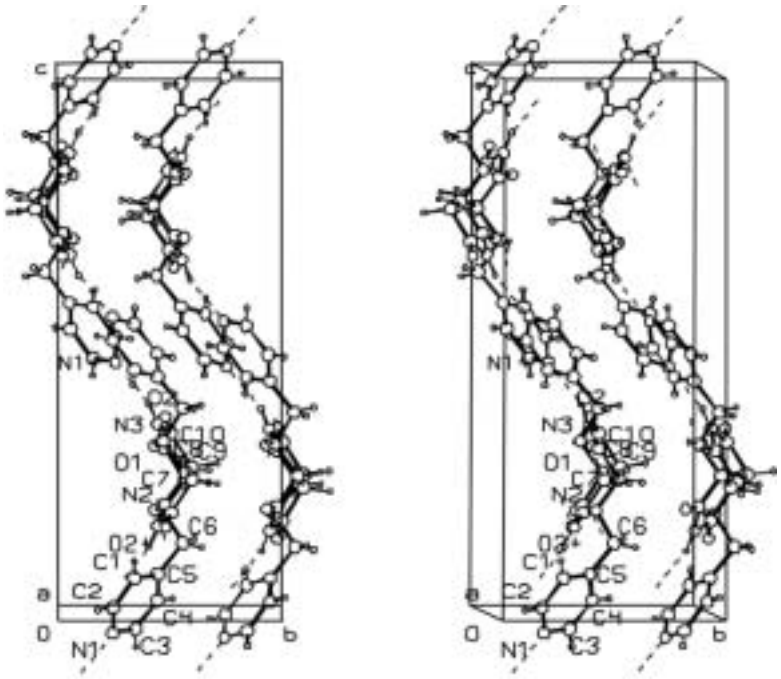


Figure 2. A stereoview of the herringbone-like molecular packing of the title compound.

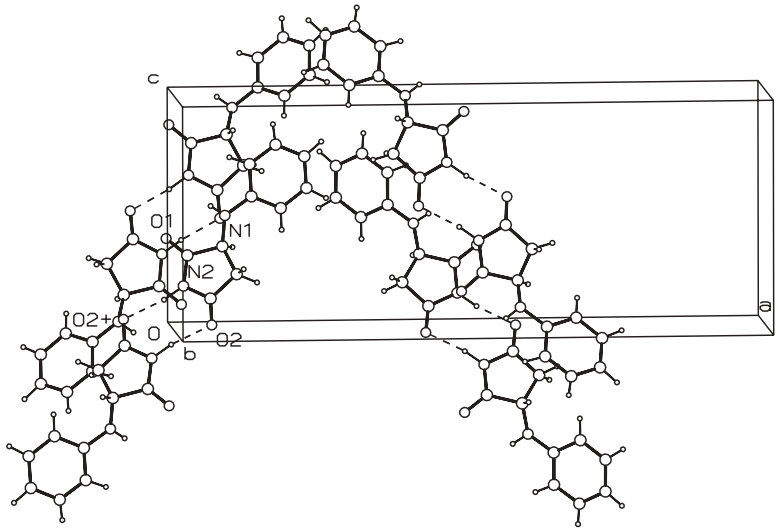


Figure 3. The unit cell of PHAMPD[19] with eight molecules forming four dimeric pairs around the crystallographic centres of inversion. The original atomic labelling is retained.

Remarkably, no more dimer formation has been observed so far. Only the crystal packing of 3-phenylpyrrolidine-2,5-dione (PHPYRO)[20] with an infinite chain [its graph[21,22] is $C_1^1(4)$] of the NH ··· O hydrogen bonds (Figure 4) suggests dimerization ability. In the hydrogen bonded molecular helices formed around the 2_1 screw axes (space group $Pna2_1$), the acceptor oxygen is O^2 adjacent to the phenyl ring and this pattern – in accordance with its low density of 1.28 Mg m^{-3} – may give rise to a dimeric form *via* phase transition. Indeed, a second polymorph with monoclinic (space group $P2_1/c$) crys-

