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Solid state structural analysis of new pentamidine analogs designed as chemotherapeutics that target DNA by X-ray diffraction and ^{13}C, ^{15}N CP/MAS NMR methods

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ABSTRACT

The paper presents the solid-state analysis of the crystalline form of 1,5-*bis*[(4-cyanophenyl)-*N*-methylamino]pentane (**1**) and polycrystalline powder sample of 1,5-*bis*[(4-amidinophenyl)-*N*-methylamino]pentane dihydrochloride (**2**). The methods used are X-ray diffraction technique and ^{13}C, ^{15}N CP/MAS NMR spectroscopy in an attempt to detect the effects of possible polymorphism. Both methods indicate that only single conformers exist in the solid-state for **1** and **2**. 1,5-*Bis*[(4-cyanophenyl)-*N*-methylamino]pentane **1**, crystallizes in the orthorhombic space group $P2_12_12$. The asymmetric unit contains one half of the ordered molecule. Only weak intermolecular interactions were found in solid-state, in which methyl groups are engaged.

1. Introduction

Pneumocystis is an opportunistic fungus that is the cause of lethal pneumonia (PCP) in immunocompromised persons [1,2]. Pentamidine isethionate is a drug used clinically against PCP, and also against leishmaniasis and trypanosomiasis but a number of toxic side effects have been observed during treatment including abscesses at the injection site, nephrotoxicity, cardiotoxicity and hypoglycemia [3,4]. Chemically pentamidine belongs to diamidines that have a broad range of activities against eukaryotic parasites, bacteria, viruses and tumors. Diamidines have been found to bind specifically to adenine–thymine minor groove sequences of DNA. There are strong evidences that DNA binding is involved in the mechanism of action of these compounds [5–9]. Among *bis*-nitriles, which are intermediates while preparing *bis*-amidines, many investigators have also found active substances against tumor [10,11]. Given this knowledge, *bis*-amidines and *bis*-nitriles represent the promising templates for design and development of new drugs.

The solid-state analysis of biologically active substances is of special interest [12,13] and the CP/MAS NMR spectroscopy in the solid state or XRD technique are the most frequently methods used in those investigations [12,13]. In the paper we report the syntheses (Scheme 1) and structural features in solid-state of two new compounds: 1,5-*bis*[(4-cyanophenyl)-*N*-methylamino]pentane (**1**), and 1,5-*bis*[(4-amidinophenyl)-*N*-methylamino]pentane dihydrochloride (**2**) (*N*-methylamino analog of pentamidine). In structural analysis we have applied X-ray diffraction technique, together with ^{13}C and ^{15}N CP/MAS NMR spectroscopy combined with molecular modeling.

2. Experimental

2.1. Syntheses

All chemicals were obtained from major chemicals suppliers as high or highest purity and were used without further purification. Melting points were determined with an Electrothermal 9001 Digital Melting Point Apparatus and are uncorrected. Elemental analyses were performed on C, H, N, S Elementar GmbH Vario EL III analyzer. IR spectra were recorded on Schimadzu FTIR-8300 in KBr tablets. Notation used in NMR assignments is given in Fig. 1.

2.1.1. 1,5-Bis[(4-cyanophenyl)-N-methylamino]pentane (*1*)

1,5-Dibromopentane (1.15 g, 0.005 mol), *N*-methyl-4-aminobenzonitrile (1.32 g, 0.01 mol), sodium hydroxide (0.8 g, 0.02 mol) and *tetr*-butylammonium bromide (0.32 g, 0.001 mol) in toluene (20 ml) were refluxed together for 3 h while stirring. Then, the hot mixture was poured into ice water to obtain a white precipitate that was recrystallized from ethanol with hot filtering to give 0.9 g (54%) of white crystals of **1**. M.p. 141–142.5 °C. —$C_{21}H_{24}N_4$ (332.45) calcd C 75.87, H 7.28, N 16.85, found C 75.82, H 6.95, N 16.52. —^1H NMR (400.13 MHz, $CDCl_3$): 1.33–1.37 (m, 2H, H-11), 1.63

