Paul Niggli's contribution to stereochemistry

by *Josef Zemann**

The influence of Paul Niggli on modern crystallography was recognized not only in numerous obituaries, but also e.g. in the article "In memoriam: Paul Niggli's crystallographic œuvre" (LAVES and A. NIGGLI, 1964) and in the book "Die Symmetrie der Kristalle" (BURCKHARDT, 1988). Today he is well known for at least three important contributions: (1) He presented the 230 classical space groups in a form more convenient to the average X-ray crystallographer than the original ones, e.g. the one by SCHOENFLIES (1891) which was of great influence to Niggli, (2) he very early recognized the importance of a space group determination from systematic X-ray extinctions prior to the structure determination, and (3) he introduced to the theory of space groups the concept "Gitterkomplex" [lattice complex] as the equivalent of the crystallographic form in morphological crystallography. All three items are first thoroughly dealt with in his famous book "Geometrische Kristallographie des Diskontinuums" (NIGGLI, 1918–1919). P. Niggli continued to work on these topics also later on, as is seen from many articles in scientific journals, from the volume "Krystallographische und strukturtheoretische Grundbegriffe" (NIGGLI, 1928), his activities with the "Internationale Tabellen zur Bestimmung von Kristallstrukturen" (NIGGLI and BRANDENBERGER, 1935), the three editions of his "Lehrbuch der Mineralogie" – the third edition as "Lehrbuch der Mineralogie und Kristallchemie"[1] – (NIGGLI, 1920–1945) and his book "Grundlagen der Stereochemie" (NIGGLI, 1945).

Because of their general recognition, these three topics will not be discussed here much further. Instead, consideration will be given to some of Niggli's contributions to stereochemistry.

Of great interest are his very early papers in this field: in part for historical reasons, in part also because they give the background to his later work. His first two articles on crystal structures (NIGGLI, 1915, 1916) belong closely together. A great role play there "kristallonomisch einfache Punkte" [crystallonomic simple points], i.e. points in the unit cell having simple coordinates when measured with the cell lengths as units, e.g. 0 and $\frac{1}{2}$, but also $\frac{1}{4}$ and other simple values. Their connecting lines are called "kristallonomisch wichtige Richtungen" [crystallonomic important directions]. From the very few crystal structures known at that time, P. Niggli concluded that crystallonomic simple points are occupied either by atoms (ions) or by centers of gravity of "elementare Baugruppen" [elementary building units], like S_2 dumb-bells, CO_3 groups, etc.

Here two examples in modern notation: The atomic arrangement of NaCl can be described in space group O_h^5 – Fm3m with the Na's on position 4a 0,0,0 etc. and the Cl's on position 4b $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ etc.; all particles occupy crystallonomic simple points. – The structure of pyrite, FeS_2, is slightly more complicated. It can be described in space group T_h^6 – Pa3, with the Fe's on position 4a 0,0,0 etc. and the S's on position 8c x,x,x etc. From the experimental value for x, each two S atoms form S_2 dumb-bells, the centers of which are located on position 4b $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ etc. This means that in pyrite the Fe's and the centers of the S_2 groups occupy crystallonomic simple points, and Niggli considers those to be more important for the structure than the loca-

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[1] The complete set of copies of "Teil III" of the 3rd edition was destroyed in World War II before delivery. The author of this article had the privilege to be given a microfiche copy of the proof sheets.

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tions of the S atoms themselves; e.g. he considers as crystallonomic important directions not the lines connecting the Fe's with the S's, but rather the lines connecting the Fe's with the centers of gravity of the S_2 dumb-bells.

With the usual choice of the origin of the unit cell at (whenever possible) a highly symmetric position, also the other crystallonomic simple points will, as a rule, have high symmetry - in several typical cases even the same symmetry as the origin. From the experience at that time, Niggli considered structures with the atoms (ions) on such points to be of special importance to stereochemistry. In NIGGLI (1915) he derives all possible AB structure types with one atom at the origin and the other ones on crystallonomic simple points of first order[2].

