Deconstruction of Crystalline Networks into Underlying Nets: Relevance for Terminology Guidelines and Crystallographic Databases

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ABSTRACT: This communication briefly reviews why network topology is an important tool (for understanding, comparing, communicating, designing, and solving crystal structures from powder diffraction data) and then discusses the terms of an IUPAC project dealing with various aspects of network topology. One is the ambiguity in node assignment, and this question is addressed in more detail. First, we define the most important approaches: the “all node” deconstruction considering all branch points of the linkers, the “single node” deconstruction considering only components mixed, and the ToposPro “standard representation” also considering linkers as one node but, if present, takes each metal atom as a separate node. These methods are applied to a number of metal−organic framework structures (MOFs, although this is just one example of materials this method is applicable on), and it is concluded that the “all node” method potentially yields more information on the structure in question but cannot be recommended as the only way of reporting the network topology. In addition, several terms needing definitions are discussed.

INTRODUCTION

Well-formulated nomenclature and terminology, thought through with a view to the past, careful considerations of the present, and a clairvoyant eye on the future, are of immense value to scientific development and society, especially in areas where we see emerging technologies. One such area is metal−organic frameworks (MOFs), materials that have attracted considerable attention in recent years particularly because of their actual and potential applications in areas such as gas storage, separation, catalysis, sensors, etc.1,2 They often also have beautiful periodic structures, many previously unknown in chemistry, and the deconstruction of these structures into their underlying nets has been the topic of some discussion.3,4

However, the significance of net topology goes well beyond these types of materials. For example, it was recently suggested,5 and subsequently shown,6 that network topologies are good starting points for screening of possible allotropes of the group 14 elements and related compounds such as silicon carbide. But also hydrogen-bonded systems can be profitably viewed this way,7−10 and many examples of the large group of materials known as Zintl compounds are also well described using network topology.11

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The method also offers a convenient way to classify, name, and put on an equal platform a large number of materials. In the IUPAC 2013 recommendations on the terminology of MOFs and coordination polymers it is stated that The use of topology and topology descriptors to enhance the description of crystal structures of MOFs and 3D-coordination polymers is strongly recommended.\textsuperscript{12}

In this communication, we focus on some of the difficult points when interpreting and communicating a structure in the form of an underlying net. This is indeed one of the topics of a second IUPAC task group,\textsuperscript{13} the members being the coauthors of this article, and we will therefore briefly touch upon other objectives of the project Terminology guidelines and database issues for topology representations in coordination networks, metal−organic frameworks and other crystalline materials.\textsuperscript{14}

A goal that is closely connected is To elaborate recommendations for including the information about network descriptors and topological properties into crystallographic databases. If we want to accomplish this, ambiguities in the vertex (or node) assignment need to be clearly dealt with. How this problem arises, and for which type of compounds, will now be elaborated. The discussion below is mostly centered on MOFs, but relevant also for other types of materials. We will end with a short discussion of the relative merits of the different approaches.

In presenting these results, and discussing their significance, we will by necessity use a number of terms, most of which have no firm IUPAC or IUCr definitions, or where the nomenclature of mathematics and structural chemistry clash. Suggesting such definitions is also part of the task group’s assignment, and a prospective list will be found at the end of the manuscript. Recommended definitions will be forthcoming in the project’s final report to be published in Pure and Applied Chemistry.

Finally, we note that four systems are currently in use for designating network topologies: the RCSR, as provisionally recommended by IUPAC for coordination polymers and MOFs,\textsuperscript{12} ToposPro TTD codes, EPINET codes, and the codes of the International Zeolite Association, comprising zeolite topologies only. Those data have different sources, from empirical to theoretical, and overlap to varying degrees. The IUPAC project will ultimately suggest how these can be best used together for the advancement of chemistry, considering also free access and multiplatform issues.