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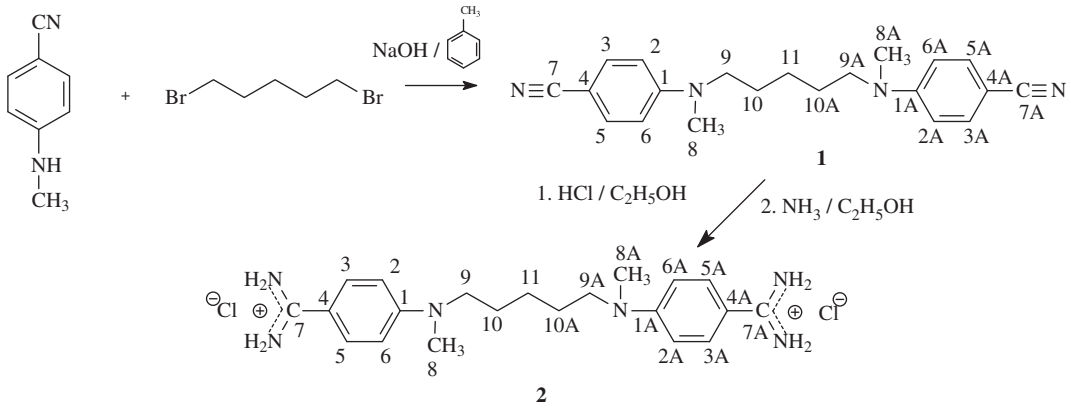

Scheme 1. Details of synthetic procedures of **1** and **2** with atom numbering.

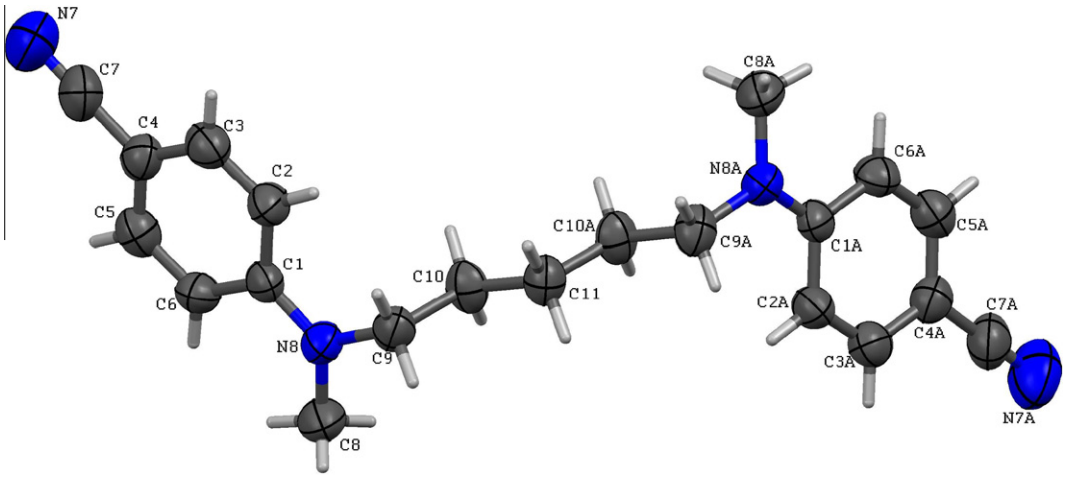

Fig. 1. A perspective view of the molecular conformation of **1**.

(q, J = 7.6 Hz, 4H, H-10, H-10A), 2.98 (s, 6H, H-8, H-8A), 3.37 (t, J = 7.6 Hz, 4H, H-9, H-9A), 6.60–6.62 (m, 4H, H-2, H-6, H-2A, H-6A), 7.43–7.45 (m, 4H, H-3, H-5, H-3A, H-5A) ppm. —^{13}C NMR (100.61 MHz, $CDCl_3$): 24.67 (C-11), 26.90 (C-10, C-10A), 38.54 (C-8, C-8A), 52.29 (C-9, C-9A), 97.26 (C-4, C-4A), 111.36 (C-2, C-6, C-2A,C-6A), 120.82 (C-7, C-7A), 133.67 (C-3, C-5, C-3A, C-5A), 151.58 (C-1, C-1A) ppm. -IR: 3100–3090 (ν_{CHarom}), 2950 (ν_{CH3as}), 2920 (ν_{CH2as}), 2862 (ν_{CH3sym}; ν_{CH2sym}), 2206 ($\nu_{C\equiv N}$), 1589,1512 ($\nu_{C=C}$), 1458 (δ_{CHalif}), 1373 (ν_{CN}), 1257 (ν_{COas}), 1003 (ν_{COsym}), 818 (γ_{HC}) cm^{-1}.