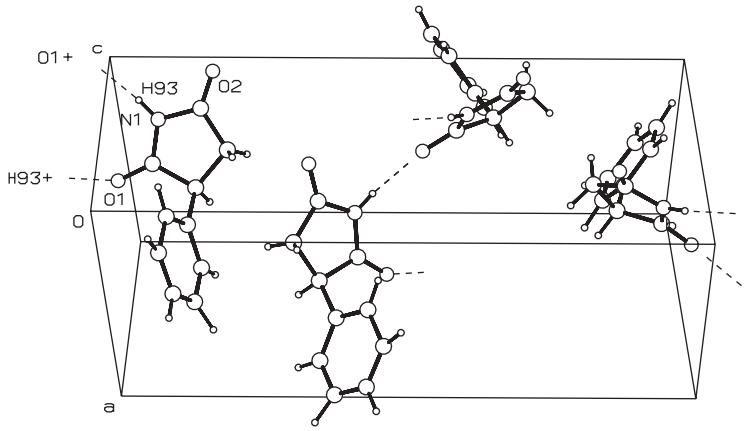


Figure 4. The unit cell of PHPYRO[20] showing fragments of infinite hydrogen bonded molecular helices along the *c* axis with original atomic labelling.

tals of low quality (for X-ray diffraction) was also observed earlier.[20] Further functions on the phenyl ring (R' = *m*-NO_2, TANPUT,[23] R' = *m*-Br and R'' = *p*-OC_2H_5, BEPSCM[24]) invariably stabilize infinite molecular helices (Figures 5 and 6) linked by NH ··· O bonds in which, with the same $C_1^1(4)$ graphs, O^5 oxygens are the acceptor again. In the asymmetric unit of TANPUT, there are two molecules linked alternatively by two independent NH ··· O bonds. The parameters in Table III point to some difference in their strength. Nevertheless, they jointly provide the highest density of 1.52 Mg m^{-3} among the molecules lacking any heavy atom substituent (*e.g.* Cl, Br, .. like BEPSCM). Further on, when the »secondary amine« bridge in PHAMPD[19] (Figure 3) was enlarged by a CH_2 group, then the novel compound 3-(*N*-benzyl)-aminopyrrolidine-2,5-dione could be crystallized only as monohydrate (BZAPYR[25]). In this binary system, each water is surrounded by four host molecules forming two strong and one weak and one very weak hydrogen bond with them (Figure 7). In the very weak OH ··· N contact (H ··· A = 2.65 Å) water donates a proton to pπ-pπ electrons delocalized on the O=C–NH–C=O

moiety. Since there is no direct host-host contact in this network, graph comparison can be regarded as irrelevant.

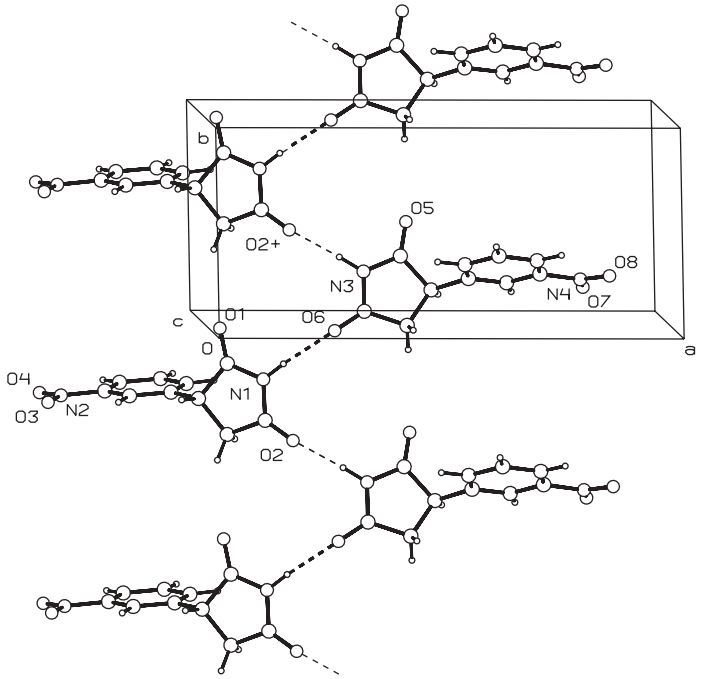


Figure 5. The unit cell of TANPUT[23] showing an infinite hydrogen bonded molecular helix along the *b* axis. The original atomic labelling is retained.