\section*{RESULTS AND DISCUSSION}

\textbf{Method.} The network topologies discussed in this article were obtained using the freeware programs ToposPro\textsuperscript{15} and

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{figure1.png}
\caption{pcu net formed by idealized hexacyanometallate derivatives, M\textsuperscript{2}[M\textsuperscript{1}(CN)\textsubscript{6}]. Only one network description is possible, and thus the network topology can be unambiguously assigned.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{figure2.png}
\caption{Abstractions of units of MOF structures according to Method 1, “all nodes”. Small spheres: O red, C black. (a) A tritopic carboxylate linker with one branch point (large green balls). The metal Zn\textsubscript{2} SBUs are 3-c (large blue balls) and 4-c (magenta ball). On the right the abstraction with metal nodes red and nonmetal (linker) node black. (b) A tetratopic carboxylate linker with two branch points (large green balls) joined to Cu\textsubscript{2} SBUs corresponding to 4-c nodes (magenta balls). On the right is the corresponding abstraction with linker nodes in black and metal SBU nodes red.}
\end{figure}
SYSTRE\textsuperscript{16} operating on the original crystallographic information files for the compounds in question, or on files derived from these. Throughout we discuss the topologies using the three-letter symbols in the web-based and free Reticular Chemistry Structural Resource database, RCSR,\textsuperscript{17} or, in the absence of such symbols, the ToposPro TTD codes. Some of those network topologies appear also in the theoretical EPINET database.\textsuperscript{18}

**Purpose of Network Analysis and Topology Descriptors.** When discussing cases that are less straightforward, it is worth remembering the main purposes of using this kind of analysis. This has been elaborated at some length, but the main points are that we do this in order to

1. Understand materials synthesized and crystal structures obtained.
2. Compare new materials to literature.
3. Efficiently communicate new materials.
4. Truly make something new by design.
5. Solve crystal structures from powder diffraction data.

We are here mostly concerned with points 1–3.

**Uncomplicated and Unambiguous Network Compounds.** It can be argued that MOFs, or at least coordination...
networks, have been around on a large scale since 1709 when the still commercially available pigment Prussian Blue, approximately Fe₄[Fe(CN)₆]₃, was first marketed.21 Disregarding the crystallographic disorders and defects common for these hexacyanometallates, they can most easily be described as metal ions octahedrally coordinated by six cyanide ions, where the cyanide ions make linear bridges to other octahedral coordination centers. The resulting underlying net, i.e., a network, which bears only the information about connectivity of structural units and is formed by these six-connecting nodes or vertices, is completely unambiguous and called the pcu topology as the vertices in the net correspond to the points of the primitive cubic packing, see Figure 1.

Other examples of network compounds with simple unambiguous topologies include MOF-5, also with the pcu topology; ice-Îc and diamond with the dia topology; and quartz with the qtz topology.

Network Compounds with Ambiguous Topology, a General View. However, MOFs and other compounds formed from polytopic organic linkers, i.e., ligands that can connect more than two metal ions, or similar units, often have particularly complex topologies,20 and a variety of approaches to them have been adopted by different authors, and a given structure is often described in several different ways. This article illustrates these different approaches for a few cases. Our purpose is to provide necessary data for a possible consensus on a preferred mode (or modes) of description, which would be useful for organizing and correlating structural data, now appearing in large quantities (it has been predicted that by 2025 there will be 40 000 MOF structures2). It is thus similar in spirit to an earlier paper on questions of terminology of network materials.21 We should also remark that the following discussion concentrates on MOFs only as a useful example for the issues around network topology assignment for all network based solids.

It is a truism that the structures of chemical compounds often lend themselves to different descriptions, and some may even go as far as questioning the existence of chemical bonds.22 Seldom is any particular one completely incorrect, and different descriptions are useful in different contexts—for example, whether one’s purpose is taxonomy or design methodology. To take a simple example, the structure of the sphalerite form of ZnS can be described, equally correctly, as (a) a binary version of the 4-coordinated diamond net dia, (b) a cubic closest packing of S with Zn in one-half of the tetrahedral holes, or (c) a cubic closest packing of Zn with S in one-half of the tetrahedral holes.

The general goal is to abstract the topology of the structure as an underlying net which is provisionally defined as a periodic simple connected graph.23 The abstract graph is composed of vertices and edges; however in an embedding, as in a crystal structure, we prefer to refer to nodes and links. The question addressed in this paper is how to identify the nodes of the structure. We will see that at least four different ways have been used in the past.