2.1.2. 1,5-Bis[(4-amidinophenyl)N-methylamino]pentane dihydrochloride (*2*)

1,5-*Bis*[(4-cyanophenyl)-*N*-methylamino]pentane (**1**) (0.66 g, 0.002 mol) and ethanol (25 ml) saturated with dry hydrochloride were stirred in a round-bottomed flask at room temperature for 20 h, then dry diethyl ether (60 ml) was added. The precipitate was washed with ether and dried in vacuum under $CaCl_2$ for 4 h. Crude iminoester was stirred together with ethanol (25 ml) saturated with dry ammonia at room temperature for 48 h, then the solvent was evaporated off and the remaining solid was washed with aqueous sodium hydroxide and water, dried, and converted into dihydrochloride by solution of hydrochloride in ethanol. 0.76 g (74%) of white solid of **2** was obtained. M.p. 315.0–318.0 °C (decomp). $C_{21}H_{30}N_6 \times 2HCl \times 4H_2O$ (511.51) calcd. C 49.31, H 7.88, N 16.43, found C 49.67, H 7.87, N 16.07. —^{1}H NMR (400.13 MHz, DMSO-d_6): 1.32 (m, 2H, H-11), 1.52–1.57 (m, 4H, H-10, H-10A), 2.99 (s, 6H, H-8, H-8A), 3.43 (t, J = 7.2 Hz, 4H, H-9, H-9A), 6.77–6.80 (m, 4H, H-2, H-6, H-2A, H-6A), 7.73–7.76 (m, 4H, H-3, H-5, H-3A, H-5A), 8.63 (s broad, 4H, 2 × 2NH), 8.91 (s broad, 4H, 2 × 2NH) ppm. —^{13}C NMR (100.61 MHz, DMSO-d_6): 23.66 (C-11), 26.14 (C-10, C-10A), 38.06 (C-8, C-8A), 51.24 (C-9, C-9A), 110.82 (C-2, C-6, C-2A, C-6A), 111.74 (C-4, C-4A), 129.67 (C-3, C-5, C-3A, C-5A), 152.78 (C-1, C-1A), 164.10 (C-7, C-7A) ppm. -IR: 3550–2780 (ν_{NH}; ν_{CHarom}; ν_{CH3}; ν_{CH2}; ν_{CH2}), 1682 ($\nu_{=NH}$), 1612 ($\nu_{C=C}$), 1570 (δ_{NH}), 1510–1420 ($\nu_{C=C}$; δ_{CH2}), 1396 (ν_{CN}), 852 (γ_{HC}) cm^{-1}.

2.2. Crystallography

Crystals of 1 suitable for X-ray analysis were grown by slow evaporation from ethanol solution. Diffraction data were collected on an Oxford Diffraction KM4CCD diffractometer [14] at 293 K, using graphite-monochromated Mo Kα radiation. The unit cell parameters were determined by least-squares treatment of setting angles of highest-intensity reflections chosen from the whole experiment. Intensity data were corrected for the Lorentz and polarization effects [15]. The structure was solved by direct methods by use of the SHELXS97 program [16] and refined by the full-matrix least-squares method with the SHELXL97 program [17]. One reflection was excluded from the reflection file due to its large ($|F_o|^2 - |F_c|^2$) difference. The function $\Sigma w(|F_o|^2 - |F_c|^2)^2$ was minimized with $w^{-1} = [\sigma^2(F_o)^2 + (0.0327P)^2]$, where $P = (F_o^2 + 2F_c^2)/3$. All non-hydrogen atoms were refined with anisotropic thermal

Table 1
Crystal data, data collection and structure refinement for **1**.

Compound	**(1)**
Empirical formula	$C_{21}H_{24}N_4$
Formula weight	332.44
T (K)	293(2)
Wavelength (Å)	0.71073
Crystal system, space group	Orthorhombic, $P2_12_12$
Unit cell dimensions	
a (Å)	10.624(1)
b (Å)	19.855(3)
c (Å)	4.4848(7)
Volume (Å^3)	946.0(2)
Z, D_x (Mg/m^3)	2, 1.167
μ (mm^{-1})	0.071
F(0 0 0)	356
θ range for data collection (°)	4.11–25.68
hkl range	$-12 \leqslant h \leqslant 12$ $-24 \leqslant k \leqslant 14$ $-4 \leqslant l \leqslant 5$
Reflections	
Collected	3637
Unique (R_{int})	1737 (0.06)
Observed ($I > 2\ \sigma(I)$)	573
Data/restraints/parameters	1737/0/118
Absorption correction	Multi-scan
Goodness-of-fit on F^2	0.786
$R(F)$ ($I > 2\ \sigma(I)$)	0.0384
$wR(F^2)$ (all data)	0.0892
Max/min. $\Delta\rho$ (e/Å^3)	0.134/−0.127

Table 2
Selected bond lengths (Å) and angles (°) and selected torsional angles (°) for **1**.

C1—N8	1.357(3)
N8—C9	1.453(3)
C9—C10	1.531(3)
C7—N7	1.149(4)
C1—N8—C9	121.8(3)
C10—C11—C10A	110.7(4)
C1—N8—C9—C10	−83.1(4)
N8—C9—C10—C11	−175.0(2)
C9—C10—C11—C10A	180.0(3)

Table 3
Hydrogen bonding geometry (Å and °) for compound **1**.