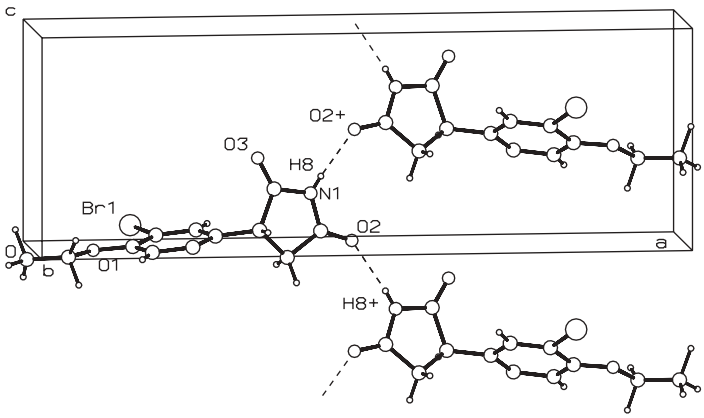


Figure 6. The unit cell of BEPSCM[24] showing a hydrogen bonded molecular helix along the *c* axis. The original atomic labelling is retained.

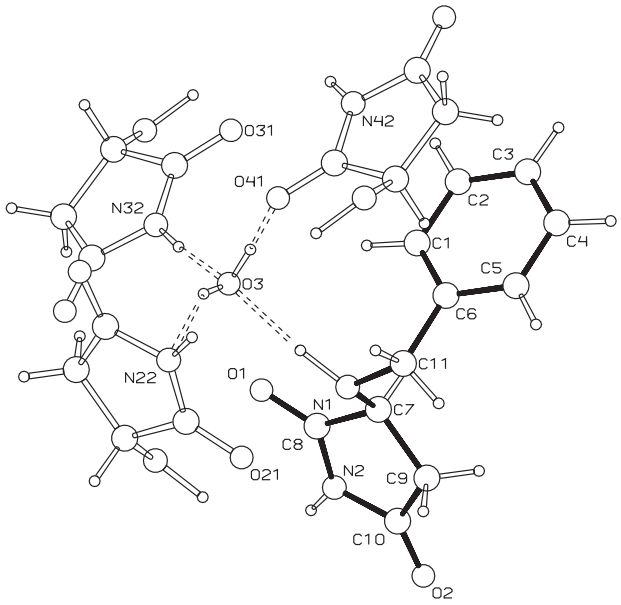


Figure 7. Coordination of a water molecule to four host molecules in BZAPYR.[25] The original atomic labelling is retained.

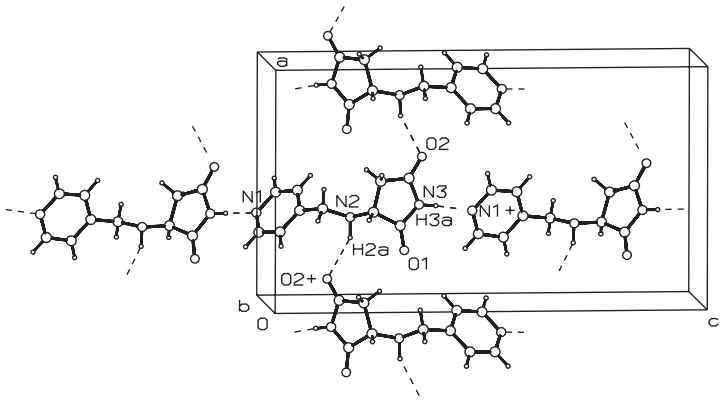


Figure 8. The two perpendicular, hydrogen bonded molecular ribbons in the unit cell of the title compound.

