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Protonated Bis(quinuclidine) Included in a Novel "Bichannel" (Urea)–Chloride Host: a Very Large Urea Channel

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The synthesis, crystal structure and properties of a novel ternary inclusion compound formed by protonated bis(quinuclidine) inserted in a urea–chloride anionic host, urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$ (1), are reported. In the host structure each urea molecule interacts with adjacent urea molecules via N—H···O hydrogen bonds forming a channel arrangement. Each hexagonal channel is linked through halide ions by N—H···Cl hydrogen bonds forming a bichannel-like structure. The channel width of the host (14.32 Å) is one of the largest known for a urea host derivative. The compound may be described as a commensurate species, $c_g/c_h = 0.98$. Conductivity determined for 1 at room temperature is 4.00×10^{-6} (Ω cm)$^{-1}$, and it increases with temperature to 3.33×10^{-4} (Ω cm)$^{-1}$ at 77°C. Aspects of the thermal stability of the product are discussed.

Keywords: Inclusion compound; Urea; Quinuclidine; Bichannel urea host; Urea–chloride host

INTRODUCTION

Inclusion compounds are suitable systems for investigating the nature of molecular guest–host interactions; they have received increasing attention as materials for electronic and optoelectronic applications [1–3]. Host materials such as urea, thiourea, cyclodextrins, calixarenes, zeolites and perhydrotriphenylene are able to form specific host–guest architectures in which the guest molecules are incorporated along channels, within layers, or in isolated cages [4–6]. Among these compounds, urea inclusion compounds have been particularly investigated. X-ray diffraction studies have shown that typical binary compounds such as those with hydrocarbons and urea lead to hexagonal channel structures [7–9]. Guest species containing amino or azacyclo groups may also be included in urea and thiourea hosts [10–14]. Using 1,2-diazabicyclo[2.2.2]octane or hexamethylenetetramine as guest and thiourea as host, it has been possible to obtain binary layered inclusion compounds [14,15]. Using a bulky amine such as dicyclohexylamine, a hexagonal channel was obtained [13]. The urea host is formed by hydrogen bonding, and a variety of guest molecules of appropriate size and shape can be included. The small urea channels, however, are appropriate only for storing unbranched hydrocarbon chains. In this way it is possible to obtain derivatives with a urea matrix containing channels large enough to accommodate bulky molecules. The internal diameter of the hexagonal channel of urea hosts is about 5.3–5.7 Å. The synthesis of ternary compounds offers the possibility to expand the urea channel. Furthermore, if both host and guest are charged species, there is also the possibility of obtaining ion-conductive materials.

Examples of ternary complexes of urea are few [16–20]. The structural characterization of the isomorphous series of $[(C_2H_5)_4N^+X^-]\cdot(NH_2)_2CO\cdot 2H_2O$ (X = Cl, Br, CN) has been reported. In these compounds the cations are sandwiched between hydrogen-bonded urea–water–halide/pseudohalide puckered layers [19,20]. Recently,

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the ternary layer urea inclusion compounds urea$_2$[quinuclidine$_2$H]$^+$Br$^-$ and urea$_2$[quinuclidine$_2$H]$^+$I$^-$ were synthesized using the partial hydrolysis of lithium bromide and iodide. In the structure, the diquinuclidinium ions occupy the interlayer region of the urea–bromide and –iodide hosts [21]. In the two structures, each urea molecule interacts with adjacent urea molecules via N—H···O hydrogen bonds forming a ribbon-like arrangement. The ribbons are linked through halide ions by N—H···X hydrogen bonds forming layered structures. Using lithium chloride a very different structure was obtained, which we describe in this work.

We chose quinuclidine (1-azabicyclo[2.2.2]octane) as a guest, considering both its small cyclic structure and relatively mild basic properties. The proton affinity of quinuclidine is 236 kcal mol^{-1}, and the three-dimensional model with space occupation indicates that there is no strain energy as in other pyramidal nitrogen compounds such as triethylamine [22].

In this paper we describe the synthesis, characterization and conductivity properties of urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$ (compound **1**).

