**Phosphaorganic Frameworks**

**Activation of Di-tert-butyldiphospatetrahedrane: Access to (tBuCP)_n (n = 2, 4) Ligand Frameworks by P–C Bond Cleavage**

Gabriele Hierlmeier and Robert Wolf*

*In memory of François Mathey

**Abstract:** The first mixed phosphatetrahedranes were reported only recently and their reactivity is virtually unexplored. Herein, we present a reactivity study on di-tert-butyldiphospatetrahedrane (1), which is the dimer of tert-butyolphosphaalkyne. The (tBuCP)_2 tetrahedron is activated selectively by N-heterocyclic carbene (NHC) nickel(I) and nickel(0) complexes, resulting in novel complexes featuring diverse (tBuCP)_n-frameworks (n = 2, 4). Release of the (tBuCP) framework from one of the complexes was achieved by addition of CO gas. Furthermore, 1 can be used as a source for P₂-units by elimination of di-tert-butylacetylene in the coordination sphere of nickel.

Tetrahedranes have long fascinated the chemical community due their simple structure and typically high reactivity. The first tetrahedral molecule to be prepared was white phosphorus, P₄, which was discovered as early as 1669, although its tetrahedral structure was recognised only in the early 20th century. P₄ is produced on a megaton scale each year and used as the common building block for incorporation of P atoms into organophosphorus compounds. The heavier homologue As₄ and the mixed interpnictogen compound Sb₄As₄P₃ are likewise accessible. Purely carbon-based tetrahe- dranes are also well-known, in line with the diagonal relationship between carbon and phosphorus in the periodic table. Indeed, the synthesis of (tBuC)₄ in 1978 was a pinnacle of organic synthesis.

In contrast, the first mixed C/P tetrahedranes were only reported last year. We showed that the “hybrid” of (tBuC)₄ and P₄, di-tert-butyldiphospatetrahedrane (tBuCP)₂ (1), can be synthesised in a simple nickel-catalysed dimerisation reaction of tert-butyolphosphaalkyne, tBuCP. Shortly after our report, the synthesis of the related tri-tert-butyldiphospatetrahedrane (tBuC)₄P was published by Cummins and co-workers. Given the similar molecular structures and isolobal relationship between 1 and P₄, a comparison of the reaction properties of both molecules is a tantalising prospect. Indeed, the activation of the P₄ tetrahedron by main group and transition metal complexes has attracted significant interest as a means of gaining control over its transformations, and of accessing fascinating new polyphosphorus compounds. In one of our recent contributions to this area, we demonstrated the use of mononuclear cyclopentadienyl nickel(I) complexes [CpNi(NHC)] [NHC = IMes (1,3-bis(2,6-diisopropylphenyl)imidazolin-2-ylidine), IPr (1,3-bis(2,6-diisopropylphenyl)imidazolin-2-ylidine)] as “nickel(1) radicals” selectively break one P–P bond of P₄ to afford µ-P₃-bridging P₃⁺ ligands with a “butterfly”-type structure (Figure 1, centre left). Subsequent studies demonstrated that P₄ activation by Ni₉–NHC complexes affords unusual di- and trinuclear cluster compounds, including a closo-[Ni₉⋅⋅⋅NHC]₃ cluster shown in Figure 1 (bottom left). Here, we describe a study into the reactivity of diphapatetrahedrane 1 with the same N-heterocyclic carbene (NHC) nickel(I) and nickel(0) complexes. These investiga-

---

**Figure 1.** (Mixed) group 14/15 tetrahedranes and reactivity of P₄ with NHC-stabilised Ni(I) and Ni(0) complexes.([9],[10])

---

[1] G. Hierlmeier, Prof. Dr. R. Wolf  
Universität Regensburg, Institut für Anorganische Chemie  
93040 Regensburg (Germany)  
E-mail: robert.wolf@ur.de

[9] Supporting information and the ORCID identification number(s) for the author(s) of this article can be found under: https://doi.org/10.1002/anie.202015680

[10] © 2021 The Authors. Angewandte Chemie International Edition published by Wiley-VCH GmbH. This is an open access article under the terms of the Creative Commons Attribution Non-Commercial License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited and is not used for commercial purposes.
tions have led to the preparation of polynuclear nickel complexes, which incorporate a variety of (tBuCP)_n (n = 2, 4) ligands in unusual coordination modes that are not known to be accessible using other synthetic precursors. We furthermore discuss the follow-up chemistry and remarkable thermal transformation of one of these compounds. The results of this work provide a valuable first insight into the reaction behaviour of 1 toward transition metal centres and reveal that its reaction patterns are clearly distinct from those of P_4 and, indeed, also from those of tBuCP, the monomer of 1.

