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Synthesis and Characterization of (R)-N-1-Cyclohexylethylamine Borane

Kevin Turani-I-Belloto,^[a] Dominique Granier,^[b] Pascal G. Yot,^[b] Johan G. Alauzun,^[b] and Umit B. Demirci^{*[a]}

A new solid amine borane is reported here. The reaction of (R)-(-)-1-cyclohexylethylamine (a liquid at room temperature) with borane dimethylsulfide yields crystals of pure (R)-N-1-cyclohexylethylamine borane $C_6H_{11}CH(CH_3)NH_2BH_3$ (CHEAB). The successful synthesis was confirmed by molecular analyses (FTIR, Raman and NMR). The crystals are in the form of millimeter-

sized translucent sticks, which allowed determining that CHEAB crystallizes in the orthorhombic system, the space group being $P2_12_12_1$. All of the characterization results, together with DFT calculations, strongly suggest the occurrence of intermolecular dihydrogen bonds between antiparallel NH_2BH_3 groups that are in a trimeric configuration.

Introduction

Dihydrogen bond (DHB) is an unconventional hydrogen bond as it consists of a proton-hydride electrostatic interaction involving an XH (X = N, O) bond and an YH (Y = B or a transition metal) bond.^[1] As such, the $H^{\delta+} \cdots H^{\delta-}$ distance is less than the sum of the Van der Waals radii for two H (i.e. 2.4 Å).^[2,3] DHB plays an important role in chemistry of amine boranes,^[4] as exemplified by two following applications.

Amine boranes are potential hydrogen storage materials.^[5] A typical representative is ammonia borane NH_3BH_3 . It is solid at temperatures lower than 90 °C because the molecules are stabilized by DHBs,^[6] and it dehydrogenates above this temperature by reaction of the interacting $H^{\delta+}$ and $H^{\delta-}$.^[7] Another example is 1,2-BN cyclohexane $C_4H_8NH_2BH_2$,^[8] which when heated in toluene up to 150 °C, undergoes dehydrocoupling of three molecules driven by intermolecular DHBs. This results in the release of H_2 and the formation of a borazine-linked trimer.

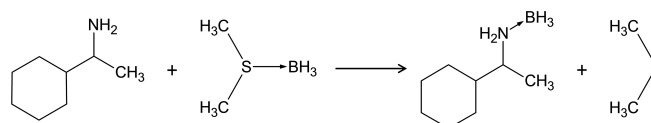
Amine boranes serve as precursors for the synthesis of (porous) polymers through dehydropolymerization (i.e. dehydrocoupling) driven by intermolecular DHBs.^[9] For example, polyaminoboranes $[-NHRBH_2-]_n$, which are isoelectronic to polyolefins, are produced from monoboranes like diisopropylaminoborane iPr_2NBH_2 (and in the presence of a primary amine RNH_2 , at -40 °C),^[10] and porous borazine-linked polymers are

produced from bisboranes like 1,4-phenylenediamine bisborane (at 120 °C).^[11] Like Chen et al.,^[12] we believe that one could take advantage of DHBs differently, and that DHBs could be applied in crystal engineering, molecular self-assembly, and synthesis of new advanced materials, for example. Our recent works are along these lines. For instance, we recently showed that ethane 1,2-diamineborane $C_2H_4(NH_2BH_3)_2$ can serve as precursor for the production of borocarbonitrides with specific surface areas up to $511 \text{ m}^2 \text{ g}^{-1}$ and being able to capture carbon dioxide (142 mg g^{-1} at 0 °C).^[13]

By synthesizing new amine boranes of formulae RNH_2BH_3 ,^[14] our objectives are to foster understanding of amine boranes' properties (possibly for opening up potential applications) and to show the existence of DHBs. Herein, and for the first time, is presented (R)-N-1-cyclohexylethylamine borane $C_6H_{11}CH(CH_3)NH_2BH_3$, coined as CHEAB thereafter. CHEAB is the first of our amine boranes that has produced single crystals upon synthesis (Figure 1).