EXPERIMENTAL

Commercially available reagents were used as received. The product was obtained from solutions of urea, quinuclidine and lithium chloride in a 2:2:1 molar ratio in methanol by slow solvent evaporation at room temperature. After about 24 h, well-formed thin needle crystals may be separated. The product was washed with cold methanol and dried under vacuum. The crystals, showing characteristic behaviour under a polarized light microscope, can be rapidly distinguished and separated from lithium chloride, quinuclidine or pure urea; purity was further verified by polycrystalline powder diffraction analysis of the samples. The crystals decompose in water producing a characteristic visual dynamic effect [23]. ^{13}C CP-MAS NMR spectra were recorded on a Bruker AMX-300 spectrometer at a frequency of 75.47 MHz for ^{13}C. The number of scans was 4096 with 5.0 μs 90° pulses, 5 ms cross-polarization contract time, 50 ms acquisition time during proton decoupling, and 5 s recycle delay. The samples were spun at a frequency of 4 kHz. Chemical shifts are given with respect to TMS using glycine (175 ppm) as external standard. Single crystals were selected for X-ray diffraction analysis. Data were collected on a Smart Apex diffractometer with graphite monochromatic MoKα radiation ($\lambda = 0.71073$ Å) at 293 K. Intensity data for 7090 reflections ($h = -12$ to 12, $k = 0$ to 16, $l = -19$ to 20) were collected in the 2θ scan mode in the range 3.60–46.60° ($R_1 = 0.0672$ and $wR_2 = 0.2138$). The 7090 reflections collected are the unique data. Intensity data for **1** were corrected for Lorentz, polarization and absorption effects. The empirical absorption corrections were determined from the intensities using Sadabs in the Saint_NT V6.02a (Bruker, 2000) program. A Bruker Smart-NT V5.624 program was used for data collection and cell refinement, and a Bruker Saint-NT V6.02a program for data reduction. The structure was solved by direct methods using the SHELXS-97 program [24]. The crystals correspond to space group *Pc*. The correct absolute structure has been determined, and evidence of this supplied in the form of the Flack parameter. The positions of the hydrogen atoms were calculated geometrically using the riding model with fixed isotropic temperature factors.

Fully completed CIF were deposited in the Cambridge Crystallographic Data Centre: CCDC 179060.

A Parc model 63100 Electrochemical Impedance Analyser in the range 1–10^5 Hz was used for the conductivity measurements.

RESULTS AND DISCUSSION

Synthesis of the compound was carried out in methanol; the partial hydrolysis of lithium chloride produces protons and chloride ions necessary for obtaining both the ionic guest and the host. Moreover, this synthesis method allows the removal of water from the solvent, so the crystals do not contain water as occurs in other known urea ternary compounds reported previously [19,20].

Structural determination was carried out, and a representation of the molecular components of the structure of **1** is shown in Fig. 1. From Fig. 2, the structure may be described as a series of infinite, non-intersecting hexagonal channels lacking one

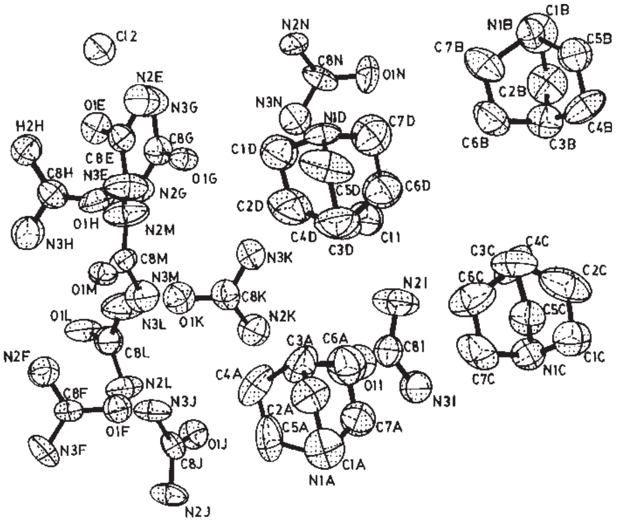

FIGURE 1 Representation of molecular components of urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$.