D—H···A	d(D—H)	d(H···A)	d(D···A)	<(DHA)
C8—H8C···N7^{i}	0.96	2.64	3.602(4)	177

Symmetry code: (i) 1.5−x, 0.5 + y, −z.

parameters. The coordinates of the hydrogen atoms were calculated in idealized positions and refined as a riding model with their thermal parameters calculated as 1.2 (1.5 for methyl group) times U_{eq} of the respective carrier carbon atom. An empirical extinction correction was also applied according to the formula $F_c' = kF_c[1 + (0.001\chi F_c^2\lambda^3/sin2\theta)]^{-1/4}$ [17], and the extinction coefficient χ was equal to 0.0025(3). The deposition number CCDC 791244 for **1** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk.

2.3. NMR spectra and molecular modeling

The ^{1}H NMR and ^{13}C NMR 1D and 2D spectra in solution were recorded with a Bruker Avance DMX 400, as well as the solid state ^{13}C and ^{15}N CP/MAS NMR spectra. Typical acquisition conditions for ^{13}C CP/MAS NMR at 100.62 MHz were: pulse duration, 4.5 μs;

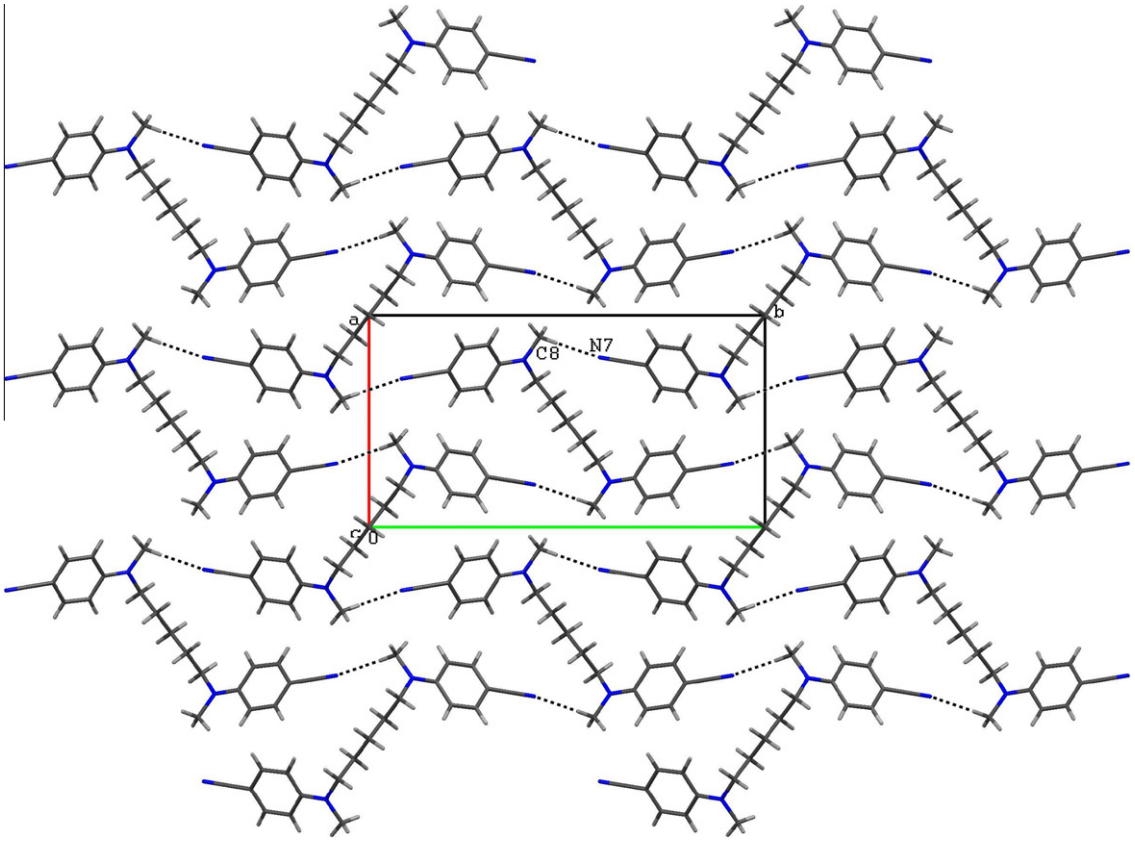

Fig. 2. Interconnections within a layer of **1**.

contact time, 2 ms; repetition time, 10 s; spectral width, 24 kHz; number of transients, 1000. Spinning speeds ranged from 8 to 10 kHz. Nonprotonated carbons and methyl groups were selectively observed by dipolar-dephasing experiment with delay time 50 μs. Chemical shifts δ (ppm) for ^{13}C were references to TMS. Spectral conditions for ^{15}N CP/MAS NMR at 40.55 MHz and high-power proton decoupling field were: spectral width, 55 kHz; pulse duration, 3.8 μs; contact time, 5 ms; repetition time, 10 s; number of transients, 6800. Chemical shifts for ^{15}N were calibrated indirectly on glycine resonance (−345 ppm), and referenced to CH_3NO_2 signal (see Fig. 4).