Results and Discussion

CHEAB is obtained by Lewis acid-base reaction using (R)-(-)-1-cyclohexylethylamine (CHEA) and borane dimethylsulfide in diethyl ether, under argon atmosphere and at 20 °C (Scheme 1). The reaction is exothermic ($-57.7 \text{ kJ mol}^{-1}$; Figure S1). The low boiling points of both the solvent (b.p. 34.6 °C) and the dimethylsulfide product (b.p. 37.3 °C) make the isolation of CHEAB by vacuum distillation easy. The yield is then high, with > 99%. CHEAB is a white solid, in fact constituted of millimeter-



Scheme 1. Synthesis of CHEAB by reaction of (R)-(-)-1-cyclohexylethylamine with the borane dimethylsulfide complex in diethyl ether, under argon atmosphere and at 20 °C.

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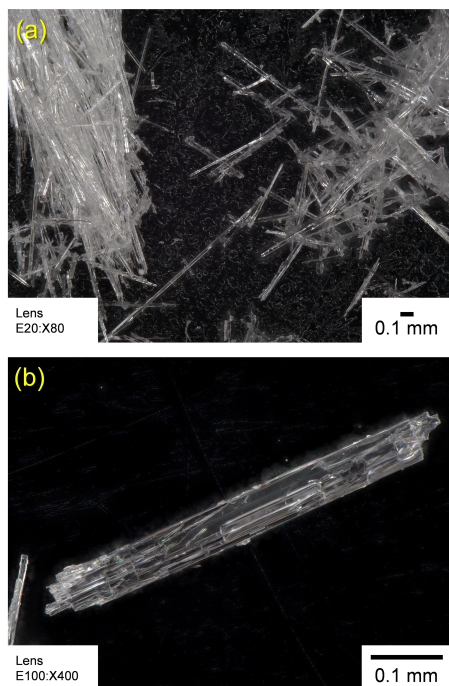


Figure 1. Digital micrographs of CHEAB at two different magnifications.

sized translucent sticks, whereas the precursor (R)-(–)-1-cyclohexylethylamine is liquid (at ambient temperature). It is thus reasonably safe to conclude that the solid state of CHEAB is the result of DHBs between NH_2 of one molecule and BH_3 of another one.

The formation of CHEAB was verified by FTIR spectroscopy (Figure S2). In comparison to the spectrum of the amine precursor, that of CHEAB shows three prominent differences. There are additional bands at $2500\text{--}2100\text{ cm}^{-1}$ due to the vibrations of the B–H bonds.^[15] The N–H vibration bands ($3450\text{--}3100\text{ cm}^{-1}$) are more intense and are all red shifted (by $50\text{--}130\text{ cm}^{-1}$), indicating a change in the N–H bond strength.^[16] The vibration band due to the B–N bond of the molecules is visible at 715 cm^{-1} . All this supports the formation of the NH_2BH_3 group, thus that of CHEAB, as well as the subsequent occurrence of intermolecular DHBs. The Raman spectra of CHEAB and of its amine precursor (Figure S3) allows making the similar observations and arriving at the same conclusions.

The ^{11}B MAS NMR spectrum of CHEAB in solid state (Figure S4) shows a single signal at -23.1 ppm in the form of a two-horned peak with a shoulder at low frequency. It is typical of an NBH_3 environment of a well crystallized amine borane.^[17] The ^{11}B NMR of CHEAB dissolved in CD_3CN (Figure S5) shows a single signal, a quartet at -20.5 ppm of normalized intensity $1:2.5:2.5:1$ and with a $^1J_{\text{B-H}}$ coupling constant of 94.9 Hz . This is typical of an NBH_3 environment.^[18] In summary, both spectra show signals consistent with the formation of CHEAB as a single product. The ^1H NMR spectrum of CHEAB is more complex (Figure S6). It shows a number of signals due to the various C–H bonds (featured by 2 multiplets and 1 doublet between 1 and 2.6 ppm, for 15 H atoms), the two N–H bonds (doublet at 3.8 ppm for 2 H atoms) and the three B–H bonds (multiplet at

$0.5\text{--}2\text{ ppm}$ for 3 H atoms), and confirms the successful formation of CHEAB. Note that, as analyzed by ^1H NMR, the product was isolated in sufficient purity (traces of the diethyl ether solvent).

