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Article Constructor Graphs as Useful Tools for the Classification of Hydrogen Bonded Solids: The Case Study of the Cationic (Dimethylphosphoryl)methanaminium (*dpma*H⁺) Tecton

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Abstract: The structural chemistry of a series of dpmaH (dpmaH = (dimethylphosphoryl)methanaminium) salts has been investigated using constructor graph representations to visualize structural dependencies, covering the majority of known dpmaH salts. It is shown that the structurally related α -aminomethylphosphinic acid can be integrated in the systematology of the dpmaH salts. Those dpmaH salts with counter anions that are weak hydrogen bond acceptors (ClO₄⁻, SnCl₆²⁻, IrCl₆²⁻, IrCl₆²⁻, J⁻) tend to form head-to-tail hydrogen bonded moieties purely consisting of $dpmaH^+$ cations as the primarily structural motif. In structures with weak to very weak hydrogen bonds between the $dpmaH^+$ cations and the counter anions, the anions fill the gaps in the structures. In salts with medium to strong hydrogen bond acceptor counter ions (Cl⁻, NO₃⁻, PdCl₄²⁻), the predominant structural motif is a double head-to-tail hydrogen bonded ($dpmaH^+$)₂ dimer. These dimeric units form further NH···· X hydrogen bonds to neighboring counter anions X, which results in one-dimensional and two-dimensional architectures.

Keywords: hydrogen bonding; constructor graph; crystal engineering; pattern analysis; phosphoryl compounds

1. Introduction

Neutral (dimethylphosphoryl)methanamine (*dpma*) has been synthesized decades ago [1,2], and its crystal structure is well-known [3]. Furthermore, it has been shown that *dpma* is a potent bidentate ligand stabilizing mononuclear [3–5] and oligonuclear [6] complexes as well as coordination polymers [7]. In recent years, we have shown that the N-protonated *dpma*H⁺ cation is an excellent monodentate ligand. Based on its conformational flexibility—via rotation about the central P–C bond—(Scheme 1) the *dpma*H⁺ ligand is able to fit with the needs of coordination, packing and hydrogen bonding in complexes such as [Mn(H₂O)₂Cl₃(*dpma*H)], [*M*Cl₂(*dpma*H)₄][*M*Cl₄]₂, (*M* = Co(II), Cu(II)) [8].

Above all, the *dpma*H⁺ cation is an excellent tecton to construct hydrogen bonded architectures. In terms of the concepts of Crystal Engineering, the *dpma*H⁺ is a building block with a threefold hydrogen donor functionality at one end (the NH₃ group) and a hydrogen-bond acceptor functionality at the oxygen atom of the phosphoryl moiety at the other end (Scheme 1, left part). *dpma*H⁺ can thus be abstracted by a block-shaped informative icon (Scheme 1, right part) which reduces the functionality of the tecton to the striking symbols of a blue plus sign (indicating the NH₃ group) and a red dot

(indicating the oxygen atom), which enables a general und uniform description of simple and especially complicated structures e.g., for *dpma*H⁺ salts.



Scheme 1. Hydrogen bonding functionality of the *dpma*H⁺ tecton (**left part**) and the corresponding icon useable in constructor graphs (**right part**).

Coding of individual hydrogen bonding motifs by graph sets classification and notation has been widely used in the literature [9–11]. The main disadvantage of classical graph sets in the context of a broader comparison of chemically varied compounds is that similar connectivities may not be displayed in the corresponding graph set descriptors. A system less frequently used which is more favorable to classify larger sections of hydrogen bonded structures and to compare related structures is based on so-called constructor graphs [10,11]. In this methodology, each molecule is substituted by a dot and hydrogen bonds are symbolized by arrows pointing from the donor to the acceptor group. Infrequent use of constructor graphs may be related to the fact that the advantage of the abstraction of multifunctional molecules to dots is not intuitively comprehensible whereas the individual graph sets coding similar hydrogen-bond motifs are very handy for the description of one isolated structure or structure type. Further developments in this area concerns automated analysis and interpretation of hydrogen bonded networks [12]. The present contribution is meant to exemplarily show the ability of constructor graphs to classify extended hydrogen bonded structures. Furthermore, the usage of informative icons instead of simple dots in these constructor graphs is suggested, which should be more intuitive. Modified constructor graphs illustrated by such informative icons will be used throughout this contribution. Hopefully, a broader use of constructor graphs may support activities to systematize structurally and hierarchically related hydrogen bonded solids.