The title compound is obtained when the phenyl ring in BZAPYR is replaced by a pyridine moiety. Here, as it was shown above (Figure 2), both ring- and bridging-NH groups are donor again but in a different role (Figure 8). First of all, an N–H $\cdots$ N chain is formed along the *c* axis, which can be attributed to a novel acceptor N1 from the pyridine ring. Since the succinimide NH group is embedded in this chain, the secondary amine group of

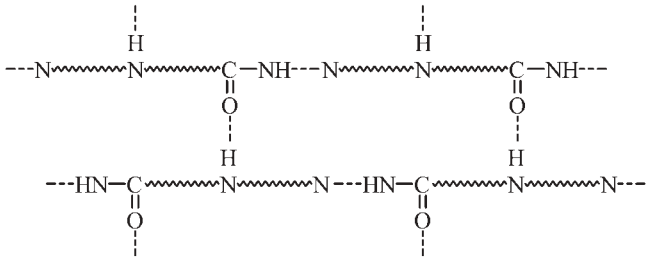


Figure 9. The hydrogen bond scheme of the molecular ribbons in the title compound.

the bridge donates a hydrogen bond to O^5 oxygen. This H-bond layout (Figure 9) can be characterized by the graphs: $C_1^1(10)$ for the NH···N chain, $C_1^1(6)$ for the perpendicular NH···O chain, and $R_4^4(27)$ for a 27-membered ring closed by the chains previously described. The strength difference between these hydrogen bonds was estimated by the use of a donor and acceptor scale suggested by Abraham *et al.*[26] The sum of proton donor (log $K\alpha$) and acceptor (log $K\beta$) scale values taken from Table IV in Ref. 26 is substantially greater (3.6) for the NH···N bond than that of 1.4 for the visibly weaker (Table III) NH···O bond. Jointly, they give rise to a density of 1.38 Mg m^{-3} similar to that of PHAMPD. A rather complicated hydrogen bond network is formed, when *mutatis mutandis* the spacer bridge (–NH–CH_2) of the title compound is enlarged (–HN–CO–CH_2–CH_2–) and the terminal pyridine ring is replaced by a polyfunctional »adenine« group. As shown by Figure 10 in CUBRUM,[27] there is an infinite $N18^{\#}$–$H18^{\#}$···$N1^{\#}$ hydrogen bond chain, strengthened by a parallel O···HN chain established between the primary amine group ($N6^{\#}$) of adenine and $O19^{\#}$ from the succinimide ring. Both chains can be described with a $C_1^1(13)$ graph. Of course, they close a ring described by graph $R_2^2(7)$. The adenine groups are crosslinked by a second strong NH···N bond formed with the second proton from the primary amine group to one of the ring nitrogens ($N3^{\#}$). This chain is also accompanied by a parallel strip of CH···N contacts with $N7^{\#}$ as acceptor. The parallel NH···N and CH···N chains of $C_1^1(6)$ type close a ring described by $R_2^2(8)$ graph. These twinned and approximately perpendicular hydrogen bond strips define a two-dimensional pattern depicted in Figure 11**,** to which several additional graphs could be assigned. Here, to avoid redundancy, only graphs formed by the NH···N bonds are given: $R_4^4(34)$ for the greater, and $R_4^4(30)$ for the smaller ring. Similarity between CUBRUM and the title compound is shown by the NH···O hydrogen bond helices formed between the

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[#] For structures retrieved from CSD the original atomic labelling is retained and applied.

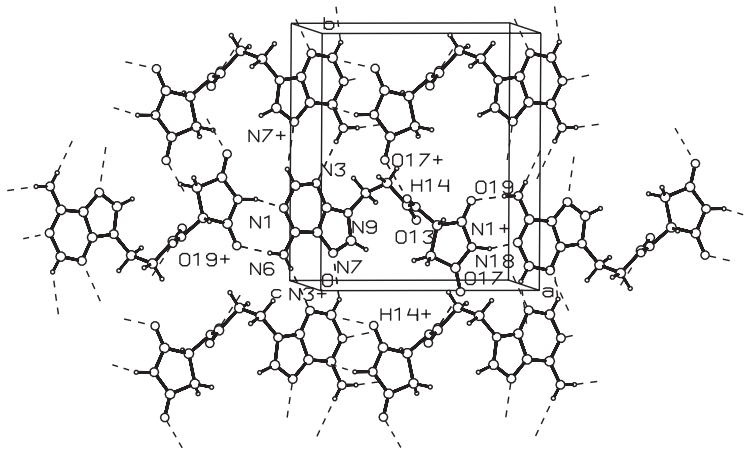

Figure 10. The unit cell of CUBRUM[27] showing hydrogen bonds with the original atomic labelling. (From left to right: N18–H18 ··· N1 and N6–H61 ··· O19, from up to down: N6–H62 ··· N3, C2–H2 ··· N7 and N14–H14 ··· O17). In this pattern, both succinimide oxo groups are acceptor.

