**p-N,N’-tetraacetylodiaminodurene. The structure and vibrational spectra.**

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**Abstract**

The crystal and molecular structure of p-N,N’-tetraacetylodiaminodurene (TADD) is reported based on the X-ray diffraction studies. The N-acetyl moieties are planar and all
N-acetyl groups are perpendicular to the ring plane. Methyl groups both of acetyl moieties and of durene form a number of non-conventional hydrogen bonds with nitrogen and oxygen atoms. The vibrational spectra very well reflect the structure of molecules and their contacts. They are compared with calculated data by using various theoretical approaches. The neutron scattering spectra show two tunnel lines of low energy values (at ±0.9 and ±2.3 eV at 4 K), which can be ascribed to methyl groups of N-acetyl moieties, which behave more freely than those attached to the phenyl ring.

**Introduction**

The motivation of understanding the studies on p-N,N’-tetraacetylodiaminodurene (TADD) was the paper by Beagley et al. devoted to the synthesis and X-ray diffraction structure of p-bis(N,N-diacetyloamino)benzene [[[2]](#endnote-1)]. The N-acetylo-derivatives are interesting as shown in a number of publications [[[3]](#endnote-2),[[4]](#endnote-3),[[5]](#endnote-4),[[6]](#endnote-5),[[7]](#endnote-6),[[8]](#endnote-7),[[9]](#endnote-8),[[10]](#endnote-9),[[11]](#endnote-10),[[12]](#endnote-11),[[13]](#endnote-12),[[14]](#endnote-13),[[15]](#endnote-14),[[16]](#endnote-15),[[17]](#endnote-16),[[18]](#endnote-17)2-17]. Among numerous applications a particular attention evoke the polyacetoanilines composites. Some attention also evokes the fact that *ortho* substituted N,N-diacetyanilines can be exploited as a selective acetylation reagent [[[19]](#endnote-18)]. The papers devoted to the tetraacetyl derivatives and above all tetraacetylethylenediamine deserve also a particular attention [[[20]](#endnote-19),[[21]](#endnote-20),[[22]](#endnote-21)19-21].

On the other hand the insertion of four methyl groups to the benzene ring evokes some interest from the point of view of the durene property concerning a formation of the molecular complexes [[[23]](#endnote-22),[[24]](#endnote-23),[[25]](#endnote-24),[[26]](#endnote-25),[[27]](#endnote-26),[[28]](#endnote-27),[[29]](#endnote-28),[[30]](#endnote-29),[[31]](#endnote-30)22-30]. The present work on TADD expands our previous study on the methyl group dynamics in tetramethylpyrazine and durene [24,[[32]](#endnote-31),[[33]](#endnote-32),[[34]](#endnote-33),[[35]](#endnote-34) 31-34]. Up to now we were interested rather in the relationship between the methyl group rotational potential and a strength of hydrogen bond formed between the proton donor and proton acceptor in the solid state. We constructed the acid/base arrangements with an appropriate value of proton affinity in order to analyze the behavior of the CH3 group in different molecular complexes. To the best of our knowledge, the conventional hydrogen bond interactions play a decisive role in the crystal packing of the acid/base molecular complexes. Nevertheless the unconventional interactions between the C-H bonds and the proton acceptor atoms should be also taken into account.

Recently, we analyzed the cocrystals of tetramethylpyrazine (TMP) with chloranilic (CLA) and bromanilic (BRA) acid [[[36]](#endnote-35)]. At low temperatures (below ca. 50 K) four tunneling peaks were observed for TMP⋅CLA in the INS experiment, whereas no tunneling peaks were found for TMP⋅BRA in the analyzed energy range of ±30 eV.

The INS spectra as well as the crystal structure analysis for TMP⋅CLA indicated four independent methyl groups in the asymmetric unit of the crystal structure. In this crystal the alternating donor and acceptor molecules form chains *via* the conventional O-H⋅⋅⋅N hydrogen bonds. In the crystal lattice only two of four methyl groups are linked by the weak auxiliary C-H⋅⋅⋅O hydrogen bonds, whereas the remaining two groups exhibit only so called short contacts. It means that the number of the observed tunneling peaks depends on the number of the independent CH3 groups in the crystal structure. Both the C-H⋅⋅⋅O hydrogen bonds and short contacts affect the methyl groups rotational potential – each peak is observed at a different energy.