This investigation commenced with the nickel(I) radical [CpNi(IPr)], which had previously been shown to react with white phosphorus under P–P bond scission (see Figure 1). Reaction of two equivalents of [CpNi(IPr)] with 1 in THF instantaneously afforded a deep red solution at room temperature (Scheme 1). Analysis of the crude reaction mixture by ^1H NMR spectroscopy revealed consumption of the paramagnetic starting material and release of IPr. A singlet resonance at 93.0 ppm was observed in the ^31P{^1H} NMR spectrum. Crystals grown from toluene were characterised by single-crystal X-ray analysis (SCXRD) as the tetranuclear complex [CpNi(tBu_2C_2P_2)]_2 (2, Figure 2, left) containing two 1,2-diphosphacyclobutene-1,2-diide ligands coordinated by four (CpNi) units in a realgar-type fashion. Notably, the reaction with [CpNi(IPr)] induces P–C bond cleavage to give a metalated 1,2-diphosphacyclobutene rather than forming the isomeric “butterfly” compound [(CpNi(IPr)]_2(μ-κ^2-P-tBu_2C_2P_3)]. Such a complex was observed with the valence isoelectronic P_4 molecule, see Figure 1.[9] In agreement with this, DFT calculations of the hypothetical reaction of 1 with two equivalents of a radical (methyl radical or [CpNi(IPh)]) (IPh = 1,3-diphenylimidazol-2-ylidene) suggest that the cyclobutene compound is thermodynamically preferred over the butterfly compound (see Figure S50 and S51 in the SI).

Structurally characterised 1,2-diphosphacyclobutadiene transition metal complexes are scarce, and the ligand commonly coordinates via the π-system in these examples.[13] The P1–P2 bond length in 2 (2.2244(7) Å) suggests the presence of a P–P single bond, whereas the C–C bond length of 1.360(3) Å is indicative of a C=C double bond.[12] The bond metric data is thus consistent with the presence of a dianionic (tBu_2C_2P_2)^2~ ligand, which coordinates in a μ^1,η^1,η^1-mode through the lone pairs of the P atoms, which was previously unknown for this type of ligand. Dark red, crystalline 2 was isolated in 56% yield and further analysed by NMR spectroscopy. The ^1H NMR spectrum of 2 showed one signal for the tBu groups (δ = 1.32 ppm) and one resonance for the cyclopentadienyl ligand (δ = 5.17 ppm). In the ^13C-[^1H] NMR spectrum four resonances were observed, which are consistent with the presence of a Cp and a (tBu_2C_2P_2)^2~ ligand. The UV/Vis absorption spectrum of 2 reveals two bands at 400 and 520 nm, the latter accounting for its red colour.

Next, we assessed the reactivity of nickel radicals bearing bulkier pentamethylcyclopentadienyl (Cp*) and pentaarylcylopentadienyl (CpBIG) ligands (Scheme 1).[13] As with the...
previous reaction, deep red solutions were obtained when a 1:2 stoichiometry was used. However, analysis by \(^1\)H NMR spectroscopy revealed broad resonances corresponding to unconsumed paramagnetic starting material, which disappeared upon addition of another equivalent of I.