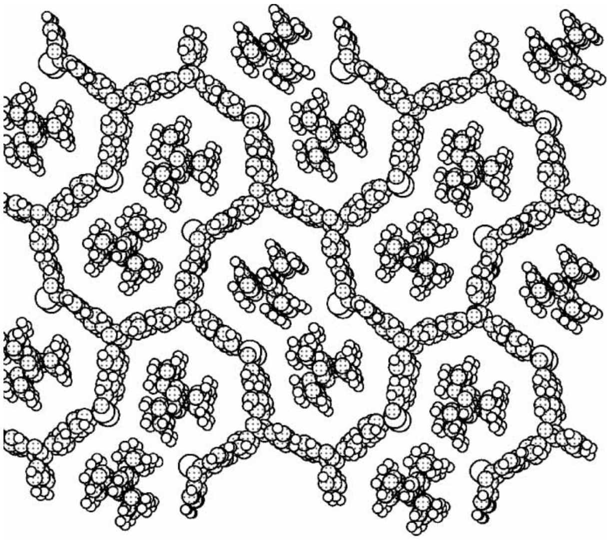

FIGURE 2 Channel structure of urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$.

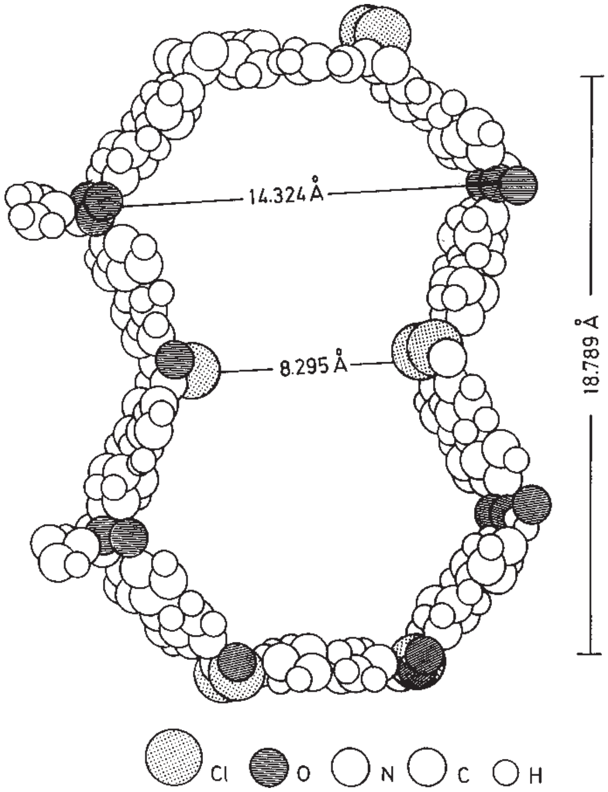

FIGURE 3 Representative distances of the cross-section viewed along the channel axis.

side. The channel structure results from two kinds of hydrogen bonds: urea molecules interact with adjacent urea molecules via 3.083-Å long N—H···O hydrogen bonds with an angle of 167.69°, and urea molecules also interact with chloride ions through N—H···Cl hydrogen bonds. This results in an anionic framework channel as a component of the crystal packing. The chloride atoms are located exactly on the vertex where the channel sides are missing. Each chloride ion forms four N—H···Cl hydrogen bonds of length 3.171–3.255 Å with angles in the range 156.57–163.72°. Figure 3 shows the most representative distances of the cross-section viewed along the channel axis. The channel of this host structure, the largest known until now, is 14.324 Å in diameter, allowing the incorporation of large guests such as diquinuclidinium ions in which the N···H···N hydrogen bond is 2.706 Å long, with an angle of 177.08°. Structural parameters as well as the most relevant bond lengths and angles are given in Tables I and II.