Crystallographic atom coordinates for **1** and the optimized ones for **1** and **2** were used for computation of shielding constants σ (ppm) of ^{13}C atoms as the help in assignment of resonances in the solid-state NMR spectra. We employed the DFT method with B3LYP/6-311(d,p) hybrid functional for structure optimization, and the CPHF–GIAO approach for the NMR shielding constants computations using Gaussian 03 program [18].

3. Results and discussion

3.1. X-ray structure of 1,5-bis[(4-cyanophenyl)-N-methylamino]-pentane (*1*)

The crystal and molecular structures of **1** were analyzed by X-ray diffraction. All details of the measurement, crystal data and structure refinement are given in Table 1. Selected bond lengths, bond angles and torsion angles are listed in Table 2, and hydrogen bonding parameters in Table 3. A perspective view of the molecular conformation, together with the atom numbering scheme, is shown in Fig. 1 (the drawings were performed with Mercury program [19]).

1,5-*Bis*[(4-cyanophenyl)-*N*-methylamino]pentane (**1**), new analog of pentamidine synthesized by us, crystallizes in the orthorhombic space group $P2_12_12$. The central C11 atom is located at a twofold crystallographic axis (according to symmetry $1-x$, $1-y$, z) and the asymmetric unit contains one half of the ordered molecule. At the angle between the best planes of the two planar aromatic rings being 73.52(7)°, the nitrile groups and methyl-carbons are nearly coplanar with these rings. The conformation of the central linkage is *trans* for C9—C10—C11—C10A and N8—C9—C10—C11 but *gauche* for C1—N8—C9—C10 (the appropriate torsion angles are listed in Table 2), and the distance from C1 to C1A atom is equal to 8.85 Å. This symmetry is very similar to that described in our earlier paper for 4,4′-[1,5-(3-oxapentanediylbis(amino))]-bisbenzonitrile [15]. The crystal structure of **1** is stabilized by an intermolecular C—H···N hydrogen bond between the methyl and nitrile groups (see Table 3). The molecules are linked by C8—H8C···N7 interactions forming folded layers parallel to the (0 0 1) plane (Figs. 2 and 3). Such participation of the nitrile substituents in the hydrogen bonds were also found for other analogs of pentamidine [20]. They could be responsible for the efficient interaction with the biological target.

3.1.1. ^{13}C and ^{15}N CP/MAS NMR spectra of *1* and *2*

The values of chemical shifts from ^{13}C and ^{15}N CP/MAS NMR spectra together with shielding constants obtained from DFT computations provided complementary information on the solid-state structure of **1** and **2**. We could expect single, twofold or even fourfold resonances for each carbon, dependent on the symmetry and/or the number of conformations present. The ^{13}C CP/MAS NMR spectra of **1** and **2** are shown in Fig. 4. In the spectrum of **1** multiplets are not observed, meaning that only a single structure is detected in solid-state, which is consistent with the single-crystal structure described in Section 3.1. Small high frequency shifts of C7 (C7A) and C8 (C8A) atoms (as compared with the solution resonances) can be correlated with the existence of weak intermolecular C—H···N hydrogen bonds. DFT computations of the isotropic shielding constants σ (ppm) using B3LYP/6-311G (d,p) hybrid functional applied to the crystallographic and optimized coordinates of compound **1** were related to the experimental chemical shifts δ (ppm). We observed two linear (almost parallel) correlations (correlation coefficients r^2 were close to 0.98) for both functions $\sigma_{cryst} = f(\delta)$ and $\sigma_{optimized} = f(\delta)$. This finding could be proof that these simple computations give quite satisfactory results and can be used in the powder sample analysis of compound **2**, for which a single-crystal form was not obtained. In the NMR spectrum of **2** the single pattern of resonances was also observed, and only one structure for compound **2** was detected. In theoretical analysis we have considered two conformations of **2** shown in Fig. 5: first in which NCH_3 groups have the alignment as in the crystal structure of **1** *i.e.* they are pointed out in opposite directions, and the second in which NCH_3 groups are oriented in the same direction. DFT calculation showed small energy differences of the conformers (about 5 kJ/mol), but the experimental resonances δ are consistent only with the calculated shielding constants σ obtained for the former conformation. The experimental δ values in solid-state are very close to the solution ones, indicating that only a very weak hydrogen bond could exist in solid-state. The conformation of the aliphatic linker of *bis*-amidine **2** is much more extended than this of *bis*-nitrile **1**, and the distance from C1 to C1′ is equal to 9.42 Å. The molecule of **2** is flattened as compared to **1**, and the angle between two planar aromatic rings is equal to 134.0°. The resonances of each pair of *ortho* carbons to NCH_3 groups are inequivalent (108.9 for C2, C2A and 112.2 ppm for C6, C6A). The assignment is based on the dipolar diphased spectra revealing quaternary atom resonances. The observed splitting between C2 and C6 signals are mainly due to the shielding effects of the nitrogen lone pairs and pointed out the lack of phenyl ring rotation. Amidine C7, C7A atoms give one broad resonance at 164.0 ppm, proving an equivalency of these groups.