He showed that altogether four such structures are theoretically possible. Only two of them were already known, i.e. the structure types of halite, NaCl, and of sphalerite, ZnS (BRAGG, 1913, 1914). The other two were unknown in 1915 among actual crystals. The one of them is a cubic structure with the A atom at the corners and the B atom at the center of the cell, or vice versa (Fig. 1). It corresponds to the CsCl structure, but this was first determined only some years later (DAWEY and WICK, 1921). The other one can be described as two congruent diamond-type structures that are mutually shifted by half of the space diagonal of the unit cell. Thereby, one obtains an atomic
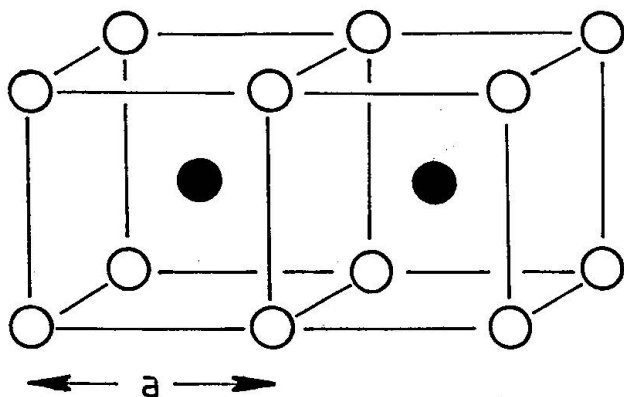

Fig. 1   Two unit cells of the CsCl structure type.

Fig. 2   One quarter of the unit cell of the NaTl structure type.

arrangement in which each A is located at the center of a cube with edge length $a/2$, of which four corners are occupied by A's and four corners by B's, and vice versa (Fig. 2). From this stereochemical feature, Niggli concluded that the second structure would be only little stable[3]. A representative was found only much later, when ZINTL and DULLENKOPF (1932) made an X-ray investigation of NaTl, an intermetalic phase very sensitive to air and to water.

This is an early example for theoretically deriving structures with special constraints. Niggli devoted later much time on related problems, e.g. with "homogene" and "heterogene Bauverbände". He was a pioneer in this important theoretical field of stereochemistry.

Other interesting problems are dealt with in Chapter IV of "Geometrische Kristallographie des Diskontinuums" (NIGGLI, 1918-1919). He recognizes there clearly that for simple crystal structures (with particles on highly special positions only) the space group is not unequivocally defined by the mere knowledge of the positions of the centers of gravity of the atoms (ions), unless they have very high symmetry. He discusses this extensively for the NaCl structure type. The atomic arrangement as determined by BRAGG (1913, 1914) corresponds to space group O_h^5-Fm3m, and this is also the true space group if the Na's and Cl's have sufficient high symmetry, i.e. symmetry O_h-m3m or spherical symmetry. With the assumptions that the cubic cell contains $4 \times$ NaCl and that Na's and Cl's occupy fourfold positions, he

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[2] For the term "order" of crystallonomic simple points see NIGGLI (1915), p. 369. - Using a later nomenclature (NIGGLI and LAVES, 1930) one would say that the A's as well as the B's build "systemssymorphe Raumgitter", i.e. they form cubic lattice complexes with point symmetries corresponding to the crystal system.
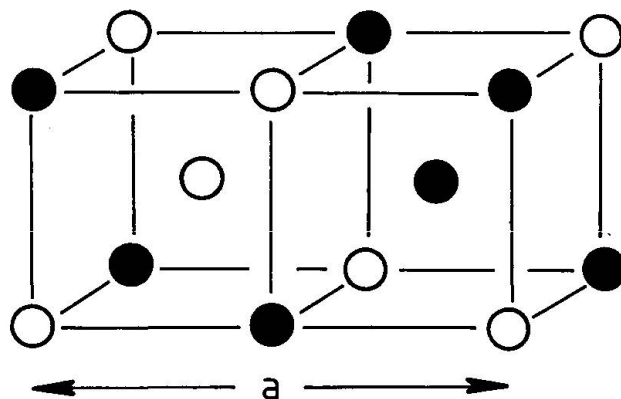
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[3] This is supported for ionic structures by the Madelung constant $\alpha = 1.5134$, as compared with $\alpha = 1.7627$ for the CsCl type.

systematically derives the possible space groups. Candidates are the 14 cubic space groups that have either two nonvariant four-fold positions or (at least) one monovariant fourfold position (Tab. 1). But only in 12 of them one can build the atomic arrangement as determined for NaCl; in the remaining two, O^6-$P4_332$ and O^7-$P4_132$, the (nonvariant) fourfold positions cannot be used for this purpose, because they are not F lattice complexes. As the 12 suitable space groups are distributed over all five cubic crystal classes, Niggli finds that structures for which the centers of gravity of the particles have the NaCl-type arrangement can in principle belong to any of the five cubic crystal classes, provided that the particles have a shape with symmetry lower than O_h-m3m.

