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A re-interpretation of the crystal structure of ' $(\text{NH}_4)_2(\text{NH}_3)[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ ' in terms of $(\text{NH}_4)_{2-2z}[\text{Ni}(\text{NH}_3)_2]_z[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$

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Abstract

A re-interpretation and re-evaluation of single-crystal X-ray diffraction data of a previously reported ' $(\text{NH}_4)_2(\text{NH}_3)[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ ' (J. Solid State Chem. 162 (2001) 254) give a new formula $(\text{NH}_4)_{2-2z}[\text{Ni}(\text{NH}_3)_2]_z[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ with $z = 0.152$. This new formula results from defects in an idealized ' $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ ' basic structure, where two adjacent NH_4^+ cations are replaced by one $\text{Ni}(\text{NH}_3)_2^{2+}$ unit. Cl^- anions from the basic structure complete the coordination sphere of the new Ni^{2+} to $[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]^{2-}$.

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1. Introduction and structural considerations on the title compound

Recently, preparation and crystal structure of $(\text{NH}_4)_2(\text{NH}_3)[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ (Fig. 1a, in the following called the *title compound*) were reported [1]. The fractional coordinates and occupancies obtained by refinement of a structural model on the basis of X-ray diffraction data from a single crystal are listed in Table 1. Neglecting the partially occupied N3 site, and considering only the sites of the remaining fully occupied non-H atoms (H atoms were not considered in the refinement reported in Ref. [1]) leads to an idealized ' $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ ' basic structure which is isotypic with $(\text{NH}_4)_2\text{MnCl}_4 \cdot 2\text{H}_2\text{O}$ or $(\text{NH}_4)_2[\text{Mn}(\text{H}_2\text{O})_2\text{Cl}_4]$ [2]. The crystal structure of the *title compound* can then be described as a tetragonally distorted anti-fluorite type arrangement of isolated *trans*- $[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]^{2-}$ anions (Ni1, N2 and Cl, cf. Table 1) and NH_4^+ cations (N1). The neglected N3 site resulted from difference electron density which was attributed to non-complexing NH_3 (N3) molecules in interstices (cf. Fig. 1).

This interstitial NH_3 (N3) molecule constitutes a 'problem' [1] of the crystal structure model of the *title compound* (cf. Fig. 1a). N3 has two N1 atoms (NH_4^+) at a distance of only 1.99 Å as its closest neighbors. These distances are considerably too small. Non-complexing NH_3 molecules are a rarely observed structural feature of ammines of simple salts, in particular for such which were prepared at high temperatures (the *title compound* was prepared at 350°C). Ammines with non-complexing NH_3 molecules have mostly a very high molar ratio NH_3/metal and a high NH_3 equilibrium pressure. One example which may serve here for comparison is $[\text{Al}(\text{NH}_3)_6]\text{I}_3 \cdot \text{NH}_3$ [3]. In its crystal structure a non-complexing NH_3 molecule is surrounded quite irregularly by five I atoms and seven NH_3 groups. The shortest N...N distance of that 'interstitial' NH_3 to another NH_3 is 3.13 Å. For this contact hydrogen bonding was discussed, but this distance is still considerably longer than the distance $\text{N3}(\text{NH}_3)\cdots\text{N1}(\text{NH}_4)$ reported of 1.99 Å for *title compound*.

2. Considerations leading to a new interpretation of the N3 site

A new structural interpretation of the electron density of N3 belonging to an ammonia molecule [1] in the *title*

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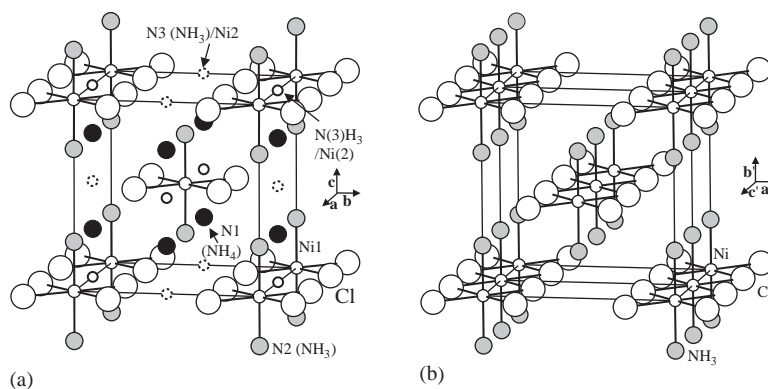