It was thought that natural abundance ^{15}N CP/MAS NMR spectroscopy would be a useful probe in complementary structural

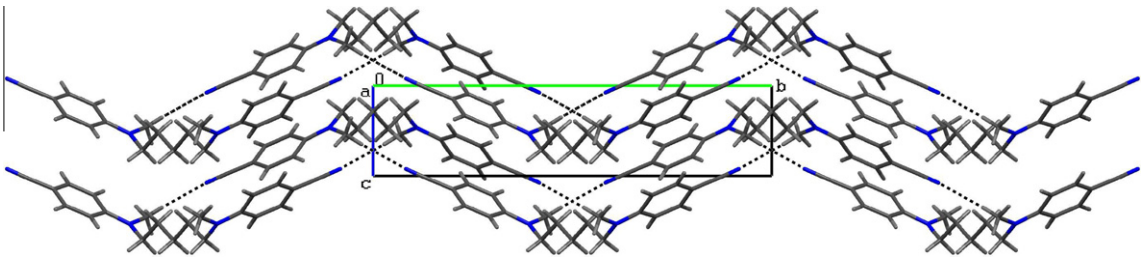

Fig. 3. Packing arrangement of **1** indicating folded layers parallel to (0 0 1) plane.

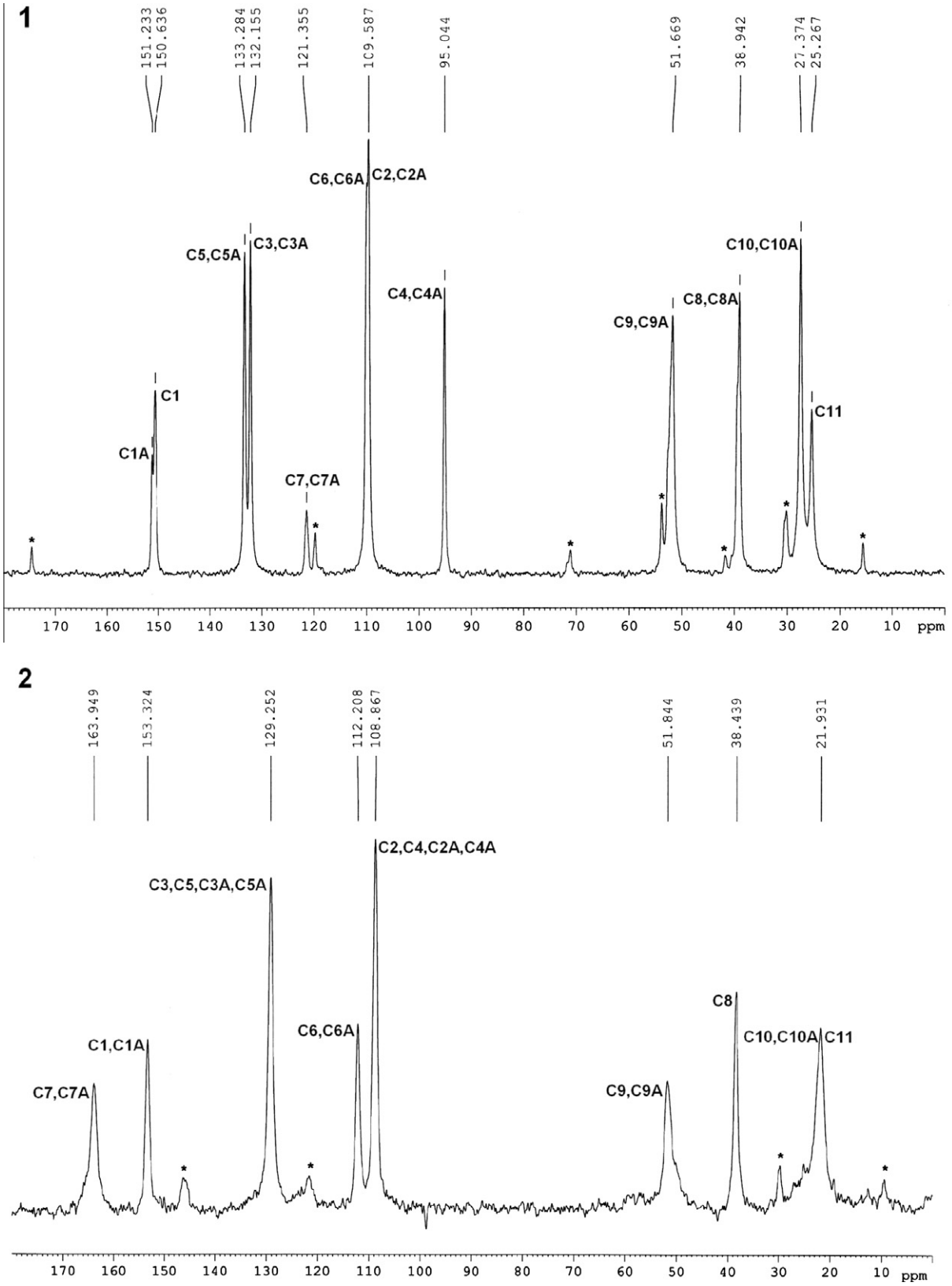

Fig. 4. ^{13}C CP/MAS spectra of **1** and **2**. Sidebands are marked with an asterisk.

analysis of **1** and **2**, because it possesses a large chemical shift range resulting in good dispersion of signals. The ^{15}N NMR spectra in solid state of **1** and **2** are shown