In the same chapter Niggli also realizes that even for spherical particles one cannot definitely exclude for NaCl type structures a symmetry lower than O_h^5-Fm3m. To see that, consider space groups T^1-P23, T^4-$P2_13$ and T_d^1-P43m. In each of them there exists a univariant fourfold position (Tab. 1), and by occupying it with the Na's and Cl's and further choosing appropriate special values for the parameter X, one can exactly build the atomic arrangement of NaCl in each of them. Deviations from the special x-parameters violate symmetry O_h^5-Fm3m, but if they are suffiently small they cannot be detected with X-ray methods.

A third remark on NaCl-type compounds there concerns possible multiple lattice constants. Niggli states that from the limited accu-

Tab. 1 The fourfold positions in the cubic space groups (in modern notation.)

T^1 - P23 (No. 195) Origin at 23

4e 3 x,x,x; $x,\bar{x},\bar{x}$; $\bar{x},x,\bar{x}$; $\bar{x},\bar{x},x$

T^2 - F23 (No. 196) Origin at 23

4a 23 $0,0,0$; $0,\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$
4b 23 $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,0$; $0,\frac{1}{2},0$; $0,0,\frac{1}{2}$

T^4 - $P2_13$ (No. 198) Origin at 3

4a 3 x,x,x; $\frac{1}{2}+x,\frac{1}{2}-x,\bar{x}$; $\bar{x},\frac{1}{2}+x,\frac{1}{2}-x$;

$\frac{1}{2}-x,\bar{x},\frac{1}{2}+x$

T_h^2 - Pn3 (No. 201)

4b $\bar{3}$ $\frac{1}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{4},\frac{3}{4},\frac{3}{4}$; $\frac{3}{4},\frac{1}{4},\frac{3}{4}$; $\frac{3}{4},\frac{3}{4},\frac{1}{4}$
4c $\bar{3}$ $\frac{3}{4},\frac{3}{4},\frac{3}{4}$; $\frac{3}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{4},\frac{3}{4},\frac{1}{4}$; $\frac{1}{4},\frac{1}{4},\frac{3}{4}$

T_h^3 - Fm3 (No. 202) Origin at m3

4a m3 $0,0,0$; $0,\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$
4b m3 $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,0$; $0,\frac{1}{2},0$; $0,0,\frac{1}{2}$

T_h^6 - Pa3 (No. 205) Origin at $\bar{3}$

4a $\bar{3}$ $0,0,0$; $0,\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$
4b $\bar{3}$ $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,0$; $0,\frac{1}{2},0$; $0,0,\frac{1}{2}$

O^2 - $P4_232$ (No. 208) Origin at 23

4b 32 $\frac{1}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{4},\frac{3}{4},\frac{3}{4}$; $\frac{3}{4},\frac{1}{4},\frac{3}{4}$; $\frac{3}{4},\frac{3}{4},\frac{1}{4}$
4c 32 $\frac{3}{4},\frac{3}{4},\frac{3}{4}$; $\frac{3}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{4},\frac{3}{4},\frac{1}{4}$; $\frac{1}{4},\frac{1}{4},\frac{3}{4}$

O^3 - F432 (No. 209) Origin at 43

4a 43 $0,0,0$; $0,\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$
4b 43 $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,0$; $0,\frac{1}{2},0$; $0,0,\frac{1}{2}$

O^6 - $P4_332$ (No. 212) Origin at 3

4a 32 $\frac{1}{8},\frac{1}{8},\frac{1}{8}$; $\frac{5}{8},\frac{3}{8},\frac{7}{8}$; $\frac{7}{8},\frac{5}{8},\frac{3}{8}$; $\frac{3}{8},\frac{7}{8},\frac{5}{8}$
4b 32 $\frac{5}{8},\frac{5}{8},\frac{5}{8}$; $\frac{1}{8},\frac{7}{8},\frac{3}{8}$; $\frac{3}{8},\frac{1}{8},\frac{7}{8}$; $\frac{7}{8},\frac{3}{8},\frac{1}{8}$