Fig. 1. (a) Crystal structure of the *title compound*, $(\text{NH}_4)_2(\text{NH}_3)[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$, $I4/mmm$, which is reinterpreted here in terms of $(\text{NH}_4)_{2-2z}[\text{Ni}(\text{NH}_3)_2]_z\text{Ni}(\text{NH}_3)_2\text{Cl}_4$ with $z = 0.152$. The N3 atoms from Ref. [1] on the $4c$ Wyckoff site, reinterpreted here as Ni2 are indicated by two different symbols for two halves. If one half of the $4c$ sites is occupied by Ni2 and all NH_4^+ are replaced by NH_3 (cf. Fig. 2) one yields the crystal structure of $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ (b) crystallizing in the orthorhombic ($Cmmm$) $\text{Cd}(\text{NH}_3)_2\text{Cl}_2$ type structure. Note that $c' = a/2$.

compound can be obtained when the orthorhombic crystal structure of $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ [4] is considered. $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ crystallizes in the $\text{Cd}(\text{NH}_3)_2\text{Cl}_2$ type structure ([5], cf. Fig. 1b), and like for the *title compound*, the coordination octahedron around nickel is *trans*- $[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ with similar interatomic distances. However, in contrast to the isolated octahedra $[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ in the idealized basic structure $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ of the *title compound*, in $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ these octahedra share common edges to form chains ${}_{\infty}^1[\text{Ni}(\text{NH}_3)_2\text{Cl}_4/2]$. The unit cell volume of the *title compound* (472.0 \AA^3) has approximately twice the volume of $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ (235.2 \AA^3).

Starting now from the idealized basic structure $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ of the *title compound*, one can obtain the $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ structure by (i) subdividing the $4c$ sites (on which N3 was placed in Ref. [1]) into two equivalent halves. Then, (ii) Ni^{2+} ions (Ni2) are inserted on one half of the $4c$ sites (cf. Fig. 1) and simultaneously all NH_4^+ ions (N1) are replaced by NH_3 molecules which are then able to coordinate the new Ni2. The other half of $4c$ sites remains empty. If this replacement is performed completely and when small shifts of N and Cl are allowed for, all Ni (Ni1 and Ni2) and all N (N1 and N2) atoms become equivalent. This results in the new unit cell parameters $c' = a/2$, $a' = b$ and $b' = c$, where a , b and c are the unit cell parameters of the *title compound* ($I4/mmm$) and a' , b' and c' are those of $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ ($Cmmm$).

This structural relation between $\text{Ni}(\text{NH}_3)_2\text{Cl}_2$ and idealized $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ leads to a new structure model for the *title compound*. One may imagine that in the *title compound* the insertion of Ni and transformation of NH_4 to NH_3 had occurred only partially. It is likely that such a partial insertion of Ni2 occurs without preference for any of the two halves of $4c$ sites. Hence, the structure remains tetragonal. However, locally two

$4c$ sites separated by $[00\frac{1}{2}]$ cannot be occupied simultaneously, because in that case a single N1 atom would complex two Ni atoms in a linear manner.

The above described way of defect formation in an idealized basic structure $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ gives the new formula $(\text{NH}_4)_{2-2z}[\text{Ni}(\text{NH}_3)_2]_z[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$. In the following the X-ray diffraction data of Ref. [1] are re-evaluated in terms of this newly suggested, alternative structure model and compared with the original model.

3. Re-evaluation of the single-crystal X-ray diffraction data of the *title compound*

In order to estimate whether the alternative structure model described in Section 1 is compatible with the single-crystal X-ray diffraction data [1], or whether it gives even better fits than the $(\text{NH}_4)_2(\text{NH}_3)[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ model, new structure refinements were performed using SHELXL-97 [6]. First, the $4c$ site occupied by N3 in Ref. [1] is occupied by Ni2 with variable occupancy. Having made this refinement it was possible to find reasonable sites for hydrogen by inspecting residual electron density maps. Therefore, a second refinement for the alternative structure model was performed, now including additionally hydrogen: For each N2 the difference Fourier maps show four equivalent maxima located at an xxz site. In order to impose the correct number of H2 atoms attached to N2 the occupancy was fixed to $\frac{3}{4}$. For N1 eight maxima on the general position xyz , forming approximately a cube around N1 occur. If N1 was NH_4^+ an occupancy of $\frac{1}{2}$ would result. If, however, a Ni2 atom is inserted, two NH_4^+ units have to become NH_3 . From that it can be shown that the occupancy of H1 is $\text{occ}(\text{H1}) = \frac{1}{2} - \text{occ}(\text{N2})/4$.