2. Results and Discussion

The hydrogen-bond functionality of $dpmaH^+$ predestines this cation for the formation of head-to-tail connected dimers, chains or higher dimensional polymers. Most $dpmaH^+$ salts containing weakly hydrogen bond accepting counter anions have the tendency to form $(dpmaH^+)_n$ polymers. Salts with stronger hydrogen bond accepting counter anions mainly comprise $(dpmaH^+)_2$ of dimers which are further connected by the counter anions.

2.1. Salt Structures Based on Head-to-Tail Connected Hydrogen Bonded Polymers: $(dpmaH^+)_n$

Hexahalogenometallate dianions of tetravalent metals like tin [13], iridium [14] and platinum [15] and some other perhalogenometallates are well known to be weak hydrogen bond acceptors, showing a tendency to bifurcated hydrogen bonds [16]. The synthesis of the $(dpmaH)_2[MCl_6]$ with M = Sn, Ir gave structures that show a highly symmetrical arrangement of the $[MCl_6]^{2-}$ anions in a monoclinic unit cell (Figure 1). The substructure formed by the anions seems to be

a common feature of many hexahalogenidometallate salts, ranging from K_3 [IrCl₆] [17] to analogous alkylammonium salts [13–15,18]. Hydrogen bonded ($dpmaH^+$)_n polymers form the one-dimensional cationic substructure.



Figure 1. (Upper part): Structure of $(dpmaH)_2$ [SnCl₆] with view along [100]; (Lower part): modified constructor graph of the polymeric, cationic $(dpmaH^+)_n$ substructure.

This polymeric chain is formed by a head-to-tail connection of *dpma*H⁺ units, which is an ubiquitous motif throughout structural chemistry. A prominent example would be dicarboxylic acids [19], which are well documented neutral compounds forming such a hydrogen bonding scheme. However, a few cationic chains are also documented in combination with weakly coordinating anions [20–22]. Starting from linear zig-zag arrangements, it is obvious that the formation of a ring motif should be possible.

In the structure of *dpma*HI exactly such a four-membered ring is present (Crystal data of *dpma*HI (C₃H₁₁INOP): $M_r = 235.00$, T = 292 K, crystal size: $0.15 \times 0.13 \times 0.07$ mm³, monoclinic, $P2_1/n$, a = 12.0399(5), b = 8.7857(4), c = 15.4854(5) Å, $\beta = 90.689(4)^{\circ}$, MoK α -radiation ($\lambda = 0.71073$ Å), 11651 measured reflections ($R_{int} = 0.0363$), completeness > 99% up to 2theta = 51°, 3014 unique reflections, 2664 observed reflections ($I > 2\sigma(I)$), solution and refinement were carried out with the SHELX system [23], 136 refined parameters, $R^1(I > 2(I)) = 0.0461$, wR^2 (all unique reflections) = 0.0906. CCDC 1417987 contains the supplementary crystallographic data for this crystal structure. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/getstructures). Each four-membered centrosymmetric ($dpmaH^+$)₄ moiety (Figure 2a, graph set descriptor $R^4_4(20)$) is furthermore connected to neighboring iodide counter anions to form a two-dimensional hydrogen bonded polymer.



Figure 2. (a) Showing the primary structural motif of a four-membered, hydrogen bonded ring $(N-H\cdots I \text{ hydrogen bonds to the adjacent iodide counter anions are omitted}); (b) modified constructor graph of the tetrameric ($ *dpma*H⁺)₄ ring unit.

The next, more complex motif can be obtained by connecting two adjacent hydrogen bonded parallel chains. Figure 3a shows the corresponding strand (cationic substructure) of the structure of the *dpma*H[ClO₄] salt [24]. To realize an optimal interaction between the neighboring chains, they are shifted against each other by one half of the repetition unit. Consequently, hydrogen bonded ring motifs with the graph set descriptor $R^2_3(9)$ characterize the connections between parallel chains.