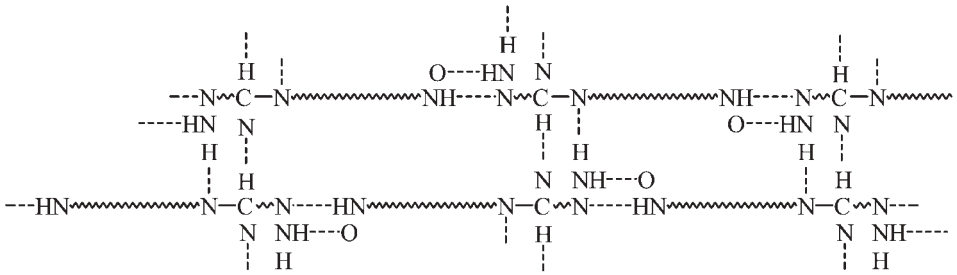

Figure 11. The hydrogen bond scheme of molecular ribbons in CUBRUM[27] for the principal N18–H18 ··· N1 and perpendicular N6–H62 ··· N3 bonds. The accompanying NH ··· O and CH ··· N hydrogen bonds are also shown.

bridging secondary amine group N14$^{\#}$ and O17$^{\#}$ of the succinimide ring. This helix of $C_1^1(6)$ type is perpendicular to the main N18$^{\#}$–H18$^{\#}$ ··· N1$^{\#}$ chain (*vide supra*) formed by the stronger donor and the stronger acceptor[26] and this resulting pattern (Figure 12) can be characterized by graphs $R_4^4(38)$ for the greater and $R_4^4(30)$ for the smaller rings.

In the homochiral (*S*)-(–)-2-((4-bromo-2-fluorophenyl)methyl)spiro(isochinoline-4(1H),3'-pyrrolidine)-1,2',3,5'(2H)-tetrone (HEMVEA)[8] and in its isostructural[28] 6-fluoro derivative (HEMVIE),[8] the bulky substituent, closed on C3$^{\#}$ forming a spirocarbon, substantially simplifies the hydrogen bond network (Figure 13). The NH group donates an NH ··· O hydrogen bond to

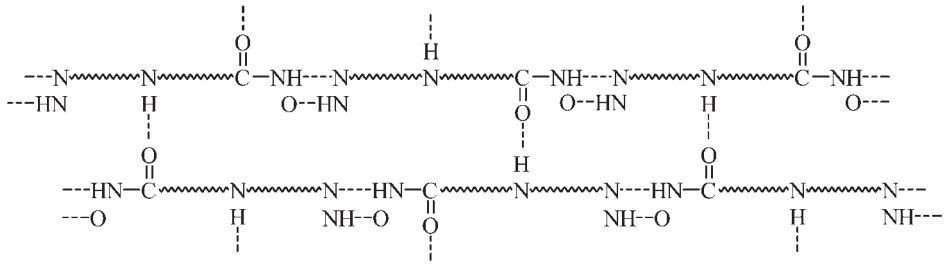

Figure 12. The hydrogen bond scheme of molecular ribbons in CUBRUM[27] for the principal N18–H18 ··· N1 and perpendicular N14–14 ··· O17 bonds. The accompanying NH ··· O hydrogen bond is also shown.

one of the oxo groups of the isochinoline ring forming an infinite helical $C_1^1(8)$ chain parallel with the *b* axis, while the succinimide oxo groups in both crystals are hindered in forming H-bonds. In addition, in these crystal lattices bounded together by isostructurality ($\Pi = 0.003$),[28] the entering fluorine atom substantially weakens the NH ··· O hydrogen bond helix of HEMVIE (Table III). Similarly in REWWOF,[9] the OH groups sitting on the bulky