MOFs, which will be our prime examples, consist of at least two components, known as secondary building units (SBUs), a terminology borrowed from the zeolite field. One kind is the organic part; in this article we start by considering MOFs in which the organic component is a polycarboxylate (but which may, however, contain a metal atom in, e.g., porphyrin based linkers). The second kind is the metal-containing part, often a finite or multinuclear coordination entity with a well-defined geometry. The deconstruction generally considers the center of any metal cluster to be a single node of the net, but individual atoms may also be considered, as in one approach we will discuss. For simplicity, in the first part of this article we consider only MOFs with paddlewheel SBUs with either three or four carboxylate carbon atoms acting as points of extension (points of connection to the organic linker). Examples are illustrated in Figure 2.

Different Approaches to Node Assignment. In a review of the structures of MOFs with finite metal SBUs and with polytopic linkers, each metal SBU was considered as one node, and all branch points of the linkers were used as nodes.20 We refer here to this approach as the “all node” deconstruction, Method 1 (for earlier examples of this see refs 8, 24, and 25). Another common mode of deconstruction is to take metal SBUs and polytopic linkers as just one node each. We refer to that as the “single node” deconstruction, Method 2. A variation on this is the “standard representation” of the program TopsisPro,25 which again takes organic linkers as one node but considers also each metal atom as a separate node—this is Method 3. TopsisPro also has a “cluster representation” mode which may produce several topologies. The one with the most nodes will be the same as the “all node” deconstruction (Method 1), but there may be others in which groups of SBUs

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are linked into larger groups called tertiary building groups (TBUs) or supramolecular building blocks (SBBs) and represented by one node; this is Method 4. The “standard representation” (Method 3) is chosen by default in ToposPro because in this case the decomposition is unambiguous and can be performed with a strict algorithm, also for the MOFs with infinite (e.g., rod-like) coordination entities, allowing automatic classification of any kind of periodic structure.

For convenience we refer to a node with \( k \) links as \( k \)-c (for \( k \)-coordinated), nets with \( k_1, k_2, \ldots \)-coordinated nodes are referred to as \( (k_1, k_2, \ldots) \)-c. In what follows nets are identified by the RCSR three-letter symbols such as \( xyz \). To illustrate the nets we generally use the augmented version, symbol \( xyz-a \), in which the vertices of the original net are replaced by the coordination (vertex) figure.

In a review of structures with polytopic linkers, a distinction was made between basic nets and derived nets. The basic nets are the so-called default nets that are preferred for linking single shapes (e.g., tetrahedra) or pairs of shapes (e.g., triangles and squares). They generally have just one kind of link (in the jargon edge-transitive nets), and those relevant to MOF structures have been systematically derived and described. Derived nets are obtained from basic nets by splitting vertices into groups of vertices of lower coordination—e.g., for example, tetrahedrally coordinated vertices into a pair of triangularly coordinated vertices.

A key finding in ref 20 was that Method 1, the “all node” approach, was able to distinguish topological (and therefore supramolecular) isomers in the case of a number of coordination polymers of tetracyanoquinodimethane (TCNQ). The easiest way to understand this is to consider that once a polytopic 4-c node, see Figure 2b, has been split into two 3-c nodes it will have direction.

### Table 2. Nets Assigned to Two Different MOFs by the Four Methods Described in the Text

<table>
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<th>method</th>
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<th>3</th>
<th>4</th>
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<td>single node</td>
<td>ToposPro standard</td>
</tr>
<tr>
<td>Eddaoudi</td>
<td>Figure 4e</td>
<td>ntt</td>
<td>ntt</td>
<td>( 3.3.4.6T26^a )</td>
</tr>
<tr>
<td>Lah</td>
<td>Figure 4f</td>
<td>ntt</td>
<td>4.6T4(^{17} )</td>
<td>4.12T1(^{17} )</td>
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\(^{a}\)non-crystallographic net.
A two-dimensional (2D) example would be a square planar metal ion coordinating a tetratopic ligand giving the square grid net sq1 if the ligand is considered as a 4-c node. However, depending on the orientation of these ligands, two different 2D arrangements are possible, and if the ligand is split into two 3-c nodes the new topologies will be the bex-net or the mcm-net (a.k.a. the Cairo tiling), recognizing these two supramolecular structure isomers, see Figure 3.