in Fig. 6. In the spectrum of **1** sharp signals are observed for C≡N and NCH_3 groups at −125.3 and −301.0 ppm, respectively, indicating absence of any exchange broadening. The spectrum of **2** is not so good, but we can clearly identify the NCH_3 signal at −298.5 ppm, and doublet of amidine nitrogens at 278.3 and −281.5 ppm. The two signals arise from

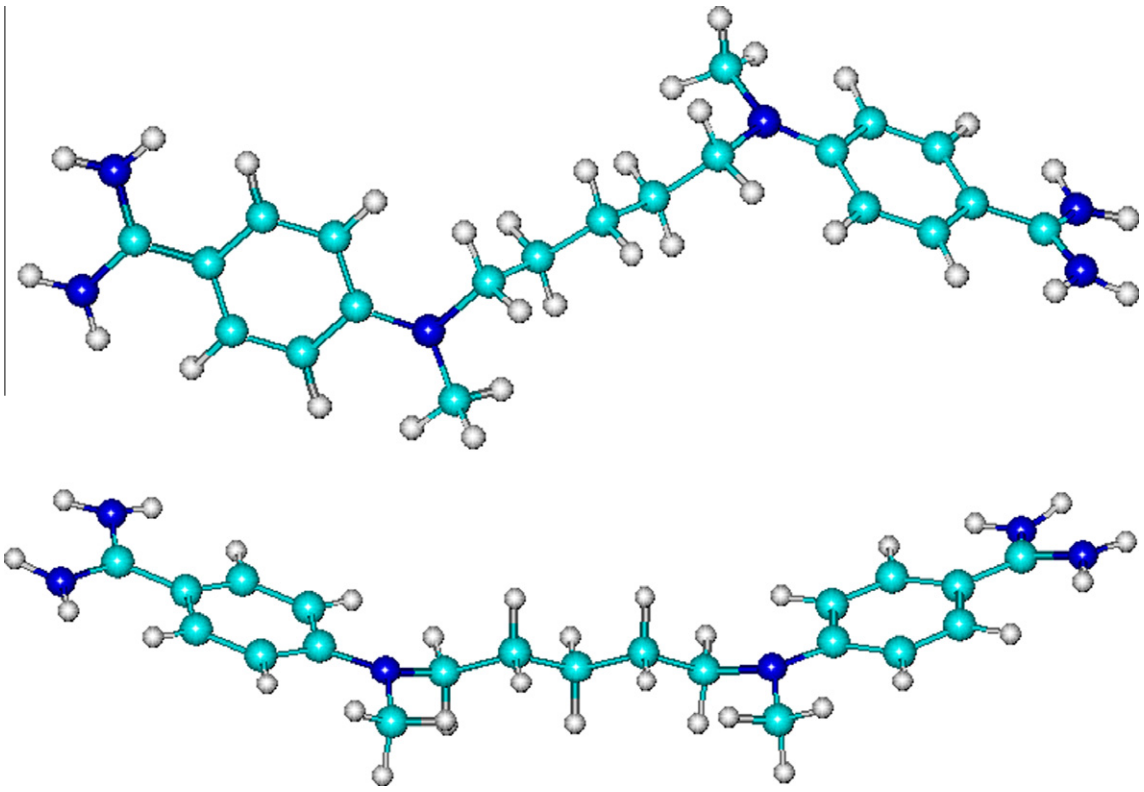

Fig. 5. Two analyzed conformations of **2** in solid-state: upper – NCH_3 groups oriented in opposite directions; bottom – NCH_3 groups oriented in the same direction.