Single-crystal X-ray diffraction studies revealed the formation of dinuclear species \([\text{Cp}^*\text{Ni}(\text{Bu}_4\text{C}_4\text{P}_4)]\) \([\text{Cp}^* = \text{Cp}^*\text{Bu}^\text{rg} (3b)]\), where two molecules of I have formally undergone radical coupling to produce a \(\beta\text{Bu}_4\text{C}_4\text{P}_4\) moiety (Scheme 1, bottom). Notably, however, compounds 2 and 3 were not formed upon reaction of the nickel complexes with the known ladderane-type phosphaalkyne tetramer (\(\beta\text{Bu}_4\text{C}_4\text{P}_4\)) (6), which is the known dimerisation product of I.\([6,14]\) This illustrates the value of I as a synthetic precursor to novel P/C ligand frameworks. Compounds 3a and 3b were isolated in 33% (3a) and 44% yield (3b) as dark-red, crystalline solids. Single-crystal X-ray diffraction revealed a three-rung ladder structure composed of two Ni and four P atoms, which is fused with two four-membered P\(_2\)C\(_2\) heterocycles. The P–P bond lengths range from 2.1717(9) to 2.2252(5) \(\AA\), with the shortest bond length for the \(\text{P}_2\)C\(_2\) rings. Analysis of 3a and 3b by \(^1\)H NMR spectroscopy reveals a selective reaction and two pseudo-triplet resonances at \(-9.9\) ppm and \(299.1\) ppm \((J = 40.3\) Hz), which is reminiscent of the \((\beta\text{Bu}_4\text{C}_4\text{P}_4)\) framework in compounds 3a/b. An X-ray diffraction experiment confirmed the presence of \([([\text{IMes}]\text{Ni})_2(\beta\text{Bu}_4\text{C}_4\text{P}_4)]\) (4), a dinuclear complex featuring a Ni–Ni bond and a \(\beta\text{Bu}_4\text{C}_4\text{P}_4\) moiety (Figure 3). The P–P bond lengths for the bonds within the diphosphacyclobutene ring are significantly longer compared to 3a/b, with 2.6304(7) (P1–P2A) and 2.6702(7) \(\AA\) (P3A–P4), whereas the exocyclic P2A–P3A bond is similar to that in 3a/b (2.2445(7) \(\AA\)). The Ni1–Ni2 bond length of 2.4293(4) \(\AA\) compares well to other nickel(I) dimers (e.g. \([([\text{IPr}]\text{Ni})_2(\mu-\text{CP})(\mu-\text{Cl})]\)).\([16]\)

![Figure 3. Dimerisation of 1 in the coordination sphere of \([([\text{IMes}]\text{Ni})_2\text{Bu}_4\text{P}_4\) (top) and molecular structure of 4 in the solid state (bottom).\([19]\)](Image)

Thermal ellipsoids are set at 50% probability level. Hydrogen atoms and solvent molecules and a minor disordered component (P2B and P3B) are omitted for clarity.

The bonding situation of the truncated model complex 4’ \([[(\text{IPh})\text{Ni}]_2(\text{P}_3\text{C}_3\text{Bu}_4)]\) was analysed by means of intrinsic bond orbitals (IBO) on the BP86/det2-TZVP level of theory. Two of these orbitals show multicentre bonds between Ni1/P1/P2A and Ni2/P3A/P4 (see Figure S52 in the SI for a depiction of the IBOs), which is reminiscent of the trinuclear Wade clusters we obtained from a similar reaction with P4.\([16]\) Moreover, the Mayer bond indices for the P–P bonds are small with values of \(<0.1\) and 0.11 for the P1–P2A and P3A–P4 bonds, suggesting only a weak interaction. The low Mayer bond index of 0.31 for the Ni1–Ni2 bond contradicts a strong covalent interaction. Compound 4 can be isolated in 28% yield by crystallisation. The \(^1\)H and \(^{13}\)C\([\text{H}]\) NMR spectra are in agreement with the structure determined by SCXRD, showing the expected signal sets for the IMes ligand and the \((\beta\text{Bu}_4\text{C}_4\text{P}_4)\) moiety. The \(^3\)P\([\text{H}]\) NMR spectrum is inconsistent with the asymmetrical structure of 4. However, we propose that the higher symmetry in the NMR spectrum results from a process which leads to an averaging of...
the signals for P1/P4 and P2/P3. Decoalescence was not observed upon cooling to 193 K. This suggests that the proposed fluxional process has a low activation barrier (see the SI for variable temperature NMR spectra).