Figure 3. (a) part of the hydrogen bonded chain structure and modified constructor graph of dpmaH[ClO₄]; (b) hydrogen bonded strands in the structure of *dpma*HX·*dpma* ($X = [ClO_4]^-$ [24], I⁻ [25]) and its modified constructor graph (the *dpma* molecule is shown as brick-shaped icon composed of a black circle and a red dot; red colored circle indicates a composite tecton); (c) hydrogen bonded strands constructed from head-to-tail connected {*dpma*H}+[HSO₄]⁻ ion pairs [26] and the corresponding modified constructor graph (brick-shaped icon composed of a black circle and a red minus sign is shown for the HSO₄⁻ anion; red colored circle indicates a composite tecton).

A more complex cationic, one-dimensional strand-type motif with the general formula $dpmaHX \cdot dpma$ ($X = [CIO_4]^-$ [24], I⁻ [25]) is closely related to the double chain structure discussed before (Figure 3b). In this more complex case, the $dpmaH^+$ cation (icon as introduced before) and one neutral dpma molecule (brick-shaped icon composed of a black circle and a red dot) can formally combined to give a new tecton which is indicated by a red colored circle in Figure 3b. This new assembly generally has the same functionality as the "pure" $dpmaH^+$ tecton alone. Each $dpmaHX \cdot dpma$ unit is head-to-tail connected to two adjacent ones. In addition, this primary chain structure is connected to another parallel chain in the same way as in the prototype structure of $dpmaH[CIO_4]$ (Figure 3a). The structural similarity between them can be easily summarized by the two constructor graphs shown in Figure 3a and b, but not by the associated individual graph set descriptors of the hydrogen bonded rings (R²₂(10); R³₄(11); R⁴₆(15)) in the structure of $dpmaHX \cdot dpma$ and those in the structure of $dpmaH[CIO_4]$.

A neutral chain-type structure similar to that present in the structures of the *dpma*HX·*dpma* salts can be obtained by combination of the *dpma*H⁺ cation with an HSO₄⁻ anion. The HSO₄⁻ anion is a typical tecton which is predestined to do a head-to-tail connection. In this case, the implementation of a brick-shaped icon composed of a black circle and a red minus sign for the HSO₄⁻ anion is useful (Figure 3c). In the structure of *dpma*H[HSO₄] [26], we find the desired head-to-tail connected ion pairs (Figure 3c; highlighted by a red colored circle) which are connected to the neighboring ones in the same manner as in the structure of *dpma*HX·*dpma*. This similarity can easily be visualized by the three corresponding constructor graphs in Figure 3. Within the {*dpma*H}⁺[HSO₄]⁻ ion pair, the HSO₄⁻ anion is a medium-strong hydrogen bond donor and at the same time a hydrogen bond acceptor. Therefore, the HSO₄⁻ anion in this structure forms a hydrogen bonding scheme which is identical to that of neutral *dpma* molecule in *dpma*HX·*dpma*. For this structure, the individual graph set descriptors for the ring motifs (R⁴₄(12) and R²₂(9)) again do not fit with those of the structurally related structures *of dpma*HX·*dpma* and *dpma*H[ClO₄].

As an intermediate conclusion, two things should be mentioned:

- A comparison of the chain-type and strand-type structures discussed before has been simplified by the use of the modified constructor graphs.
- The intuitive dissection of these complex hydrogen bonded structures into subunits (tectons) that look like as they were taken from a "chemical toolbox", unquestionable supports the understanding of the individual structures and should support the prediction of structural features.

The next systematic step towards a higher dimensional framework would be the construction of layered structure consisting of parallel chains. For steric reasons, this motif is not possible for the *dpma*H⁺ tecton. Furthermore, it would be very unusual for a phosphoryl group to accept more than two hydrogen bonds. A systematic search for a related tecton shows that the zwitterionic α -aminomethylphosphinic acid (H₃N–CH₂PO₂Me) [27] exactly forms the predicted layered structure. This alternative tecton can be easily generated from the *dpma*H⁺ tecton by the exchange of a methyl group with an oxido group. This modification of the basic tecton generates one more hydrogen-bond acceptor atom at the formal negatively charged part of the zwitterionic α -aminomethylphosphinic acid (Figure 4).



Figure 4. Hydrogen bonding pattern of the zwitterionic α -aminomethylphosphinic acid [27] and its modified constructor graph.

In the structure of α -aminomethylphosphinic acid, each of the three hydrogen atoms of the aminium group forms an unbifurcated hydrogen bond to a symmetry related molecule. The formation of a three-dimensional hydrogen bonded connection of α -aminomethylphosphinic acid tectons or of

closely related molecules seems to be possible, in general, but to the best of the author's knowledge, such a structure has not been determined to date.