TABLE I Structural parameters for urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$

Crystal system	Monoclinic
Space group	*Pc*
Empirical formula	$C_{19}H_{47}ClN_{12}O_5$
Formula weight	559.14
a/Å	11.284(5)
b/Å	14.577(6)
c/Å	18.415(7)
β/°	102.435(7)
Cell volume/Å^3	2958(2)
Z (formula units/cell)	4
Density (calculated)/g ml^{-1}	1.256
Absorption coefficient/mμ, mm^{-1}	0.179
F(000)	1208
Radiation source	MoKα
Wavelength/Å	0.7107
Data collection temperature/K	293(2)
2θ range/°	3.60–46.60
Index ranges	$-12 < h < 12$; $0 < k < 16$; $-19 < l < 20$
Reflections collected	7803
Observed reflections	7090
Residuals (observed data)	0.0672
Residuals (all data)	0.0715
w Residuals (all data)	0.2138
Crystal size/Å	0.18 × 0.07 × 0.03

TABLE II Relevant bond lengths and angles of urea$_5$ [quinuclidine$_2$H]$^+$Cl$^-$

Bond length/Å	
O(1K)–C(8K)	1.251(5)
N(1A)–C(7A)	1.482(6)
N(2K)–C(8K)	1.313(6)
C(1A)–C(2A)	1.49(1)
C(3A)–C(4A)	1.499(7)
C(4A)–C(5A)	1.512(7)
N(1A)–C(1A)	1.492(6)
N(1A)–C(5A)	1.483(6)
N(3K)–C(8K)	1.322(6)
C(2A)–C(3A)	1.503(6)
C(3A)–C(6A)	1.506(7)
C(6A)–C(7A)	1.507(7)
Bond angle/°	
C(1A)–N(1A)–C(7A)	109.6(5)
C(7A)–N(1A)–C(5A)	107.0(5)
C(3A)–C(2A)–C(1A)	110.1(5)
C(4A)–C(3A)–C(6A)	107.4(6)
C(3A)–C(4A)–C(5A)	109.1(5)
C(3A)–C(6A)–C(7A)	110.6(5)
N(3K)–C(8K)–N(2K)	118.5(4)
N(2K)–C(8K)–O(1K)	121.1(4)
C(1A)–N(1A)–C(5A)	108.9(5)
N(1A)–C(1A)–C(2A)	111.4(5)
C(4A)–C(3A)–C(2A)	108.0(6)
C(2A)–C(3A)–C(6A)	109.7(6)
N(1A)–C(5A)–C(4A)	111.5(5)
N(1A)–C(7A)–C(6A)	110.2(5)
N(3K)–C(8K)–O(1K)	120.3(4)

A fundamental issue for conventional binary channel urea inclusion compounds is knowing the correlation between the position of the guest molecules in different tunnels and the overall three-dimensional ordering of the guest molecules [25–27]. In commensurate systems there is a well-defined positioning of the guest molecules at specific locations within each channel, thus giving rise directly to a three-dimensional ordered arrangement of guest molecules controlled by the three-dimensional periodicity of the host structure. Most binary urea inclusion compounds are incommensurate [26], but amine–urea binary inclusion compounds have commensurate properties [10]. The positional relationship between molecules in adjacent channels is conveniently described in terms of the offset, denoted by Δ_g, along the channel axis between the centres of mass of guest molecules in adjacent channels. An inclusion compound is described as commensurate if there are sufficiently small integers p and q to satisfy the equality $pc_h = qc_g$. We apply these concepts determined for binary channel structures to the novel ternary channel urea inclusion structure of **1**. From crystallographic studies we can obtain c_g and c_h; the former correspond to the parameters associated with the $h\,k\,l$, that is 0 0 1 (direction c), while the latter corresponds to lattice parameter c along the channel axis. Table III shows the c_g and c_h values, and the c_g/c_h ratio for **1**. They clearly indicate that the compound is commensurate, that is around each guest molecule there is the same host structural conformation. Figure 4 shows a schematic representation of this commensurate system for **1**.

TABLE III c_g, c_h and c_g/c_h values for urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$

Compound	c_g/Å	c_h/Å	c_g/c_h
1	17.983	18.42	0.98

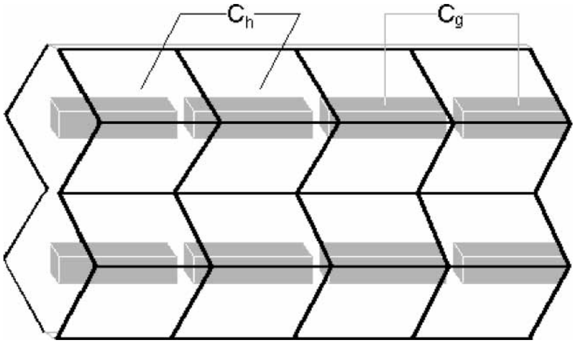

FIGURE 4 Schematic representation of the commensurate properties of urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$.