O^7 - $P4_132$ (No. 213) Origin at 3

4a 32 $\frac{3}{8},\frac{3}{8},\frac{3}{8}$; $\frac{7}{8},\frac{1}{8},\frac{5}{8}$; $\frac{5}{8},\frac{7}{8},\frac{1}{8}$; $\frac{1}{8},\frac{5}{8},\frac{7}{8}$
4b 32 $\frac{7}{8},\frac{7}{8},\frac{7}{8}$; $\frac{1}{8},\frac{3}{8},\frac{5}{8}$; $\frac{5}{8},\frac{1}{8},\frac{3}{8}$; $\frac{3}{8},\frac{5}{8},\frac{1}{8}$

T_d^1 - P43m (No. 215) Origin at 43m

4e 3m x,x,x; $x,\bar{x},\bar{x}$; $\bar{x},x,\bar{x}$; $\bar{x},\bar{x},x$

T_d^2 - P43m (No. 216) Origin at 43m

4a 43m $0,0,0$; $0,\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$
4b 43m $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,0$; $0,\frac{1}{2},0$; $0,0,\frac{1}{2}$

O_h^4 - Pn3m (No. 224) Origin at 43m

4b 3m $\frac{1}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{4},\frac{3}{4},\frac{3}{4}$; $\frac{3}{4},\frac{1}{4},\frac{3}{4}$; $\frac{3}{4},\frac{3}{4},\frac{1}{4}$
4c 3m $\frac{3}{4},\frac{3}{4},\frac{3}{4}$; $\frac{3}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{4},\frac{3}{4},\frac{1}{4}$; $\frac{1}{4},\frac{1}{4},\frac{3}{4}$

O_h^5 - Fm3m (No. 225) Origin at m3m

4a m3m $0,0,0$; $0,\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$
4b m3m $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,0$; $0,\frac{1}{2},0$; $0,0,\frac{1}{2}$

To obtain the NaCl structure type, one has to choose in space groups T^1-P23 and T_d^1-P43m x(Na) = $\frac{1}{4}$, x(Cl) = $\frac{3}{4}$ (or vice versa), in space group T^4-$P2_13$ x(Na) = 0, x(Cl) = $\frac{1}{2}$ (or vice versa).

racy of X-ray diffraction work, the true lattice constant of a NaCl-type compound could possibly also be a multiple of the generally accepted one. He discusses the problem theoretically for spherical particles and crystal class $O = 432^4$. From Tab. 1 follows that no solutions exist when using the usual lattice constant and cell content, because in the two space groups deserving consideration, i.e. O^2-P4_232 and O^3-F432, the fourfold positions are nonvariant (Tab. 1). A doubling of the lattice constant causes that the new cell contains $32 \times AB$. But in neither of the two space groups of class $O-432$ with 32fold positions, i.e. O^3-F432, 32f x,x,x etc. and O^4-F4_132, 32e x,x,x etc., is it possible to build the atomic arrangement of NaCl by using the position indicated. Niggli, however, finds solutions of the problem by using not the 32fold positions, but rather several positions of lower multiplicity. One possibility is: space group O^1-P432 with 32 A on 1a + 1b + 3c + 3d + 12h + 12i, and 32 B on 6e + 6f + 8g + 12h. Several positions are monovariant, and one has to choose special values for the x's. But if one or several of the x values deviate from the required special value, the symmetry is lowered to class $O-432$ (and the lattice constant is doubled as compared with that of the NaCl-type).

The last three remarks are all related to the influence of the limited accuracy of X-ray measurements to possible errors in the derived crystal structure. The problem was, of course, much more serious when P. Niggli wrote his "Geometrische Kristallographie des Diskontinuums" than it is today – but it has not disappeared. If the symmetry of a crystal structure should be lowered in comparison to that indicated by the centers of gravity of its atoms (ions) only because the symmetries of the electron shells were slightly lower than the symmetry of the respective position, this could also now hardly be proven by X-ray methods. Further: Also today is it a difficult problem in crystal structure research to determine the detailed geometry of an atomic arrangement if it deviates only very slightly from higher symmetry, or from a geometry that can be described in a smaller cell. The detailed consideration of

weak classes of extra-refections is sometimes of great help; but in spite of that the problem remains often to be a very difficult one.