In contrast to TMP⋅CLA, the methyl groups in the TMP⋅BRA complex are stabilized by three C-H⋅⋅⋅O relatively strong hydrogen bonds and by additional three short contacts. These effects restrict the rotation of the CH3 groups and enlarge their potential barrier. Our systematic analysis confirmed that the rotation and deformation of methyl groups substantially depend on the environment. This environment decides about a barrier for methyl group rotation and the fact that no tunneling peaks are observed in the INS spectra.

In the TADD molecule four CH3 groups are connected with aromatic ring and the other four methyl groups are aliphatic. This crystal was found to be best suited for the studies on the unconventional C-H⋅⋅⋅X (X = O, N) interactions and the environment effect on potential barrier of the CH3 group.

Moreover in our opinion this molecule, as the electron donor, will be a good precursor for the molecular complexes created with the organic electron acceptors. We expect that in this case the value of the barrier height for the CH3 rotation will be a measure of both the hydrogen bond strength and/or π-electron interactions. The results presented in this paper are an initiation of forthcoming studies of novel complexes. Namely we present the data for the possible pure component of these new complexes.

In the present paper we would like to describe the X-ray diffraction structure of TADD at 100 K. The aim of the undertaken studies was a careful recognition of the crystal packing particularly from the viewpoint of the acetyl groups. The objects of our particular interest were methyl groups and a comparison of the contacts of the groups in acetyl moieties and those connected with the benzene ring. In vibrational spectra of TADD the subject of our interest was an analysis of the bending and torsional vibrations of methyl groups as well as the behavior of carbonyl groups. The results were analyzed by using the theoretical DFT calculations. For a better investigation of the dynamics of methyl groups we have undertaken the studies on inelastic neutron backscattering spectroscopy, which could bring essential information about the potential of rotating groups particularly at the lowest temperature.

**Experimental and calculations**

The synthesis of N,N’-tetraacetylodiaminodurene (TADD) is described elsewhere [1]. Crystals for the diffraction studies were grown from ethanol solution. The X-ray diffraction data were collected by using a KUMA KM4CCD -axis four circle diffractometer equipped with an Oxford Cryosystem Cooler using graphite monochromated MoK radiation. The structure was solved by direct methods with SHELXS-97 and refined by the full-matrix least-squares on all F2 data using SHELXL-97 programs [[[37]](#endnote-36)]. All non-hydrogen atoms were refined with anisotropic thermal parameters. The H atoms of the methyl groups were found from the different synthesis and refined with isotropic thermal parameters 1.5 times Ueq(C). The crystal data and structure refinement are presented in Table 1.

All Crystallographic data are deposited at the Cambridge Crystallographic Data Center (CCDC No 1044828).

**Table 1**

The infrared spectra over the frequency range 4000-400 cm-1 in Nujol and Fluorolube suspensions were recorded using a FTIR Brucker IFS 113v spectrophotometer. The Raman spectrum of powder sample was recorded on a Nicolet Magma 860 FT Raman spectrometer. A diode-pumped Nd: YAG laser was used as an excitation source with a power of *ca*. 200 mW. Backscattering geometry was applied. The resolution was set up for 2 cm-1; 512 scans were measured.

High resolution neutron spectra were measured on the backscattering spectrometer SPHERES [[[38]](#endnote-37)] of the Jülich Centre for Neutron Science at the Heinz Maier-Leibnitz Zentrum, Garching, Germany at temperatures between 4 and 40 K in the energy range *±*6 eV in the CH3 tunneling neutron scattering region. Software SLAW [[[39]](#endnote-38)] and FRIDA [[[40]](#endnote-39)] were used for data treatment and fitting the curves, respectively.

The optimization of the molecular structure and the frequency calculations for the isolated molecule and crystal models of N,N’-tetraacetylodiaminodurene (TADD) have been performed based on the density functional theory (DFT) using the DMol3 [[[41]](#endnote-40), [[42]](#endnote-41)] and CASTEP [[[43]](#endnote-42)] programs as a part of Material Studio Package [[[44]](#endnote-43)]. The results have been obtained within generalized gradient approximation (GGA) at PBE (Perdew-Burke-Ernzerhof) correlation functional [[[45]](#endnote-44)] in both DMol3 and CASTEP programs for isolated molecule and for crystalline state, respectively. The calculations have been performed using double numerical plus polarization (DNP) basis set and the norm-conserving pseudo-potential as implemented in DMol3 and CASTEP, respectively. DFT semi-empirical dispersion correction method [[[46]](#endnote-45)] in CASTEP calculation has been implemented. In the CASTEP program the plane-wave cut-off energy was 830 eV. Density Functional Perturbation Theory (DFPT) [[[47]](#endnote-46)] was used to calculate the vibrational properties. The IR and Raman spectra, calculated at the Gamma point, have been generated from DMol3 and from CASTEP frequency calculation results.