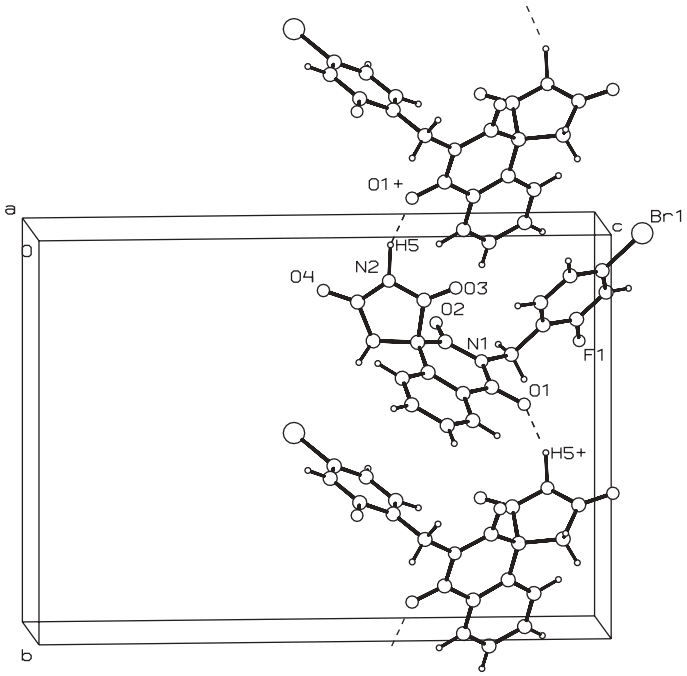

Figure 13. The unit cell (space group $P2_12_12_1$) of HEMVEA[8] showing one infinite hydrogen bonded helix of the chiral molecules with original atomic labelling.

substituent (based on a saturated naphtalene skeleton) also prevent the succinimide oxo groups from participating in hydrogen bonds. This steric effect, even in the presence of two clorine atoms, seems to account for the low density of 1.30 Mg m^{-3}. The H-bond pattern developed can be described by two graphs: $C_1^1(6)$ for NH $\cdots$ O and $C_1^1(8)$ for OH $\cdots$ O bonds forming infinite helices around the screw axes in space groups $P2_12_12_1$.

To summarize, while the participation of the succinimide oxo groups in hydrogen bonding (**2**: CUBRUM, **1**: BEPSCM, BZAPYR, PHAMDP, PHPYRO, SUCCIN, TANPUT and the title compound or **0**: HEMVEA, HEMVIE and REWWOF) depends on the size of the substituents and their hydrogen-bond-active functions, the NH group is the principal component of each hydrogen bond network discussed.

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SAŽETAK

O vodikovoj vezi u derivatima sukcinimida. Kristalna struktura 3(4-piridil-metil)amino-pirolidin-2,5-diona

Gyula Argay, László Fábián i Alajos Kálmán

Struktura 3(4-piridil-metil)amino-pirolidin-2,5-diona određena je rentgenskom strukturnom analizom utočnjavanjem difraktometrijskih podataka s obzirom na kvadrat strukturne amplitude do faktora nepouzdanosti $R = 0{,}0526$ za 1999 opaženih refleksa. Gusto pakiranje u rompskim kristalima [prostorna grupa *Pbca* (No. 61); $a = 11{,}595(4)$, $b = 8{,}325(1)$, $c = 20{,}452(4)$ Å, $V = 1974{,}2(8)$ Å^3, $Z = 8$, $D_c = 1{,}381$ Mg/m^3, $\mu = 0.824$ mm^{-1}] ostvareno je slaganjem slojeva previnutih molekula, složenih unutar sloja po uzorku »riblje kosti«, uzduž osi *c* te stopljenih beskonačnim lancima vodikovih veza N–H ··· O i N–H ··· N koje su međusobno okomite jedna na drugu. Takav način međumolekulskog povezivanja ponukao nas je na pripravu kritičkog pregleda i potankog opisa mreža vodikovih veza opaženih kod sukcinimida s nesupstituiranim >NH skupinama korištenjem baze podataka Cambridge Structural Database (CSD), a također i naših ranijih vlastitih strukturnih određivanja. Uobičajeni motivi sustava vodikovih veza analizirani su grafički na način kako je to predložila M. C. Etter. U članku se također raspravlja o utjecaju vodikovih veza na gusto pakiranje molekula.