Table 1
Structural data of the title compound^a

Atom, xyz	Wyckoff symbol		Original model, Ref. [1] 4c occupied by N3 occupancy fixed to 0.5	Alternative model 4c occupied by Ni2	Alternative model 4c occupied by Ni2 including H atoms ^b
Ni1, 000	2a	U_{11} (Å ²)	0.0228(4)	0.0223(5)	0.0226(3)
		U_{33} (Å ²)	0.0158(5)	0.0154(6)	0.0156(4)
		U_{eq} (Å ²)	0.0204(4)	0.0200(4)	0.0202(3)
Cl, xx0	8h	x	0.22915(7)	0.22916(6)	0.22913(5)
		U_{11} (Å ²)	0.0288(4)	0.0285(4)	0.0287(3)
		U_{33} (Å ²)	0.0248(5)	0.0244(5)	0.0246(3)
		U_{12} (Å ²)	−0.0067(3)	−0.0068(3)	−0.0067(2)
		U_{eq} (Å ²)	0.0275(4)	0.0271(3)	0.0273(3)
N1 (NH ₄ ⁺), 0 $\frac{1}{2}$ $\frac{1}{4}$	4d	U_{11} (Å ²)	0.030(1)	0.030(1)	0.0290(9)
		U_{33} (Å ²)	0.023(1)	0.023(2)	0.023(1)
		U_{eq} (Å ²)	0.0278(8)	0.00274(8)	0.0270(6)
N2 (NH ₃), 00z	4e	z	0.2584(4)	0.2587(4)	0.2585(3)
		U_{11} (Å ²)	0.028(1)	0.027(2)	0.0265(8)
		U_{33} (Å ²)	0.017(2)	0.017(2)	0.018(1)
		U_{eq} (Å ²)	0.00245(8)	0.0239(8)	0.0235(6)
N3 (NH ₃) or Ni2, 0 $\frac{1}{2}$ 0	4c	U_{11} (Å ²)	0.028(4)	0.032(6)	0.026(3)
		U_{22} (Å ²)	0.016(3)	0.019(7)	0.016(3)
		U_{33} (Å ²)	0.019(3)	0.022(6)	0.020(3)
		U_{eq} (Å ²)	0.021(1)	0.024(2)	0.021(2)
		<i>occ</i> (N3/Ni2)	0.5(-), (N3)^c	0.081(3) (Ni2)	0.076(3) (Ni2)
Residuals for $I > 2\sigma(I)$		R_1, wR_2	0.027, 0.0745	0.0226, 0.0631	0.0184, 0.0457
Residuals for all data		R_1, wR_2	0.0263, 0.0751	0.0232, 0.0637	0.0190, 0.0453
Max/min residual density (e/Å ³)			0.341, −0.622	0.351, −0.583	0.206, −0.483

Space group $I4/mmm$, $a = 7.709(1)$ Å, $c = 7.942(2)$ Å, fractional coordinates, atomic displacement parameters (trace of the U_{ij} tensor), relative occupation factors and residuals. Besides the original model [1], i.e. N3 on 4c site, the results for alternative models assuming Ni2 on the 4c site are presented, without and with considering hydrogen atoms are listed.

^aBased on diffraction data measured on a single crystal at ambient temperatures using an IPDS (Stoe) equipped with MoK α radiation. The data were corrected for absorption using the crystal shape [1]. 1955 original reflections (1755 with $I > 2\sigma(I)$) were averaged according to the $4/mmm$ Laue symmetry giving 190 independent (187 with $I > 2\sigma(I)$) reflections and internal R values of $R_{int} = 4.02$ (1.85 for $I > 2\sigma(I)$). Refinement was performed on the basis on F^2 . For more details see Ref. [1].

^bHydrogen sites: H1 $32o$ 0.424(5), 0.060(5), 0.193(7), $occ(H1) = 1/2 - occ(Ni2)/4$, $U_{iso}(H1) = 0.08(2)$ Å², H2 $16m$ 0.074(3), 0.074(3), 0.298(8), $occ(H2) = 3/4$, $U_{iso}(H2) = 0.08(2)$ Å².

^cFree refinement gives a composition $(NH_4)_2(NH_3)_{0.77(5)}[Ni(NH_3)_2Cl_4]$ [1].

4. Discussion

The results of the refinements for the alternative model (without and with hydrogen) are listed in Table 1 in comparison with the data from Ref. [1]. Refinement of the alternative structure model without H shows, that it is fully compatible with the diffraction data presented in Ref. [1]. The residuals are even slightly better than for the original model. Taking into account additionally hydrogen on disordered positions around N1 and N2 gives a further, considerable improvement of the residuals. The arrangements of the disordered hydrogen atoms are comparable with those of NH₃ and NH₄⁺ in compounds with similar local environments

like Mg(NH₃)₂Cl₂ [7,8] or NH₄Cl [9], and will, therefore, not be discussed in detail here. The results of the refinement of the alternative model including hydrogen are used to calculate interatomic distances (Table 2).