Of the many other stereochemical problems in which Niggli was engaged, only two will shortly be mentioned here.

The one concerns shorthand formulas for crystal structures. It is well known that the listing of the unit cell of a crystal structure, together with the coordinates of all the atoms in the cell, contain the complete information for the classical stereochemical discussion of this atomic arrangement. However the bond lengths, bond angles, and the kinds of mutual coordination are not immediately seen, and therefore a shorthand notation of the "important" features of a crystal structure is often very useful.

Already in his very early papers, NIGGLI (1915, 1916) is well aware of that. He uses, e.g. the following notations: sylvite – [K,6Cl];[Cl,6K]; sphalerite – [Zn,4S];[S,4Zn]; fluorite – [Ca,8F];[F,4Ca] to indicate the mutual surroundings (first coordination spheres). For more complicated structures he does not note the shortest interatomic distances, but rather the distances between the centers of gravity of the elementary building units; e.g. he uses:

pyrite – $[Fe,6S_2];[S_2,6Fe]$;

calcite –

$[Ca(6Ca)2[CO_3]];[[CO_3](6[CO_3])2Ca]$.

Later he modified the notation in several ways. In NIGGLI (1933) he explicitely states that constitutional formulas cannot present the complete atomic arrangement in space and the cases of isomerism; but two important properties can be expressed: (1) the formation of radicals, i.e. of more closely connected subunits of the structure, and (2) the coordination numbers – especially those of the "first sphere". In the same paper, Niggli gives constitutional formulas for some more complicated structures, e.g. for diopside, $CaMg(Si_2O_6)$, in which all close cation-anion contacts (in the example with the exception of Ca) are presented with the topologically correct connections. Such formulas become necessarily clumsy, and in this later books (NIGGLI, 1945a and b) he does not use them much further.

P. Niggli's attitude toward the concept of atomic and ionic radii is highly interesting. Contrary to many prominent workers in the

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[4] In the opinion of the present author, crystal class O was chosen as an example because at that time KCl, a NaCl-type compound, was widely believed to crystallize in this class (cf. e.g. GROTH, 1906).

field, like V.M. Goldschmidt, L. Pauling and W. Zachariasen, he does not consider it with too much sympathy. This is clear from passages like this: "Es gibt weder einen dem elementaren Zustand zukommenden Wirkungsbereich, noch einen dem Ion zugehörigen Radius". [There exists neither a specific effective size to be attributed to the atomic state, nor a radius of the ion. Transl. by the author.] (NIGGLI, 1930). His great reservation seems to have two roots: (1) He is well aware that the interatomic distances between the same pair of atoms (ions) are not at all constant in different structures, but vary in part considerably so that "hard spheres"cannot be an appropriate model, and (2) he attributes a relatively great role in the stereochemical behaviour of the single particles to their specific "shapes" - especially so in his early papers.

In the late twenties and in the thirties and forties of our century, rigid spheres played a greater role as models of atoms and ions than today. Their radii were considered to be essential not only for the prediction of interatomic (-ionic) distances but also for the understanding of the mutual coordinations. And as the concept was applied with considerable success, Niggli was left with his criticism much alone. Today the points of view have come closer together. It is now clear to every stereochemist (and, as a matter of fact, it was already well known in the late twenties) that an atom or ion cannot be adequately described as a hard sphere. Even for the atoms of the inert gases and for the ions with an inert-gas electron shell any somewhat realistic model needs for the description of the spherically symmetric particles at least *two* constants: the one somehow indicating the size, and the other giving a measure of the compressibility. In more complicated cases, e.g. for the transition metal ions Cu^{2+}, Mn^{3+}, Cr^{3+}, ... also the assumption of a spherical symmetry has to be left (crystal field theory, Jahn-Teller effect).

The study of P. Niggli's publications can be heartily recommended also to the stereochemists of our days, and that not only for historical reasons. It is true that P. Niggli's publications are not always easy to read, and to the international community it is a handicap that practically all his papers are written in German. But the labour will be rewarded. Just to mention one topic: His many publications on theoretically possible structure contain a wealth of interesting, useful and little known information, and several of his results were later rediscovered by authors who did not recognize that their results could already be found somewhere in the writings of Paul Niggli.

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For a complete list of Paul Niggli's publications
the reader is referred to the compilation by J. Mar-
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