Low Barrier Hydrogen Bond (LBHB) Interactions in the Solid State Crystal Structures of Phthalate Salts Based on Trisubstituted Guanidinium Cations

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Abstract

The hydrogen phthalate salt \(N, N', N''\)-tricyclohexylguanidinium hydrogen phthalate \((\text{C(HNcyclohexyl)})_3\text{H}^+\text{HT}^- (1)\) was prepared and the structure was determined by X-ray diffraction analysis and characterized by elemental analysis, NMR and IR spectroscopy. The hydrogen-bonding interactions of this colorless solid have been explored in order to examine the potential of intermolecular and intramolecular interactions and their influence on the extended structure that was observed. Compound 1 crystallizes in the monoclinic space group \(P2(1)/n\), with \(a = 10.6270 (13) \, \text{Å}, b = 16.809 (2) \, \text{Å}, c = 15.1657 (18) \, \text{Å}\) and \(\beta = 102.978 (2)\,^\circ\); \(Z = 4\). The crystal data indicate that the anion displayed a characteristic intramolecular, low barrier hydrogen bond (LBHB) (S(7) motif) via O-H…O interaction. The primary structure combines two cyclic associations, a 16-membered ring (\(R_4^4(16)\) notation) and a large 46-membered ring (\(R_4^4(46)\) notation), through N-H…O hydrogen-bonding interactions. The structure of 1 is compared with the analogue salts \(N, N', N''\)-triisopropylguanidinium phthalate 2 and \(N, N', N''\)-triisopropylguanidinium hydrogen maleate 3.

Keywords: \(N, N', N''\)-Tricyclohexylguanidinium hydrogen phthalate; Hydrogen-Bonding; Intermolecular and intramolecular interactions.

Introduction

Exploring the fundamental principles that control the assembly of molecular building blocks into extended structures is an exciting challenge at the forefront of supramolecular chemistry. This knowledge could have significant impact in the areas as diverse as design of new functional materials, drug development, crystal engineering, material sciences and understanding the structures of macromolecules \(^{[1-6]}\).

The known structures of guanidinium salts with diprotic aliphatic and aromatic acids are not common and many are acid salts. A number of studies have shown that the monoanions of dicarboxylic acids form hydrogen-bonded chains \(^{[7-10]}\). These anions can align in head-to-tail chains that are joined by strong O-H…O hydrogen bonds. Modifications of the spacing group between the two carboxyl moieties can be rationally...
applied to transform these arrangements. For example, the extended structures of the hydrogen oxalate anion have exhibited the unique dimeric homosynthetically formation [9].

Within the realm of molecular materials, the parent guanidinium cation (C(NH₂)₃⁺) has been elegantly employed for the preparation of lamellar frameworks derived from hydrogen bonded sheets and ribbons and in three-dimensional networks [11-14]. A primary reason for the success of guanidinium as a triangular building block in the crystal design can be traced to its capacity to form intermolecular contacts mediated by three pairs of directional hydrogen bonding interactions. On the other hand, N-substituted guanidinium salts received less attention and fewer reports are found in the literature (15-18).

We chose to examine the potential of charge-enhanced hydrogen bonding in the extended structures of N-substituted guanidinium salts with the ultimate goal of employing these interactions in future crystal design. In connection with the ongoing studies of the structural aspects of N, N', N''-trisubstituted guanidinium cations [19-21], we herein focus on the ability of N, N', N''-tricyclohexylguanidinium cation to form a new supramolecular motif with the mono hydrogen phthalate anion. The symmetrical substitution pattern in N, N', N''-tricyclohexylguanidinium cation retains the C₃-symmetry in the cation, as in the parent guanidinium cation, and preserving the potential for three-fold, hydrogen bonding arrays.

**Experimental**

**General**

N, N', N''-tricyclohexylguanidine was prepared according to literature methods [22]. All other reagents were purchased from Aldrich Chemical Company and used without further purification. Elemental analyses were performed on a Perkin Elmer PE CHN 4000 elemental analysis system.

The IR spectrum was recorded on a Varian 640 FT-IR spectrometer in the range 4000 to 400 cm⁻¹. The high resolution NMR spectra were acquired by a Bruker Fourier 300 NMR spectrometer, using TMS as external standard.