**Results and discussion**

*X-ray diffraction studies*

p-N,N’-tetraacetylodiaminodurene (TADD) crystallizes in space group P212121 with one molecule in the asymmetric unit. The structure with atom numbering is shown in Fig 1.

**Fig 1**

The selected bond lengths and angles are presented in Table 2.

**Table 2**

The data obtained from calculations for an isolated molecule and for the crystalline state are also included in this table (the all data are presented in supplementary Table 2S). In
Table 2 the bond length and angles are compared with the theoretical values calculated by the DMol3 and CASTEP methods. The agreement is fairly good, except for C-O bonds, whose DMol3 and CASTEP lengths are somewhat longer than X-ray ones.

In the title compound the six-member ring is essentially planar with a maximum deviation of 0.006(1) Å and four methyl groups attached to benzene ring lie close to the ring plane, whereas the methyl C atoms of the *para* substituent N-acetyl groups are well away from that plane. The four N-acetyl moieties are planar and they are approximately perpendicular to the benzene ring (dihedral angles being 89.34(4)º, 89.00(4)º, 89.58(4)º and 88.45(4)º, respectively). The two acetyl moieties form dihedral angles of 7.9(1)º and 17.8(1)º, respectively. The packing of molecules in the lattice is determined by unconventional C-H⋅⋅⋅N and C-H⋅⋅⋅O hydrogen bonds or short contacts. Their parameters are presented in Table 3 and the arrangement of the molecules in the unit cell is depicted in Fig.2a and 2b.

**Figs. 2a and 2b**

The conformation of the TADD molecule is stabilized by four C-H⋅⋅⋅N and two C-H⋅⋅⋅O intramolecular hydrogen bonds (see Table 3). The acetyl O(2) and O(4) atoms are involved in two weak intramolecular
C(17)-H(73)⋅⋅⋅O(2) and C(19)-H(92)⋅⋅⋅O(4) hydrogen bonds. The corresponding distances are equal to 2.735(2) and 2.756(2) Å, respectively.

Furthermore, the three of the four methyl groups of durene moiety form the C-H⋅⋅⋅O intermolecular hydrogen bonds (3.370(2) - 3.612(2) Å). In addition, the crystal packing is stabilized by four Cmethyl-H⋅⋅⋅O (in the acetyl group) intermolecular interactions. Analyzing the participation of the methyl groups in the intermolecular interactions one can conclude that the strongest interaction reveals for the groups containing C(17) and C(19) carbon atoms. Their C-H⋅⋅⋅O and H⋅⋅⋅O distances lie in the ranges 3.376(2) - 3.514(2) Å and 2.56(2) – 2.97(2) Å, respectively. Remaining two methyl groups C(18) and C(20) are characterized by the similar, but little longer C-H⋅⋅⋅O [3.025(2) - 3.590(2) (2) Å] and H⋅⋅⋅O [2.68(2) -2.70(2) Å] distances. Moreover, the C(20)-H(26)⋅⋅⋅O(3) contact is strongly nonlinear.

**Table 3**

Weak intra- and intermolecular C-H⋅⋅⋅N and C-H⋅⋅⋅O hydrogen bonds and the other
C-H⋅⋅⋅O contacts stabilize the packing in the crystal. The analysis of data in Table 3 seems to show that the strongest interaction with environment takes place in the case of methyl groups attached to the phenyl rings due to the C-H⋅⋅⋅N bridges. The remaining methyl groups in the acetyl units can be divided to the two parties, which differ somewhat by a packing. Generally one can tell that we are dealing with a variety of groups interacting between each other.

*Vibrational spectra*

The infrared absorption spectra measured for samples of TADD suspended in Nujol together with those calculated by the density functional theory (DFT) methods are presented in Fig 3.

**Fig. 3, Fig.4**

The experimental and calculated Raman spectrum is shown in Fig 4. The experimental frequencies are compared with values calculated by means of various theoretical approaches in Table 4S.