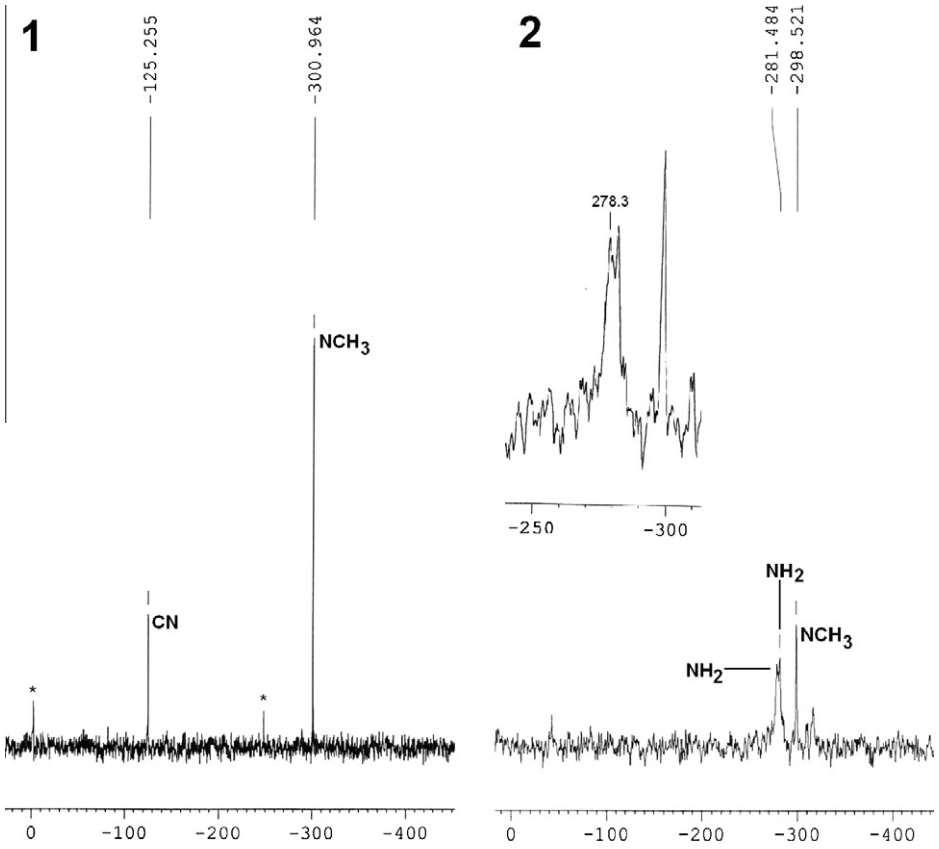

Fig. 6. ^{15}N CP/MAS spectra of **1** and **2**.

the presence of two the NH_2 groups in each amidine substituent, which differ due to slight deviation of amidine substituent from the benzene ring plane. This statement is in good agreement with the theoretical conformation showed in the top of Fig. 5.

4. Conclusions

New 1,5-*bis*[(4-cyanophenyl)-*N*-methylamino]pentane (**1**) and 1,5-*bis*[(4-amidinophenyl)-*N*-methylamino]pentane dihydrochloride (**2**) have been characterized in solid-state, showing lack of polymorphism. Single-crystal X-ray analysis showed that **1** crystallizes in the orthorhombic space group $P2_12_12$ and the asymmetric unit contains one half of the ordered molecule. The crystal structure of **1** is stabilized by intermolecular C—H···N hydrogen bond between the methyl and nitrile groups. Conformational analysis of **2** using NMR solid-state spectroscopy combined with DFT theoretical computations has confirmed that NCH_3 groups in aliphatic linker has the alignment as in the crystal structure of **1**, but its conformation is much more extended than that of *bis*-nitrile **1**, and the distance from C1 to C1′ is equal to 9.42 Å. In the present case, we can suppose that the intermolecular hydrogen bonds are very weak.

New compounds **1** and **2** were synthesized with good yields by an optimized procedure.

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