Data collection was performed at -100 and 24°C as described in the experimental section and in the supporting information. The crystal data and refinement conditions are shown in Table 1.

The determined crystal system is orthorhombic, and the space group is $P2_12_12_1$ for both temperatures. The lattice parameters of the crystal at -100°C are as follows: $a = 5.7302(2)\text{ \AA}$, $b = 11.3236(5)\text{ \AA}$, $c = 15.0398(6)$, and $V = 975.88(7)\text{ \AA}^3$. The lattice parameters of the crystal at 24°C show a unit cell expansion that is anisotropic, such as $a = 5.7767(6)\text{ \AA}$ (0.8%), $b = 11.5440(15)\text{ \AA}$ (1.9%), $c = 15.0616(19)\text{ \AA}$ (0.1%), and $V = 1004.4(2)\text{ \AA}^3$ (2.9%). The structural model of CHEAB obtained at

Table 1. Space group, unit cell parameters, goodness of fit (GoF), R values, and structural parameters for the refined structure of CHEAB.

Empirical formula	$\text{C}_6\text{H}_{11}\text{CH}(\text{CH}_3)\text{NH}_2\text{BH}_3$	
Formula weight (g mol^{-1})	141.079	
Temperature (K°C)	173/–100	297/24
Wavelength (\AA)	1.54178	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions:		
a (\AA)	5.7302(2)	5.7767(6)
b (\AA)	11.3236(5)	11.5440(15)
c (\AA)	15.0398(6)	15.0616(19)
Cell volume (\AA^3)	975.88(7)	1004.4(2)
Z	4	
Calculated density (g cm^{-3})	0.960	0.933
Absorption coefficient (mm^{-1})	0.388	0.377
$F(000)$	320.726	320.726
Crystal size (mm)	0.308×0.092×0.08	0.226×0.082×0.071
θ range for data collection ($^\circ$)	4.89–70.09	4.83–70.12
Limiting indices	$-5 \leq h \leq 6$ $-13 \leq k \leq 13$ $-18 \leq l \leq 18$	$-6 \leq h \leq 5$ $-12 \leq k \leq 14$ $-12 \leq l \leq 18$
Reflexion collected	6976	3899
Completeness	$2\theta = 135.4^\circ$: 99.7%	$2\theta = 135.4^\circ$: 99.9%
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	1829/20/112	1857/20/172
Goodness-of-fit on F^2	1.095	1.003
Final R indices [$I > 2\sigma(I)$]	0.0444 and 0.1170	0.0527 and 0.1140
R indices (all data)	0.0451 and 0.1181	0.0778 and 0.1288
LDPH (e \AA^{-3}) ^[a]	0.15 and -0.17	0.14 and -0.14
Flack parameter	$-0.4(4)$	0.0(11)

[a] Largest diffraction peak and hole.