One can draw a few conclusions related to the vibrational spectra. The splitting of the band ascribed to the stretching vibrations of carbonyl group (νc=o equal to 1700 and
1709 cm-1) is the most significant feature of the spectra. The splitting can indicate two independent carbonyl groups in the TADD molecule. The appearance of weak band at 3384 cm-1 is also significant. It can be ascribed to the overtone of the stretching carbonyl vibrations. The Raman bands assigned to the CH3  bending vibrations over the frequency range 1331 – 1425 cm-1 evokes some attention. In the infrared absorption spectrum an intense quartet appears arising also from the CH3 bending vibrations (1224 – 1367 cm-1). In the Raman spectrum the ν(C-CH3) bands at 523 cm-1 and three wagging bands at 589, 646 and 753 cm-1 are distinguishable. The lower values of the band frequencies assigned to the torsional vibrations of the CH3 groups attached to diacetylamino (dAA) group (see Table 4S) indicate that these groups rotate more freely than the ones attached to the phenyl ring. It is well known that the stronger interactions with adjacent atoms the larger wavenumber of the CH3 deformation vibrations is observed. It should be noted that one band assigned to the torsional vibrations of the CH3 groups attached to diacetylamino (dAA) group is placed at a frequency higher than the frequencies for the other methyl groups in the TADD molecule. Therefore one could assume that the ratio of inequivalent methyl groups in dAA is equal to 1:3. The inequivalence is related to the unconventional C-H⋅⋅⋅O hydrogen bonds and short contacts that are different for each subgroup. The C-H⋅⋅⋅O bonds regard to the interactions of the methyl groups attached to diacetyloamino group with the oxygen atoms from the same dAA one.

*Tunnel CH3 splitting spectra*

The purpose of our inelastic neutron scattering (INS) studies was to determine the dynamical parameters related to the rotations of the methyl groups in TADD. We expect that a value of the barrier height for the CH3 rotation could be a measure of the hydrogen bond strength in the potential molecular complexes formed with participation of TADD. In particular a comparison of the results from the experiments on pure compound and its complex with an organic acid could allow us to draw conclusions about an effect of the hydrogen bond strength on the CH3 rotational barrier.

In this paper we present the tunneling results for TADD in the temperature region between 4 and 40 K corresponding to the tunneling of the CH3 group.

The temperature dependence of the tunneling spectra are depicted in Fig 5. In the lowest temperature region we have found one peak and one shoulder on each side of the central elastic line. This is a reason that we can analyze in details only the temperature dependence of the former peak position (±2.3 eV at 4 K). The fittings were carried out with a standard model, consisting of a Dirac component for the elastic scattering and two pairs of Lorentzians for the tunneling excitations. The theoretical curves are convoluted with the instrumental resolution function obtained during the present experiment for vanadium. It should be emphasized that during analysis of the spectra no systematic relationship on *Q* value was find. The detailed description of the fitting procedure is given in Supplementary material. Due to the fact that the peak position is close to the eleasic line (±2.3 eV at 4 K) we have not obtained the reasonable results as regards to the change of its width with temperature.

**Fig. 5**

Fig 6 shows the Arrhenius plot related to the position of the band at 2.3 eV (4 K) corresponding to the temperature range 20 - 27 K, in which the change in the position was observed. The detailed description of the coupling of the CH3 quantum rotor to phonons can be found in papers of Prager et al. [[[48]](#endnote-47), [[49]](#endnote-48)]. Taking into account the equation given in the papers mentioned above we should assume the coupling only to molecular librations shaking the potential. According to expectations this effect leads to the well-known decrease of the tunneling frequency with increasing temperature. The energy of excitation between librational energy levels, *E01,* was estimated on the basis of the following equation:

 (1)

The energy *E01* represents the distance from the pocket groundstate to the first librational level. The sinusoidal coupling coefficient, *Asin*, and the energy, *E01*, describe the interaction between the methyl group and the heat bath.

**Fig. 6**

The estimated value of the excitation energy equals to 9.7 meV, which is a typical value for the pure components containing methyl groups as well as for their molecular complexes. The observed effects are results of two interplaying effects: the crystal packing and specific interaction of the hydrogen bond type.

In order to discuss the INS tunneling spectra of TADD it is necessary to know a number of independent methyl groups in the crystal structure. The presence of two signals at ±0.9 and ±2.34 µeV should be interpreted as due to the presence of at least two independent methyl subgroups in the TADD molecule. From the X-ray data at 100 K we found eight crystallographically inequivalent CH3 groups.