Network Compounds with Ambiguous Topology, Three Specific Examples. We now consider three groups of MOFs. The abstraction of combinations of linker and metal SBUs is illustrated schematically in Figure 2 for two examples. Other topologies involving just one organic linker are shown in Figure 4.

MOFs with the Basic Net nbo. Here we consider three MOFs with tetratopic carboxylate organic units linking square metal SBUs as illustrated in Figure 2b. They are MOF-50548 (LASYOU, https://dx.doi.org/10.5517/cc8mxh8), JUC-6255 (OFOCUL, https://dx.doi.org/10.5517/cc8mfm3), and DUT-4956 (ACOCOM, https://dx.doi.org/10.5517/ccvmw9). If the linker is considered one 4-c node, then in each case the net is nbo. However, considering the nets in which the linker is described as two 3-c nodes in each case the pattern of linkers is different, so the derived (3,4)-c nets are distinctive from each other and with symmetries different from that (Im3m) of the basic net from which they are derived. The first two derived nets, fof and fog (see Figure 5), both have symmetry R3m and generally have not been distinguished even though they are quite different topologically.56 In the third of these derived nets, tfb symmetry Fm3m, a TBU (or SBB) can be identified—this is a cluster of six paddlewheels at the vertices of an octahedron as in the metal–organic polyhedron (MOP) MOP-28.57 (QAV-BOF, https://dx.doi.org/10.5517/cc9p8rz) Each TBU is linked to 12 others with the fcu topology as indicated in Table 1. The same topology is also found in MOFs with a different 4-c metal SBU; in this case both the nbo and fcu descriptions were used.27

Recently an MOF (MMCF-2, NOBWEI, https://dx.doi.org/10.5517/cc1016f) was obtained, in which 4-c paddlewheels were linked by a tetratopic linker with just one 4-c branch point.58 Now the “all node” topology is indeed nbo as shown in Table 1.

Actually, in every case the nbo net has two kinds of nodes (as in the chemical compound NbO!), and it might be advantageous to recognize this as the “coloring” of the vertices of the original net to reduce the symmetry. In fact RCSR has an entry nbo-b for this binary version for the express purpose of reporting the symmetry (Pm3m rather than Im3m of the original).

In the ToposPro “standard” representation both metal atoms of the paddlewheel are considered as nodes of the net which is then (4,8)-c. This net has a ToposPro symbol 4,8T24. It has non-crystallographic (local) symmetries as discussed elsewhere.59

MOFs with the ntt Underlying Net. Here we consider two MOFs with the related topologies that were the first of what is now a very large family of MOFs. The first, which we label “Eddaoudi” (LIZWEX, https://dx.doi.org/10.5517/ccqzm8), has two distinct metal SBUs and a tritopic linker.40 The second, which we label “La”, has a hexatopic linker joined to 4-c paddlewheel SBUs (SIZPUN https://dx.doi.org/10.5517/ccqbwfb, MUDTAH https://dx.doi.org/10.5517/ccrqlrs, MUDTEL https://dx.doi.org/10.5517/ccsx41)41,42 Figure 4ef shows the topology of the basic unit in each case. The ratio of 3-c nodes to 4-c nodes is now 8:3 (compared to 2:1 for the nets of the previous section). The structure is notable for containing a TBU with 12 paddlewheels at the vertices of a cuboctahedron as in MOP-1.35,34 This TBU is connected to 24 3-c nodes, see Figure 6, and both authors identified the topology as (3,24)-c (RCSR symbol rht). The Eddaoudi group also noted that the metal SBUs and linkers formed a trinodal (3,4)-c net, now assigned the RCSR symbol ntt. The dozens of subsequent papers on isoreticular MOFs almost invariably refer to the topology as rht.

Interestingly, in contrast to the case with tetratopic linkers in which the most common description is with one 4-c node for the linker, as far as we know, in the compounds with hexatopic linkers, the linker is never described by a single 6-c node. However, if this is done, a non-crystallographic net is obtained.20 Even so, the fact remains that this structure type is variously described as a uninodal, binodal, or trinodal net!

MOFs with the tfc Underlying Net. The MOFs of this section have linkers of different size ranging from tritopic to octatopic. What they have in common is the same net if all branch points of the linkers are counted as nodes and a ratio of 3-c to 4-c nodes of 20:3. As we shall see, there are two kinds of 3-c nodes, say A and B, and one kind of 4-c node C with A/B/C = 12:8:3.