**Synthesis and crystallization of (C(HNcyclohexyl)₃)⁺HPHT (1)**

In a round bottom flask, a combination of 0.465 Mmol of phthalic acid and 0.465 Mmol of N, N', N''-tricyclohexylguanidine were dissolved in 10 mL of THF. The reaction mixture was stirred, and a colorless precipitate formed over the next few minutes. The solid was removed by filtration and the product was crystallized from a mixture of methanol and THF in a 2/1 ratio, to give colorless crystals 93% yield (m.p. 210.1°C).

In addition to confirming the molecular formula through elemental analysis, the product obtained was examined by single crystal X-ray analysis, IR and ¹H, ¹³C NMR spectrometry.

Anal. Calculated for C₂₇H₄₁N₃O₄ C, 68.76; H, 8.76; N, 8.91. Found C, 68.49; H, 8.91; N, 8.82.
Structural Determination for \{C(HNcyclohexyl)₃\}⁺HPHT(1)

Crystals of C₂₇H₄₁N₃O₄ were grown from a solution of tetrahydrofuran and methanol. A single colorless prism suitable for X-ray diffraction measurements was mounted on a glass fiber. Unit cell measurements and intensity data collections were performed on a Bruker-AXS SMART 1 k CCD diffractometer at 202K using graphite monochromatized Mo Kα radiation (\(\lambda = 0.71073 \text{ Å}\)). The data reduction included a correction for Lorentz and polarization effects, with an applied multi-scan absorption correction (SADABS). The reflection data were consistent with a monoclinic system; P2(1)/n.

The crystal structure was solved and refined using the SHELXTL program suite. Direct methods yielded all non-hydrogen atoms which were refined with anisotropic thermal parameters. All hydrogen atom positions were located from the difference Fourier Map. The largest residual electron density peak (0.179 e/Å³) was associated with the O₁ atom. Full-matrix least-squares refinement on \(F^2\) gave \(R_1 = 0.0485\) and \(wR_2 = 0.1049\) at convergence.

Supporting Information Available

Full details for the structural determination of compound 1 have been deposited with the Cambridge Structural Data Centre (CCDC) with reference number CCDC 862146. This material is available free of charge from Cambridge Structural Data Centre, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, E-mail: deposit@ccdc.cam.ac.uk.

Results and Discussion

The guanidine employed in this study, N,N',N''-tricyclohexylguanidine, reacts readily and in near quantitative yield with phthalic acid in a 1:1 molar ratio, as summarized in (Scheme 1) below to yield \{C(HNcyclohexyl)₃\}⁺HPHT(1).

\[\text{Scheme 1}\]

Molecular Structure

The crystal data and the structure refinements are given in (Table1). The compound crystallized in the monoclinic space group P2(1)/n, \(a = 10.6270\) (13) Å, \(b = 16.809\) (2) Å, \(c = 15.1657\) (18) Å, and \(\beta = 102.978\) (2) ° and the structural diagram is presented in (Figure1). The figure reveals that the asymmetric unit consists of the
TCHG⁺ cation, which adopts C₃ orientation of the cyclohexyl groups and resides on a C₃ axis of the unit cell; along with the hydrogen phthalate anion. The bond distances and angles are summarized in (Table 2), and the metrical parameters are provided in (Table 3).

Table 1: Crystal data and structure refinement for N, N', N''-tricyclohexylguanidinium hydrogen phthalate {C(HNcyclohexyl)₃}⁺HPHT⁻(1).