Upon storage of solutions of 4 in C6D6 at room temperature overnight, 4 was partially converted into a new compound according to 1H and 31P{1H} NMR spectroscopy (Scheme 2, top). Heating a mixture of 1 and [(IMes)2Ni] to 60°C for three hours resulted in selective formation of this new species, which is characterised by two multiplet resonances in the 31P{1H} NMR spectrum at chemical shifts of 115.8 and 209.8 ppm (see the SI for a simulation). X-ray diffraction analysis of a single crystal grown from saturated n-hexane solutions revealed the formation of [(IMesNi)(P2)-\(\mu\)-Bu2C2P2]-IMes (5-IMes, Figure 4). The formation of 5 involves the elimination of di-tert-butylacetylene, which was identified by the 13C[1H] NMR resonance of the alkyne carbon atom detected at a chemical shift of 87.5 ppm. A related example of \(\mu\)-BuC≡C\(\mu\)-Cl elimination from a metal complex was recently reported by our group from a ruthenium complex,[7] but in contrast to that example, where the alkyne remains as \(\eta^2\)-bound ligand in the coordination sphere of ruthenium, di-tert-butylacetylene was completely liberated in the reaction reported herein, which illustrates an exciting potential for 1 and its coordination compounds to act as a source of P3 units.

Conveniently, dark brown crystals of 5 were isolated in 50% yield directly starting from [(IMes)2Ni] and 1. The compound initially co-crystallises with one equivalent of IMes (as compound 5-IMes). IMes can be subsequently removed by recrystallisation from toluene/n-hexane. The molecular structure of 5 in the solid state confirms the presence of a \(\mu\)-\(\eta^2\)-C2PTBu2 dumbbell and a \(\mu\)-\(\eta^2\)-C2P(\(\mu\)-\(\eta^2\)-P(\(\mu\)-\(\eta^2\)-P3)])-ligated. The P1–P2 bond of the latter ligand (2.4514(4) Å) is significantly elongated in comparison to 2 (2.2244(7) Å) and the P3–P4 bond length of the P2 ligand (2.0294(5) Å) is comparable to other complexes with a Ni-P2 core such as [(IMesNi(CO))2(\(\mu\)-\(\eta^2\)-C2P(\(\mu\)-\(\eta^2\)-P3))]. The 31P{1H} NMR spectrum of isolated 5 is identical to the species obtained from 4 (see above). The 1H NMR and 13C[1H] NMR spectra are in agreement with the proposed molecular structure of 5.

Further reactivity studies were conducted with complex 4 in order to examine the possible release and functionalisation of the \(\mu\)-Bu2C2P2 framework. Treatment of a solution of 4 in C6D6 with carbon monoxide (1 bar) results in an instantaneous colour change of the solution from brown to pale beige and formation of [(IMesNi(CO))2] and the known phosphaalkyne tetramer (\(\mu\)-BuCP), (6, Scheme 2) as corroborated by 1H and 31P{1H} NMR spectroscopy (see SI for spectra).[14] Moreover, the addition of hexachloroethane affords 6 and the chlorinated inverted sandwich complex [(IMesNiCl2(\(\mu\)- \(\mu\)-Bu2C2P2))] (7) as revealed by single-crystal X-ray crystallography (Figure 4). Complex 7 contains a 1,2-diphosphacyclobutadiene ligand in an unusual \(\mu\)-\(\eta^2\)-\(\eta^2\)-cooordination mode with a P1–P2 bond length of 2.2768(6) Å and a C1–C2 bond length of 1.437(3) Å. Unfortunately, 7 and its by-product, the dinuclear Ni11 complex [(IMesNiCl2(\(\mu\)-P2)], could not be separated due to similar solubilities. However, we were able to crystallise [(IMesNiCl2(\(\mu\)-Cl)] from the reaction mixture as violet blocks (see SI for SCXRD data).

In an attempt to evaluate the influence of the steric bulk of the NHC ligand, the related, bulkier [(IPr)2Ni] was reacted with one equivalent of 1 (Figure 5). However, 31P{1H} NMR spectroscopy suggested the formation of a different product with multiple resonances (see below). A similar spectrum was obtained when [(IPr)Ni(\(\eta^2\)-toluene)] was reacted with 1 (see SI for spectra). Single-crystal XRD on a crystal obtained from toluene revealed the formation of [(IPr)Ni(\(\mu\)-\(\mu\)-Bu2C2P2)] (8), that is, a mononuclear complex of the phosphaalkyne tetramer 6 (Figure 5).[14] The chemistry of the phosphaalkyne tetramer 6 is barely explored and, to the best of our knowledge, this is the first example of a coordination compound of 6. 8 was also obtained by reaction of [(IPr)Ni(\(\eta^2\)-toluene)] with 6 as a dark red solid and isolated in 35% yield. Considering the short time necessary (< 5 minutes) to form 8 from [(IPr)2Ni] and 1 in solution (much quicker than the known, “background” dimerisation of 1), we assume that coordinated 6 is formed upon dimerisation of 1 at the Ni atom.