Although the coordination spheres of Ni1 and Ni2 are basically quite similar, the Ni1–Cl and Ni2–Cl distances seem to differ considerably (2.50 Å vs. 2.74 Å). However, if on a certain 4c position a Ni2 atom is inserted, only small shifts are required for Cl (and also N) to achieve reasonable distances. This could be shown by test refinements (not reported here) with a split atom model for Cl and imposed ‘sensible’ Ni–Cl distances (using bond length restraints).

Table 2

Interatomic distances (Å) resulting from the refinement based on the alternative structural model (see Table 1) including hydrogen atoms

Ni1–4 × Cl	2.4981(7)
2 × N2	2.055(3)
Ni2–4 × Cl	2.7351(5)
2 × N1	1.9873(4)
N1 ^a –2 ^b × Ni2	1.9873(4)
8 × H1	0.87(4)
N2 ^c –1 × Ni1	2.055(3)
4 × H2	0.87(3)

^a Mixture of NH₄⁺ and NH₃ depending on Ni2 content.

^b Both these Ni2 sites cannot be occupied at the same time (see text).

^c NH₃ group.

The success of the refinement of the alternative model without and with hydrogen as well as the resulting chemically reasonable environment of Ni2 indicate that the alternative interpretation of the diffraction data given in this paper should be favored compared to the original model [1].

The ‘way’ from idealized (NH₄)₂[Ni(NH₃)₂Cl₄] to Ni(NH₃)₂Cl₂ (see Section 1) as well as the proposed type of disorder present in the *title compound* can be rationalized as proposed previously for some phases occurring in the quasi-binary system Mn(H₂O)₂Cl₂–NH₄Cl [2]. For that system the crystal structures of the two boundary phases and an intermediate phase are characterized by a primitive cubic arrangement of Cl atoms and can be constructed from two different building units, CsCl type ‘NH₄Cl units’ (Fig. 2a) and ‘Mn(H₂O)₂Cl₂ double units’ (Fig. 2b). The latter can replace a face-sharing pair of ‘NH₄Cl units’. In pure NH₄Cl the unit cell directly corresponds to a ‘NH₄Cl unit’. Random replacements of pairs of ‘NH₄Cl units’ by ‘Mn(H₂O)₂Cl₂ double units’ explains the observed solid solubility in NH₄Cl. The intermediate phase (NH₄)₂[Mn(H₂O)₂Cl₄] represents an ordered arrangement of both types of building units. (NH₄)₂[Mn(H₂O)₂Cl₄] shows itself a solid solubility for NH₄Cl, which can be achieved by substituting ‘Mn(H₂O)₂Cl₂ double units’ by pairs of ‘NH₄Cl units’. Finally, Mn(H₂O)₂Cl₂ is built only from ‘Mn(H₂O)₂Cl₂ double units’ arranged in a specific fashion [10].

Obviously, there are strong structural similarities of the quasi-binary system Ni(NH₃)₂Cl₂–(NH₄)Cl with the system Mn(H₂O)₂Cl₂–NH₄Cl: Ni(NH₃)₂Cl₂ has the same arrangement of ‘Ni(NH₃)₂Cl₂ double units’ as ‘Mn(H₂O)₂Cl₂ double units’ in Mn(H₂O)₂Cl₂.¹ The basic structure of the (NH₄)₂[Ni(NH₃)₂Cl₄] *title compound* corresponds basically to (NH₄)₂[Mn(H₂O)₂Cl₄]. The observed disorder can be described

¹ Mn(H₂O)₂Cl₂ is monoclinic, C2/m, due to hydrogen bonding [10], whereas Ni(NH₃)₂Cl₂ is orthorhombic, Cmmm; see Section 2.