<table>
<thead>
<tr>
<th>Empirical formula</th>
<th>C₂₇ H₄₁ N₃ O₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula weight</td>
<td>471.63</td>
</tr>
<tr>
<td>Temperature</td>
<td>201(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P2(1)/n</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>(a = 10.6270(13) \text{ Å}) (\alpha = 90^\circ).</td>
</tr>
<tr>
<td></td>
<td>(b = 16.809(2) \text{ Å}) (\beta = 102.978(2)^\circ).</td>
</tr>
<tr>
<td></td>
<td>(c = 15.1657(18) \text{ Å}) (\gamma = 90^\circ).</td>
</tr>
<tr>
<td>Volume</td>
<td>2639.9(5) Å³</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Density (calculated)</td>
<td>1.187 Mg/m³</td>
</tr>
<tr>
<td>Absorption coefficient</td>
<td>0.080 mm⁻¹</td>
</tr>
<tr>
<td>F(000)</td>
<td>1024</td>
</tr>
<tr>
<td>Crystal size</td>
<td>0.15 x 0.15 x 0.10 mm³</td>
</tr>
<tr>
<td>Theta range for data collection</td>
<td>1.83 to 24.69°.</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-12&lt;=h&lt;=12, -19&lt;=k&lt;=19, -17&lt;=l&lt;=16</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>17551</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>4465 [R(int) = 0.0692]</td>
</tr>
<tr>
<td>Completeness to theta = 24.69°</td>
<td>99.4 %</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.9921 and 0.9882</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on F²</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>4465 / 1 / 470</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.018</td>
</tr>
<tr>
<td>Final R indices [I&gt;2sigma(I)]</td>
<td>R1 = 0.0485, wR2 = 0.1049</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>R1 = 0.1129, wR2 = 0.1312</td>
</tr>
<tr>
<td>Largest diff. peak and hole</td>
<td>0.179 and -0.205 e.Å⁻³</td>
</tr>
</tbody>
</table>
Figure 1: Molecular structure and atom numbering scheme for \(N, N', N''\)-tricyclohexylguanidinium hydrogen phthalate \((\text{C(HNcyclohexyl)}_3)\text{HPHT}^-\text{(1)}\), (color code: Carbon yellow; Nitrogen blue; Oxygen red and Hydrogen pink).

Table 2: Bond lengths [Å] and angles [°] for \(N, N', N''\)-tricyclohexylguanidinium hydrogen phthalate \((\text{C(HNcyclohexyl)}_3)\text{HPHT}^-\text{(1)}\).

<table>
<thead>
<tr>
<th>Distances</th>
<th></th>
<th></th>
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<tbody>
<tr>
<td>N(1)-C(1)</td>
<td>1.339(3)</td>
<td></td>
</tr>
<tr>
<td>N(1)-C(2)</td>
<td>1.471(3)</td>
<td></td>
</tr>
<tr>
<td>N(2)-C(1)</td>
<td>1.336(3)</td>
<td></td>
</tr>
<tr>
<td>N(2)-C(8)</td>
<td>1.468(3)</td>
<td></td>
</tr>
<tr>
<td>N(3)-C(1)</td>
<td>1.332(3)</td>
<td></td>
</tr>
<tr>
<td>N(3)-C(14)</td>
<td>1.472(3)</td>
<td></td>
</tr>
<tr>
<td>O(1)-C(26)</td>
<td>1.218(3)</td>
<td></td>
</tr>
<tr>
<td>O(2)-C(26)</td>
<td>1.289(3)</td>
<td></td>
</tr>
<tr>
<td>O(3)-C(27)</td>
<td>1.231(3)</td>
<td></td>
</tr>
<tr>
<td>O(4)-C(27)</td>
<td>1.281(3)</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Angles</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>C(1)-N(1)-C(2)</td>
<td>124.5(2)</td>
<td>C(11)-C(10)-C(9)</td>
</tr>
<tr>
<td>C(1)-N(2)-C(8)</td>
<td>125.2(2)</td>
<td>C(12)-C(11)-C(10)</td>
</tr>
<tr>
<td>C(1)-N(3)-C(14)</td>
<td>127.1(2)</td>
<td>N(3)-C(14)-C(15)</td>
</tr>
<tr>
<td>N(3)-C(1)-N(2)</td>
<td>119.3(2)</td>
<td>N(3)-C(14)-C(19)</td>
</tr>
<tr>
<td>N(3)-C(1)-N(1)</td>
<td>120.3(2)</td>
<td>C(24)-C(25)-C(26)</td>
</tr>
<tr>
<td>N(2)-C(1)-N(1)</td>
<td>120.4(2)</td>
<td>C(20)-C(25)-C(26)</td>
</tr>
<tr>
<td>N(1)-C(2)-C(7)</td>
<td>109.1(2)</td>
<td>O(1)-C(26)-O(2)</td>
</tr>
<tr>
<td>N(1)-C(2)-C(3)</td>
<td>112.1(2)</td>
<td>O(1)-C(26)-C(25)</td>
</tr>
</tbody>
</table>
Table 3: Hydrogen parameters Bonds[Å,°] for \(N,N',N''\)-tricyclohexylguanidinium hydrogen phthalate \(\{C(HN\text{cyclohexyl})_3\}^+\text{HPHT}^-\) (1).