![Scheme 2. Reactivity of 4 upon heating and addition of CO and hexachloroethane.](image)

![Figure 4. Molecular structures of 5 and 7 in the solid state.](image)
The molecular structure of \( \mathbf{8} \) reveals that the \((tBu)_4\) ligand coordinates through two lone pairs of adjacent P atoms to a (IPr)Ni fragment. As a consequence, the P1A–P2A bond is elongated (2.5821(7) Å) in comparison to the P3A–P4A bond (2.2020(8) Å, free 6: 2.219(1) and 2.236(1) Å).\(^{[14]}\) The \(^{31}\)P\([\text{H}]\) NMR spectrum of \( \mathbf{8} \) showed an AA’XX’ spin system at chemical shifts of −42.4 ppm and 102.1 ppm. The signal at high field is broad at room temperature and was resolved by variable-temperature NMR at −20°C (see the SI for spectra and simulation). The \(^1\)H and \(^{13}\)C\([\text{H}]\) NMR spectra of \( \mathbf{8} \) are in line with the molecular structure in the solid state.

In summary, reactions of di-tert-butylidiphosphaalkyne oligomers (1) with Ni\(^1\)–NHC and Ni\(^0\)–NHC complexes afford coordination compounds with unusual \((tBu)_4\) framework. Dimerisation of 1 on the nickel atom is a notable feature observed in several of these reactions. Moreover, elimination of di-tert-butylacetylene was observed from complex 5, which has stimulated further, ongoing studies on the use of 1 as a source for P\(_2\) fragments. It is noteworthy that complexes 2–4 and 8 cannot be obtained from reactions of the nickel precursors with \(tBuCP\). Moreover, a comparison with the previously described reactivity of the same complexes with \(tBuCP\) furthermore reveals the distinct reactivity of 1. These promising results bode well for the future development of 1 and related phosphatetrahedranes as sources for a plethora of other previously inaccessible phosphaorganometallic molecules. Work in this direction is underway.

**Acknowledgements**

Financial support by the Fonds der Chemischen Industrie (Kekulé Fellowship for G.H.) and the European Research Council (CoG 772299) is gratefully acknowledged. We thank Daniel Scott and Sebastian Bestgen for helpful comments on the manuscript. Open access funding enabled and organized by Projekt DEAL.

**Conflict of interest**

The authors declare no conflict of interest.