The compound does not show topotactic properties. Obviously, the anionic matrix structure needs the presence of the cationic guest, so escape of the guest leads to destruction of the host structure. The differential thermal analysis of **1**, illustrated in Fig. 5, shows a symmetrical peak centred at 118.6°C, corresponding to the melting point. Exactly at this temperature the loss of mass due to the rupture of the hydrogen bonds of the matrix begins, with the simultaneous escape of the guest. At 300°C, when 80% of the total mass is lost, a clear change in the mass loss rate is observed. In the DTA, urea and **1** show one endothermic peak before melting that may be attributed to a structural change leading to a crystalline system of lower symmetry, a process that is not accompanied by a mass loss.

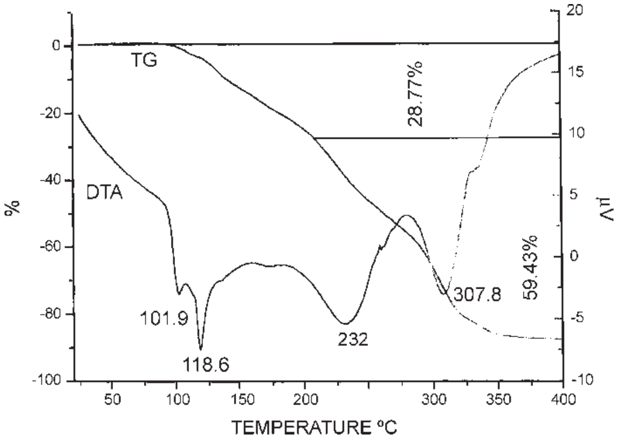

FIGURE 5 Thermal analysis, DTA and TG of urea$_5$[quinuclidine$_2$H]$^+$Cl$^-$.

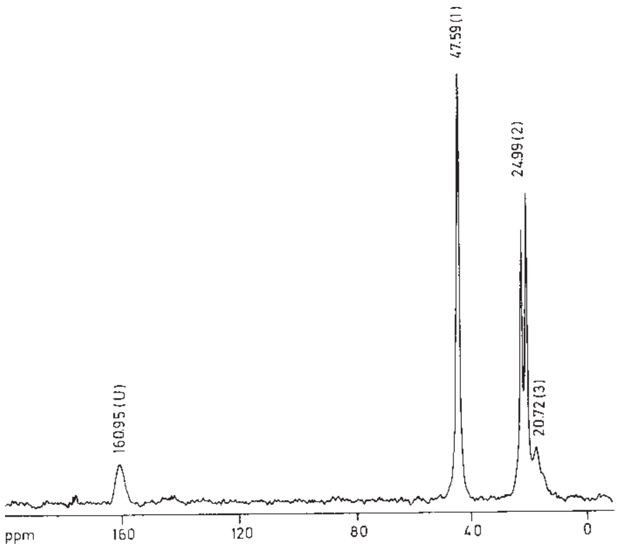

FIGURE 6 ^{13}C CP-MAS NMR spectrum of $urea_5[quinuclidine_2H]^+Cl^-$.

If the crystals of the product are left in contact with a high polarity solvent such as DMSO or water [23], the quinuclidine appears to be rapidly extracted from the solid, propelling the crystal through the liquid surface. The evolution of the quinuclidine can be detected visually by adding phenolphthalein to the solution, when the motion effect is accompanied by the formation of a beautiful red "wake" in the liquid. This experiment suggests a host structure rupture producing urea, Cl^-, protons and quinuclidine in solution.