<table>
<thead>
<tr>
<th>D-H…A</th>
<th>d(D-H)</th>
<th>d(H…A)</th>
<th>d(D…A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1)-H(1)...O(4)#1</td>
<td>0.906</td>
<td>2.062</td>
<td>2.963</td>
<td>175</td>
</tr>
<tr>
<td>N(2)-H(2)...O(3)#2</td>
<td>0.837</td>
<td>2.135</td>
<td>2.959</td>
<td>168</td>
</tr>
<tr>
<td>N(3)-H(3)...O(1)</td>
<td>0.908</td>
<td>2.062</td>
<td>2.938</td>
<td>162</td>
</tr>
<tr>
<td>O(2)-H(20)...O(4)</td>
<td>0.856</td>
<td>1.545</td>
<td>2.400</td>
<td>175</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalence atoms: #1 1-x,1-y,1-z; #2 1+x, -1+y,z

\(^*\) van der Walls radii taken from Bondi, A.J. Phys. Chem., 1964, 68, 441. Pertinent radii; C=1.70 Å, H=1.2 Å, O=1.52 Å, N=1.55 Å

\(\{C(HN\text{cyclohexyl})_3\}^+\text{HPHT}^-\) (1) is a typical \(N, N', N''\)-trisubstituted guanidinium salt with normal geometrical parameters. The central guanidinium fragment of this cation is planar [sum of NCN angles is 360°] with bond lengths and angles as expected for a central Carbon atom with \(sp^2\) hybridization, taking into account the charge delocalization over the three C-N bonds. The average C-N bond length of [1.336(3)] Å is comparable with the literature averages for substituted and un-substituted guanidinium cations of (1.321) and (1.328) Å respectively.

The C26-O1 bond length of [1.218(3)] Å indicates a deprotonated C=O carboxyl group, which is significantly shorter than [1.289(3)] Å for the C26-O2 bond as expected for a protonated carboxyl group. On the other hand, the C27/O3, O4 distances of [1.231(3)] and [1.281(3)] Å indicate a delocalized \(\pi\)-bonding arrangement as a consequence of deprotonation of the carboxylic acid group.

The benzene ring (C20-C25) is planar and the carboxylate/carboxylic moieties are rotated slightly out of the plane as indicated by the (C21-C20-C27; O4-C27-C20, O3-C27-O4; C24-C25-C26; C25-C26-O1 and C25-C26-O2) angles; positioning the two moieties almost perpendicular to each other. The three cyclohexyl groups are also rotated slightly out of the CN3 plane as indicated by the (C1-N3-C14; C1-N2-C8 and C1-N1-C2) angles, positioning the three NH groups and the hydrogen-bonded anions on one side of this plane.

Local Hydrogen-Bonding Interactions

(Figure2) displays the local hydrogen bonding network of both the cation and the anion for \(\{C(HN\text{cyclohexyl})_3\}^+\text{HPHT}^-\) (1). The cation undergoes a hydrogen bonding motif (synthon A), and forms three different N-H…O bonds with three independent anions. The approximately linearly disposed deprotonated C=O moieties, C26=O1 and C27=O3 link via hydrogen bonding with two different guanidinium cations, N3-H3…O1
and N2-H2…O3 respectively, and the third interaction is accomplished through the deprotonated O4 center, N1-H1…O4 (Table 3).