**Keywords:** bond activation · nickel · phosphaalkyne oligomers · phosphatetrahedranes · phosphorus

---

1. G. Maier, Angew. Chem. Int. Ed. Engl. 1988, 27, 309 – 332; Angew. Chem. 1988, 100, 317 – 341.
2. O. Reinmuth, J. Chem. Educ. 1928, 5, 1473.
3. D. E. C. Corbridge, Phosphorus 2000. Chemistry, Biochemistry and Technology, Elsesvier, Amsterdam, 2000.
4. a) B. M. Cossairt, M.-C. Diawara, C. C. Cummins, Science 2009, 325, 602; b) M. Seidl, G. Balázs, M. Scheer, Chem. Rev. 2019, 119, 8406 – 8434.
5. A. R. Jupp, J. C. Slootweg, Angew. Chem. Int. Ed. 2020, 59, 10698 – 10700; Angew. Chem. 2020, 132, 10786 – 10788.
6. G. Hierlmeier, P. Coburger, M. Bodensteiner, R. Wolf, Angew. Chem. Int. Ed. 2019, 58, 16918 – 16922; Angew. Chem. 2019, 131, 17074 – 17078.
7. M.-L. Y. Riu, R. L. Jones, W. J. Transue, P. Müller, C. C. Cummins, Sci. Adv. 2020, 6, eaa3168.
8. a) B. M. Cossairt, N. A. Piro, C. C. Cummins, Chem. Rev. 2010, 110, 4164 – 4177; b) M. Caporal, L. Gonsalvi, A. Rossin, M. Peruzzini, Chem. Rev. 2010, 110, 4178 – 4235; c) M. Scheer, G. Balázs, A. Seitz, Chem. Rev. 2010, 110, 4236 – 4256.
9. S. Pelties, D. Herrmann, B. de Bruin, F. Hartl, R. Wolf, Chem. Commun. 2014, 50, 7014 – 7016.
10. G. Hierlmeier, P. Coburger, N. P. van Velzen, S. Harder, R. Wolf, Angew. Chem. Int. Ed. 2020, 59, 14148 – 14153; Angew. Chem. 2020, 132, 14252 – 14257.
11. a) A. Chirila, R. Wolf, J. Chris Slootweg, K. Lammertsma, Coord. Chem. Rev. 2014, 270 – 271, 57 – 74; b) C. Jones, J. A. Platts, A. F. Richards, Chem. Commun. 2001, 663 – 666; c) S. Deng, C. Schwarzmair, M. Zabel, J. F. Nixon, M. Bodensteiner, E. V. Peresypkina, G. Balázs, M. Scheer, Eur. J. Inorg. Chem. 2011, 2991 – 3001; d) A. D. Burrows, A. Dransfeld, M. Green, J. C. Jeffery, C. Jones, J. M. Lynam, M. T. Nguyen, Angew. Chem. Int. Ed. 2001, 40, 3221 – 3224; Angew. Chem. 2001, 113, 3321 – 3324; e) F. W. Heinemann, S. Kummer, U. Seiss-Brandl, U. Zenneck, Organometallics 1999, 18, 2021 – 2029; f) C. Jones, C. Schulten, A. Stasch, Dalton Trans. 2006, 3733 – 3735; g) P. Binger, G. Glaser, S. Albus, C. Krüger, Chem. Ber. 1995, 128, 1261 – 1265; h) E.-M. Rummel, G. Balázs, V. Heinl, M. Scheer, Angew. Chem. Int. Ed. 2017, 56, 9592 – 9596; Angew. Chem. 2017, 129, 9720 – 9725.
12. a) B. Cordero, V. Gómez, A. E. Platero-Prats, M. Revés, J. Echeverría, E. Cremades, F. Barragán, S. Alvarez, Dalton Trans. 2008, 2832 – 2838; b) P. Pykkö, M. Atsumi, Chem. Eur. J. 2009, 15, 186 – 197; c) P. Pykkö, J. Phys. Chem. A 2015, 119, 2326 – 2337.
13. U. Chakraborty, F. Urban, B. Mühldorf, C. Rebreyend, B. de Bruin, N. van Velzen, S. Harder, R. Wolf, Organometalics 2016, 35, 1624 – 1631.
[14] B. Geissler, S. Barth, U. Bergsträsser, M. Slany, J. Durkin, P. B. Hitchcock, M. Hofmann, P. Binger, J. F. Nixon, P. von Raugé Schleyer, M. Regitz, Angew. Chem. Int. Ed. Engl. 1995, 34, 484–487; Angew. Chem. 1995, 107, 485–488.

[15] a) Y. Hoshimoto, Y. Hayashi, H. Suzuki, M. Ohashi, S. Ogoshi, Organometallics 2014, 33, 1276–1282; b) A. J. Arduengo III, S. F. Gamper, J. C. Calabrese, F. Davidson, J. Am. Chem. Soc. 1994, 116, 4391–4394.

[16] a) J. Wu, A. Nova, D. Balcells, G. W. Brudvig, W. Dai, L. M. Guard, N. Hazari, P.-H. Lin, R. Pokhrel, M. K. Takase, Chem. Eur. J. 2014, 20, 5327–5337; b) C.-Y. Lin, P. P. Power, Chem. Soc. Rev. 2017, 46, 5347–5399.

[17] C. Rödl, R. Wolf, Chem. Eur. J. 2019, 25, 8332–8343.

[18] G. Hierlmeier, A. Hinz, R. Wolf, J. M. Goicoechea, Angew. Chem. Int. Ed. 2018, 57, 431–436; Angew. Chem. 2018, 130, 439–444.

[19] Deposition Numbers 2043979 (2), 2043974 (3a), 2043977 (3b), 2043978 (4), 2043975 (5), 2043976 (7), 2043980 (8), and 2043981 ([IMes]Ni(Cl)(μ-Cl)) contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

Manuscript received: November 24, 2020
Accepted manuscript online: January 5, 2021
Version of record online: February 9, 2021