The ^{13}C CP-MAS NMR spectrum of **1** at room temperature, given in Fig. 6, shows a wide signal centred at 163.9 ppm (line width ≈335.6 Hz), corresponding to urea. Similar chemical shift values have been reported previously [28] for binary urea inclusion compounds. Relatively narrow signals corresponding to the carbon nuclei (C_α) bonded directly to nitrogen in the diquinuclidinium ion appear at 47.58 ppm (line width ≈89.4 Hz). For ^{13}C directly bonded to nitrogen (^{14}N, $I = 1$), MAS frequently gave a powder pattern consisting of a doublet because the ^{13}C and ^{14}N residual dipolar interaction is not averaged to zero by MAS NMR [29–31]. Despite the low amplitude motion of the diquinuclidinium guest within the host channels at room temperature, doublets are not observed. Table IV shows the chemical shifts of ^{13}C CP-MAS NMR of urea, quinuclidine and **1**. The influence of hydrogen bonding on the ^{13}C chemical shifts can be understood in general by the sigma electron push-pull effects [22,30,32,33] produced in our case by the charge displacements from quinuclidine towards the acceptor medium via hydrogen bonding association of the quinuclidinium ions. According to the isovalent hybridization concept [22], the interaction of the basic quinuclidine nitrogen atom with the protons should induce an increase in the s-character of the C_α hybrid orbitals directed towards its neighbours, but the N atom produces a high field shift of its ^{13}C NMR signal. The ^1H CP-MAS NMR signal corresponding to the proton bridge located between quinuclidine should appear at 1.43 ppm, probably overlapped with the signal of the proton linked to the γ-carbon, which can be determined by the signal integrals.

TABLE IV ^{13}C CP-MAS NMR. Chemical shifts of urea, quinuclidine and $urea_5[quinuclidine_2H]^+Cl^-$ (**1**)

		^{13}C Quinuclidine/ppm		
Compound	^{13}C Urea/ppm	α	B	γ
Urea	174			
Quinuclidine		49.02	28.34	22.26
1	163.95	47.58	24.99	20.72

TABLE V Temperature dependence of conductivity data $(\Omega\,cm)^{-1}$ for urea and $urea_5[quinuclidine_2H]^+Cl^-$ (**1**), measured in pellets

Temperature/°C	$Urea_{amb}$	$\mathbf{1}_{amb}$	$\mathbf{1}_{vac}$
25	$6.84\ e^{-8}$	$4.00\ e^{-6}$	$5.48\ e^{-7}$
40	$3.64\ e^{-8}$	$3.14\ e^{-6}$	$1.06\ e^{-6}$
60	$1.81\ e^{-8}$	$3.06\ e^{-5}$	$2.21\ e^{-6}$
77	$1.95\ e^{-8}$	$3.22\ e^{-4}$	$9.07\ e^{-10}$

Ionic conductivity studies of **1** were also carried out using a diffuse impedance instrument. Measurement was performed on pellets, because obtaining crystals of adequate size for anisotropic measurements was unsuccessful. The conductivity behaviour produced by the proton displacement probably depends on the measuring conditions, increasing with temperature and going from insulators to half-conductors. The presence of humidity allows greater cohesion of the crystalline powder and facilitates the formation of better compressed pellets. Table V shows the temperature dependence of the conductivity of urea and **1**.

CONCLUSIONS

The synthesis, crystal structure and thermal and conductivity properties of the novel ternary inclusion compound **1** ($urea_5[quinuclidine_2H]^+Cl^-$) has been investigated systematically by a wide range of characterization and analytical methods (^{13}C and ^1H CP-MAS NMR spectra, X-ray diffraction analysis, electrochemical impedance spectroscopy, DTA/TG).

In the structure, each hexagonal channel is linked through halide ions by N—H···Cl hydrogen bonds forming bichannel-like structures with the diquinuclidinium ions included in them.

The inclusion compound is commensurate, that is around each guest molecule there is the same host structural conformation.

The conductivity of polycrystalline samples of the synthesized compound depends on the measurement conditions, and increases with temperature. Conductivity values are 4.00×10^{-6} and 3.33×10^{-4} $(\Omega\,\mathrm{cm})^{-1}$ at room temperature and at 77°C, respectively.

Acknowledgements

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