Figure 2. A Stick and ball representation of the local – hydrogen bonding scheme for $N, N', N''$-tricyclohexylguanidinium hydrogen phthalate $\{C(HN_{cyclohexyl})_3\}^+\text{HPHT}^- (1)$ emphasizing the environments of both ions. It also displays the characteristic intramolecular low barrier hydrogen bond (LBHB) (S(7) motif O2-H20…O4. The cyclohexyl groups of the guanidinium cations and the hydrogen atoms in the benzene rings of the phthalate anions are omitted for clarity.

\[
\begin{align*}
\text{N} & \quad - \quad \text{H} \cdots \text{O} \\
\text{A}
\end{align*}
\]

The deprotonated O4 terminal experiences an additional intramolecular hydrogen-bonding with the protonated O2 center via an O2-H20…O4 interaction. The topology of the hydrogen phthalate anion, originating from the orientation of the two carboxyl moieties brings these groups into close proximity and leads to the formation of a characteristic low energy bridging hydrogen bond (LBHB) [graph-set notation S1′(7)], and locks the anion into the S(7) ring. The hydrogen phthalate anion adopts the planar cyclic orientation, owing to this strong intramolecular hydrogen bond. The double hydrogen-bonding interaction of the O(4) center (bifurcation), is in agreement with that observed for the oxalate and succinate anions in the trisubstituted guanidinium derivatives$^{[21]}$, however, in this case the (O-H…O) interaction is of the intramolecular type, and could be considered as a viable hydrogen-bond. This intramolecular interaction orients the hydrogen bonding sites for the anion in a plane and places the two formal C=O functions in an approximately linear disposition.
The Extended Structure, Crystal Packing and Crystal Supramolecularity

These local interactions generate a set of extended interactions. Two of the hydrogen bonds formed by these ions are oriented yielding the 16-membered ring ($R_4^4(16)$ notation), through N-H...O hydrogen-bonding interactions. This interaction orients the ions within this ring, in coplanar planes parallel to each other and approximately perpendicular to the bridging cations exhibiting a large 46-membered ring ($R_{12}^{12}(46)$ notation). (Figure3) displays a view of the extended hydrogen-bonding scheme, and the relative orientations of the two motifs viewed along the a-axis direction. These rings are further connected in 2-D via N-H...O hydrogen-bonding interaction yielding sheets also, along the a-axis direction.

Figure 3: A display of the extended hydrogen-bonding network for $N, N', N''$-tricyclohexylguanidinium hydrogen phthalate viewed along the a-axis direction. It emphasizes the association of the 16- and the 46-membered rings. The cyclohexyl groups and the hydrogen atoms in the benzene rings are not shown for clarity.

The third dimension for the layered structure of (C(HNcyclohexyl)$_3$)$^+$HPHT$^-$ is represented in (Figure4), which depicts the linking of the 16-membered rings through N-H...O hydrogen-bonding interactions along the b-axis direction by the bridging cations forming a double chain motif, and consolidating a 3-D supramolecular structure.
Attempts to crystallize $N$, $N'$, $N''$-tricyclohexylguanidinium phthalate (di) anion suitable for X-ray analysis, in order to compare its extended structure with compound 1 were not successful. However, $N$, $N'$, $N''$-trispropylguanidinium phthalate (2) was crystallized \cite{23}. The asymmetric unit of this salt consists of two symmetry independent cations along with the phthalate anion. One of the cations undergoes N-H…O hydrogen bonding interactions with three different phthalate anions, while the other uses only two N-H groups forming two hydrogen bonds with two different anions, and the third N-H group is not involved in the hydrogen-bonding network for this compound. The direct result of reducing the number of scaffolds constructed around the anion is
the elimination of the intramolecular O-H…O bonding interaction, and the formation of a planar cyclic phthalate anion. The extended structure of 2 involves two cyclic associations $R_2^2(8)$ and $R_4^4(16)$, resulting in a 2-D layered structure rather than a 3-D structure as it is the case in 1.

The solid-state structure of the analogue salt N, N', N''-trisopropylguanidinium hydrogen maleate (TPGH$^+$(O$_2$CC$_2$H$_4$CO$_2$H) was crystallized and its structure in the solid state was studied$^{[21]}$. The hydrogen maleate anion displayed an intramolecular, low barrier hydrogen bond, and the layered structure of this compound comprises a corrugated 2-D net constructed from 16- and 42- membered rings. The double hydrogen-bonding interaction of the deprotonated C=O observed in 1 was also observed in 3, and the extended structures of both salts are not significantly different. The frequent occurring of the described motifs demonstrates the inherent stability of these interactions and the possibility of using such supramolecular motifs as crystal engineering tools.