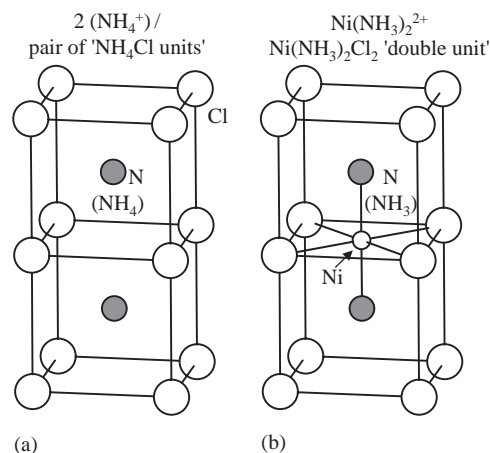


Fig. 2. (a) Face-sharing pair of CsCl type ‘NH₄Cl units’. Inserting a Ni²⁺ ion in the middle of the common face and replacing two NH₄⁺ by two NH₃ leads to a ‘Ni(NH₃)₂Cl₂ double unit’ (‘Mn(H₂O)₂Cl₂ double unit’ in the system Mn(H₂O)₂Cl₂–NH₄Cl [2]). (b) As a whole, a ‘Ni(NH₃)₂Cl₂ double unit’ can replace a pair of ‘NH₄Cl units’ which is proposed here to be that structural feature which is responsible for the additional electron density on the 4c site of the *title compound*.

by substituting face-sharing pairs of ‘NH₄Cl units’ by ‘Ni(NH₃)₂Cl₂ double units’.

Previously, the concept of ‘M(NH₃)₂X₂ double units’ (M: divalent metal, X: halogen) as building units of different crystal structures has been used (at that time without knowledge of Ref. [2]) to analyze the topological differences between the structure types of Cd(NH₃)₂Cl₂ [5] and Mg(NH₃)₂Br₂ ([4,7], the ‘M(NH₃)₂X₂ double units’ were termed ‘CsCl double units’ in these references). Furthermore, it was also pointed out [7] that the crystal structure of Hg(NH₃)₂Cl₂ [5] can be regarded as a disordered random arrangement of such ‘M(NH₃)₂Cl₂ double units’.

In literature further examples exist, where the concept of ‘NH₄Cl units’ and certain ‘double units’ was (or could have been) used to rationalize crystal structures. Besides the solubility of Mn(H₂O)₂Cl₂ in solid NH₄Cl [2], e.g. Cu²⁺ can be dissolved in solid NH₄Cl (precipitated from aqueous solution) in the form of ‘Cu(NH₃)(H₂O)Cl₂’ and ‘Cu(NH₃)₂Cl₂ double units’ [11], as supported by electron paramagnetic resonance measurements. Similarly, the solubility of Fe³⁺ in NH₄Cl suggested the presence of ‘Fe(H₂O)(OH)Cl₂ double units’ [12]. High metal concentrations in NH₄Cl were observed for ‘(NH₄)₂[Cu(NH₃)₂Cl₄]’ [13], which has a primitive cubic unit cell similar to that of NH₄Cl with *a* = 3.91(1) Å (*a* = 3.878 Å for pure NH₄Cl [14]) with *Pm* $\bar{3}$ space group symmetry, which should, however, be obviously *Pm* $\bar{3}m$. A similar primitive cubic unit cell was found for (NH₄)_{1.4}[Cu(NH₃)₂]_{0.3}Br₂ [15]. Single crystals of apparent CsCl type NH₄I were obtained at room temperature by reaction of NH₄I with Mn (molar ratio 1:2) at 350°C (A. Leineweber, H. Jacobs,

unpublished). This seemed remarkable, because pure NH_4I has the NaCl type structure at ambient temperatures and pressures [16]. Crystal structure analysis indicated, however, a similar structure as for the above mentioned Cu compounds, where the Mn content indicated a composition very close to $\text{Mn}(\text{NH}_3)_2\text{I}_2$ like in $\text{Hg}(\text{NH}_3)_2\text{Cl}_2$ (A. Leineweber, H. Jacobs, unpublished). The same batch, however, could contain at the same time orthorhombic $\alpha\text{-Mn}(\text{NH}_3)_2\text{I}_2$ with $\text{Mg}(\text{NH}_3)_2\text{Br}_2$ type structure.

5. Summary

Previously analyzed X-ray diffraction data on a single crystal of a so-called ' $(\text{NH}_4)_2(\text{NH}_3)[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ ' [1] were re-interpreted suggesting a modified formula $(\text{NH}_4)_{1.696}[\text{Ni}(\text{NH}_3)_2]_{0.152}[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$. Instead of interstitial NH_3 molecules, which would lead to unreasonably short distances to neighboring N atoms, it is supposed that starting from an idealized ' $(\text{NH}_4)_2[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ ' additional Ni^{2+} ions are inserted into interstices, distorting slightly the environment and transforming two adjacent NH_4^+ to NH_3 which then complex the new Ni^{2+} leading to a reasonable *trans*- $[\text{Ni}(\text{NH}_3)_2\text{Cl}_4]$ coordination.

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Further details of the crystal structure investigation(s) can be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: +49-7247-808-666; e-mail: crysdata@fiz.karlsruhe.de) on quoting the depository number CSD 413140 (alternative structure model including H atoms).

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