The first MOF which we identify as “Zaworotko” (EBUCIN https://dx.doi.org/10.5517/cc5849v) has the tritopic linker of Figure 2a.45 To count the relative number of nodes note that there is one 3-c node at the center of the linker and 2/3 more 3-c nodes at the periphery. Likewise for the same unit there is 1/4 of a 4-c node. There are intrinsically at least three kinds of nodes: one for the linker and two for the two different metal SBUs, and indeed the underlying (3,4)-c net, tfc, is tritopic. As there is just one branch point in the linker, the “single node” deconstruction gives the same net.

The structure identified as “MMMF-5” (VEJHID, https://dx.doi.org/10.5517/ccxyyj9) has an octatopic linker, shown schematically in Figure 4d, joined to eight 3-c SBUs.46 Now, in contrast to the previous case there are two organic branch points in the linkers and one kind of inorganic SBU, but the “all node” deconstruction gives the same (3,4)-c net tfc. This phenomenon is familiar in inorganic chemistry as “structure” and “antistructure” —for example, CaF₂ has the fluorite structure (net flu), and Li₂O has the antistructure with the roles of cation and anion reversed: in “Zaworotko” the metal

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<th>MOF</th>
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<tr>
<td>Hong</td>
<td>Figure 4g</td>
<td>tfc</td>
<td>hmc</td>
<td>3,4,12T3⁴⁴</td>
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</tbody>
</table>

*non-crystallographic net.*

Table 3. Nets Assigned to Three Different MOFs by the Four Methods Described in the Text

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SBU is 4-c, while in “MMPF-5” the SBU is a single metal 3-c and one branch point of the ligand is 4-c. Having a single metal node and a octatopic linker, the “single node” deconstruction now gives the (3,8)-c net the, which is also equivalent to the ToposPro standard representation.

In the structure identified as “Hong” (GUKQUZ, https://dx.doi.org/10.5517/ccs4j5l) a hexatopic linker, shown in Figure 4g, is linked to three 3-c SBUs and to three 4-c SBUs.47 Now there are four kinds of nodes: two 3-c linker points and the 3-c and 4-c metal SBUs. However, two of the nodes are topologically the same, and the “all nodes” description gives the same trinodal tfe net. One kind of 3-c node identified earlier, B1, is now split into two kinds, B1 and B2, with now A/B1/B2/C = 12:4:4:3. To fully describe this situation, RCSR includes the version tfe-b with symmetry reduced from Pm3m to Fm3m with a’ = 2a. See below for “colored” nets. The “single node” deconstruction now gives the (3,4,6)-c net hmc with the metal SBU as 3 and 4-c nodes and the ligand 6-c. Note that the “TBU” deconstruction gives the same rht net as for the previous MOFs (which are commonly called “rht MOFs”).

THE RELATIVE MERITS OF THE DIFFERENT APPROACHES

As noted earlier the most important point in reporting network topologies is clarity. Having names for the different approaches helps, but preferably this should be accompanied by a diagram showing the node assignment.

Comparing the different methods we also see that the “all nodes” approach has several merits but it cannot be recommended to give only that topology; the “parent” topology also needs to be stated; i.e., for MOF-505 one should write “the nbo-derived net fofo”. A tentative suggestion follows below.

A ligand that bridges more than one node, known as a polytopic ligand, will also be a node. Such ligands may also be split into two or more nodes, and it is recommended that this is done if

(a) The topology description is improved; i.e., possible network topology isomers may be differentiated.
(b) The physical description is improved, i.e., how well the network follows the actual alignment of molecules in the structure.

LIST OF TERMS

Finally, we present here a list of terms needing definitions. We do not want to propose or discuss any intermediate ideas here, to avoid future confusion, but merely state which terms we find important and currently having no unambiguous definition.

Polytopic bridging ligand; net (problems are compatibility with mathematic terminology and the possible need for 2-c nodes for catenated nets); SBU; basic net; derived net; transitivity; edge transitive nets; isoreticular; TBU; periodic simple connected graph; augmented net

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Notes

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