2.2. Salts Based on Hydrogen Bonded (dpmaH⁺)₂ Dimers

For salts containg $dpmaH^+$ and counter anions, which are good or excellent hydrogen-bond acceptors, a very different structural chemistry can be observed. The predominant motif in these structures are head-to-tail connected $(dpmaH^+)_2$ units (Figure 5). Two out of six hydrogen atoms of the two aminium groups are used for the formation of the dimer. The four other hydrogen atoms are generally available for the formation of hydrogen bonds to the counter anions.

In the structure of dpmaHCl [28], such (dpmaH⁺)₂ dicationic units are present. Each dicationic dimer donates four NH···Cl hydrogen bonds, and each chloride anion accepts two of them to form a chain structure (Figure 5). The constructor graph for this structure illustrates the structural similarity to dpmaH[ClO₄]· dpma (Figure 3b), which also consist of head-to-tail connected basic units. In the structures of dpmaH[ClO₄]· dpma, the oxygen atom of the phosphoryl group accepts two hydrogen bonds to realize chain propagation. In the structure of dpmaHCl, there is no need to form such a bifurcated hydrogen bond at the phosphoryl group because of the presence of chloride anion, an excellent hydrogen bond acceptor.



Figure 5. Hydrogen bonded structure of dpmaHCl containing $(dpmaH^+)_2$ dimers and chloride counter anions; a modified constructor graph for a section of the chain is shown.

An alternative arrangement for a *dpma*HX salt with a cation to anion ratio of 1:1 has been reported for *dpma*H[NO₃] [29]. This two-dimensionally connected structure can be understood as the next stage of aggregation (Figure 6a). Again, each dimeric $((dpmaH^+)_2 \text{ unit}$ is connected to four anions and each nitrate anion is connected to two dicationic moieties. Two of the three oxygen atoms of each nitrate anion form hydrogen bonds. The reason for additional structural complexity must be associated with the nitrate counter anion, which obviously causes a larger distance of the $(dpmaH^+)_2$ dimers. Similar hydrogen bonded networks are well known for $(H_2NR_2)_2[MCl_6]$ (R = alkyl, M = Sn, Ir, Pt [13–15,18]) and $((CH_3)_2NH_2)_4[MCl_6]Cl$ (M = Ru, W [30,31]) which can be discussed as an inverse structure type, as the dialkylaminium cations form two hydrogen bonds whereas there are four for each halogenometallate anions.

Finally, the $(dpmaH)_2[PdCl_4]$ salt [32] should be discussed. In this structure, each $(dpmaH^+)_2$ dimer and each $[PdCl_4]^{2-}$ ions form two hydrogen bonded connections to adjacent counter ions (Figure 6b). The chain structure generated by this connection can be understood as a structural motif already known from $dpmaH[NO_3]$ (Figure 6a; blue-shaded area). This is a nice proof of the potential of this system in terms of crystal engineering. The "toolbox" for salts with strong hydrogen-bond acceptors contains dimeric $(dpmaH^+)_2$ units that tends to donate four hydrogen bonds, whereas the counter anions are only twofold connected.



Figure 6. (**a**) the hydrogen bonded layered structure of *dpma*H[NO₃] and the corresponding constructor graph is shown; (**b**) the hydrogen bonded chain structure of (*dpma*H)₂[PdCl₄] and the corresponding modified constructor graph is shown.

3. Limitations

The use of constructor graphs, especially those with the informative icons, become cumbersome for more complex three-dimensional structures. There are similar limitations of use if there are several crystallographically independent building units and individual hydrogen bonding schemes for each of them.

4. Conclusions

In this contribution, it has been shown that the majority of $dpmaH^+$ salts are closely structurally related. Their structural and hierarchical dependencies can easily be visualized by so-called constructor graphs. In contrast to the use of classical constructor graphs which consist of dots and arrows [10,33,34], informative icons and colored arrows are used here for the analysis and comparison of the hydrogen bonded architectures. As abstraction is ubiquitous used in chemistry, biochemistry and related fields, this modification of a mathematical tool towards a chemist-friendly design should broaden the usability of this method. Moreover, the potential of constructor graphs is illustrated by the integration of the structurally related α -aminomethylphosphinic acid into the structure systematology of simple $dpmaH^+$ salts.

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