**IR and NMR Spectra**

IR (solid state, cm$^{-1}$): 3274 (m), 3106 (w), 2928(w), 2923(w), 1615(v.s), 1448 (v.s), 1349(v.s), 1157(m), 1058(v.s.), 899(w), 787(m), 729(m).

$^1$H NMR (d-methanol, ppm): $\delta = 8.20, 7.61, 3.50, (1.1-1.9)$.

$^{13}$C NMR (d-methanol, ppm): $\delta = 172.82, 153.97, 135.60, 133.65, 132.26, 52.63, 50.0, 49.71, 49.43, 49.14, 48.86, 48.58, 48.29, 52.63, 34.01, 26.38, 26.26$ ppm.

The broad band at ca. (3274 cm$^{-1}$) in the IR spectrum of 1 is assigned to the O-H vibration indicating the presence of O-H…O hydrogen bonds. The weak bands extending above 3500 cm$^{-1}$ are assigned to the N-H vibrations, and the relatively weak bands in the (2923-3106 cm$^{-1}$) range are characteristic of aromatic C-H vibrations. The strong absorption band at about (1615 cm$^{-1}$), is assigned to the carbonyl C=O stretching frequency, and the strong bands appearing in the range 1349–1448 cm$^{-1}$ might be assigned to the C=C vibrations of the aromatic ring.

The $^1$H NMR spectrum exhibits two signals at (7.61 and 8.20 ppm). These peaks are assigned to the aromatic protons. The broad signal (1.1-1.9 ppm) is assigned to the cyclohexyl protons, and the signal appearing at (3.50) is assigned to the N-H protons.

Finally, the $^{13}$C NMR spectrum reveals three signals in the aromatic region at (132.26, 133.65, 135.60 ppm), and two downfield resonances at (153.97) and (172.82) ppm. The signal at (172.82 ppm) is assigned to the carbonyl group, and that at (153.97 ppm) is assigned to the CN$_3$ group. The signals at (52.63, 34.01, 26.38 26.26 ppm) are assigned to the cyclohexyl groups.

Copies of the spectra are provided within the supplementary material section.
Conclusions

\(N, N', N''\)-tricyclohexylguanidinium hydrogen phthalate \\
\(\{\text{C(HNcyclohexyl)}_3\}^+\text{HPHT}^-\) exhibits a number of common structural features with \nclose relatives of this salt in terms of topology and hydrogen bond connectivity. The \npseudo-C\(_3\) symmetric trisubstituted \(N, N', N''\)-tricyclohexylguanidinium cation employs \nall hydrogen donor groups in N-H...O interactions with the hydrogen phthalate anion. \nThe hydrogen phthalate anion displayed a characteristic intramolecular, low barrier \nhydrogen bond (LBHB) (S(7) motif).

Attempts to crystallize \(N, N', N''\)-tricyclohexylguanidinium phthalate 2 were not \nsuccessful, and efforts are underway to obtain crystals of this salt suitable for X-ray \nanalysis. The fact that the trisubstituted guanidinium moieties used in these studies \nhave been proven to form strong interactions in competitive solvent systems with \nmultiple N–H...O hydrogen-bonds, to support the overall connectivity through \nhydrogen bonding and charge pairing could be treated as viable interactions.

Our continuing efforts are attempting to unravel the influence of substituents, \nand fundamental structural features of solids designed around novel \(N, N', N''\)– \ntrisubstituted guanidinium cations.

Acknowledgement

The author thanks Dr. Tara Kell for her help in the x-ray analysis of the titled \nstructure, and Dr. Glenn Facey for measuring the NMR spectra. My gratitude is \nextended to Professor Darrin Richeson for his support.

Supplementary Materials

![IR spectrum for N,N',N''-tricyclohexylguanidinium hydrogen phthalate](image-url)
$^1$H nmr spectrum for N,N',N''- tricyclohexylguanidinium hydrogen phthalate

$^{13}$C nmr spectrum for N,N',N''- tricyclohexylguanidinium hydrogen phthalate
References