In general all methyl groups in TADD create the weak hydrogen bonds or short
C-H⋅⋅⋅O and C-H⋅⋅⋅N contacts inter- and intramolecular. We may, however, roughly divide the methyl groups in TADD into two groups: (*i*) the CH3 groups connected to the phenyl ring and (*ii*) the other ones connected to diacetylamine groups (dAA). The methyl groups in the ring form slightly stronger hydrogen bonds/contacts than the ones from dAA moieties, which is supported by shorter D…A and H…A contacts and larger D-H…A angles, in particular of the intramolecular type (see Table 3). These interactions gives as a result a higher barrier for the CH3 group rotation. This observation is consistent with the calculated torsional frequencies in the solid state of TADD (see Table 4S). The torsional frequencies for the methyl groups attached to the phenyl ring are larger than those for methyl groups attached to the dAA moieties, which is related to the freer rotation of the latter groups. The exception is the torsional vibration frequency calculated for one CH3 group from the dAA group. This gives as a result the 3:1 ratio as regards to the number of methyl groups with different rotational barrier. Among the methyl groups bonded in the dAA groups the one with the C(17) carbon atom (D⋅⋅⋅A – 2.735 Å, <D-H⋅⋅⋅A – 97o) or with the C(19) carbon atom (D⋅⋅⋅A – 2.756 Å, <D-H⋅⋅⋅A – 104o) may be distinguished as they seem to form relatively stronger hydrogen bonds as compared to the other dAA methyl groups. However, the C(17) methyl groups seems to be stronger bonded by the intermolecular interactions. The remaining CH3 groups bonded to dAA seem similar as regards to the interatomic interactions.

In the tunneling spectra (see Fig. 5) two peaks with the intensities of the 3:1 ratio were recorded, which can be assigned to two types of the CH3 subgroups from dAA moieties. The intensity of the innermost peak is three times larger than that at *ca.* 2.3 µeV. Although the convolution of the model with two peaks on both sides of the elastic line is rather inaccurate as regard to the innermost peaks we can undoubtedly conclude that the innermost peaks reveal higher integral intensity than the other one. This is consistent with the expectations resulting from the crystallographic data.

In the case of methyl groups connected with the phenyl ring the splitting is probably so small that it is overlapped by the elastic scattering line.

**Conclusions**

We have investigated N,N’-tetraacetylodiaminodurene (TADD) as a potential precursor for the molecular complex with hydrogen bonds and/or with charge transfer. The crystal and molecular structure of TADD is well reproduced by the theoretical models as applied for the crystal. The spectra obtained on the basis of calculations carried out for the crystal are consistent with the experimental ones as well.

The TADD molecule is also interesting from the point of view of the methyl group tunneling in the low temperature region. From the inelastic neutron scattering (INS) at low temperature and from the calculations it appeared that the methyl groups of N-acetyl moieties behave more freely than these attached to the phenyl ring. This is supported by the lower torsional frequencies of the methyl groups attached to the N-acetyl moieties observed in the theoretical calculations performed for the TADD crystal. Moreover the intensity 3:1 ratio of the tunneling peak intensities indicates that the four methyl groups attached to the N-acetyl moieties are inequivalent. The correlation between the number of the independent methyl groups in the crystal structure and the number of tunneling peaks was observed e.g. for the neat phenantroline and its complex with picric acid [[[50]](#endnote-49)].

The excitation energy found for the tunneling effect is equal to 9.7 meV, which is similar like the values found for the other molecular complexes [49,[[51]](#endnote-50),[[52]](#endnote-51),[[53]](#endnote-52)49-52] studied by us up to date. Methyl groups both of acetyl moieties and durene form a number of non-conventional hydrogen bonds with nitrogen and oxygen atoms, which undoubtedly affects the rotational barrier for methyl groups.

In TADD molecule we deal with two types of the internal, unconventional hydrogen bonds. The methyl groups attached to the ring form weak C-H⋅⋅⋅N hydrogen bonds of the D⋅⋅⋅A lengths between 2.834 to 2.895 Å and angles between 100 and 109o (see Table 3). It seems that the strength of these bond is enough to limit the methyl groups librations. In turn the methyl groups from dAA form weak C-H⋅⋅⋅O bonds – the oxygen atom from the same dAA group – which are, however, weaker than the C-H⋅⋅⋅N bonds. This observation supports a supposition that the weak hydrogen bonds and short contacts play a decisive role in freedom of methyl rotations.

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