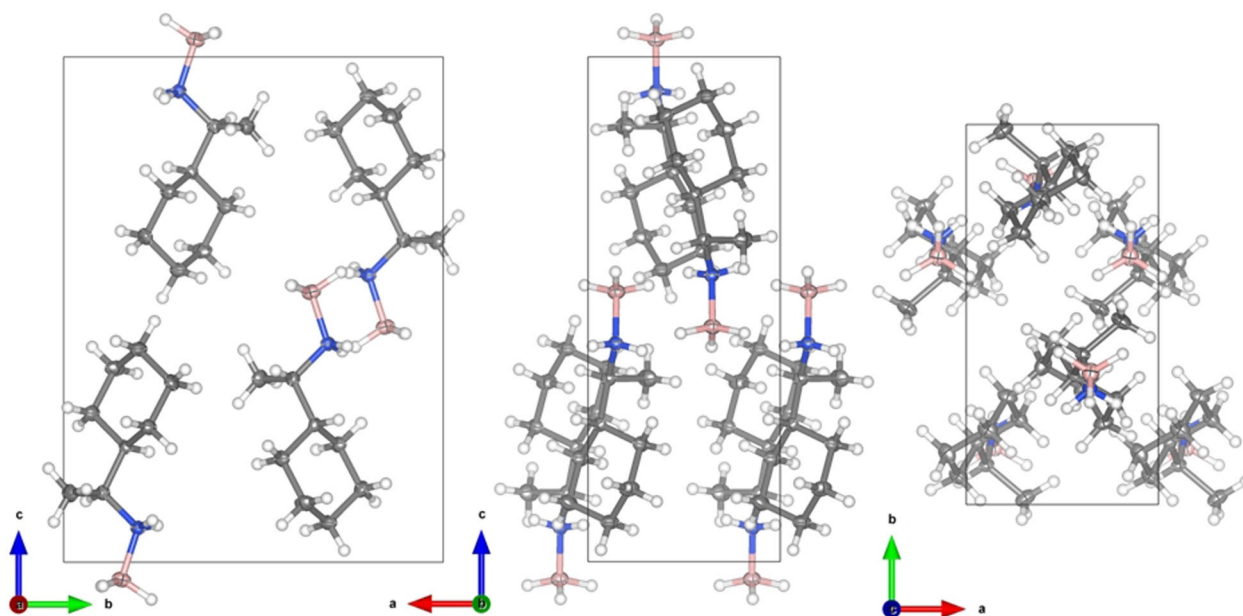


Figure 2. Crystal structure of CHEAB at $-100\text{ }^{\circ}\text{C}$, along the three crystallographic axes. The H, B, N and C atoms are represented by white, pink, blue and dark grey spheres, respectively.

$-100\text{ }^{\circ}\text{C}$ is shown in Figure 2. The CHEAB molecules are in close-packed arrangement into the unit cell where they are aligned along the c crystallographic axis. The B–N bond length is $1.6077(19)\text{ \AA}$ at $-100\text{ }^{\circ}\text{C}$ ($1.615(4)\text{ \AA}$ at $24\text{ }^{\circ}\text{C}$), which is slightly longer than, though consistent with, those reported for ammonia borane and linear alkylamine boranes.^[3,14] The CHEAB molecules give an antiparallel NH_2BH_3 trimeric configuration. In such a configuration (along the bc plane), NH_2 of one CHEAB molecule is facing BH_3 of another molecule, which is the intrinsic feature of DHB.^[1,12] The formation of CHEAB and the presence of DHBs between the molecules have been obtained by adding hydrogen atoms on the Q-peaks obtained by single X-ray structure elucidation. The two structural models obtained are quite similar. As it is seen on Table S1, the atomic coordinates obtained for both temperatures differ slightly. As a result, the bond lengths, consistent with the values observed in compounds of the same family (Table S3), present small differences. For example, the B–N is extended by 0.6% and at the difference, the C–N and C–C distances are contracted by 0.2% and 0.5% respectively. In addition, bond- and torsion-angles (Tables S4 and S5) determined from the two structural models are not affected by the temperature. For this antiparallel configuration, the two intermolecular B-to-N distances are $3.359(3)$ and $3.394(3)\text{ \AA}$ at $-100\text{ }^{\circ}\text{C}$ ($3.406(5)$ and $3.437(5)\text{ \AA}$ at $24\text{ }^{\circ}\text{C}$). They are consistent with distances found in amine boranes and amidoboranes.^[14,19] Another distance of interest is the one for parallel CHEAB molecules. It is $5.7302(1)\text{ \AA}$ at $-100\text{ }^{\circ}\text{C}$ ($5.7767(1)\text{ \AA}$ at $24\text{ }^{\circ}\text{C}$). The refinement procedure used and all the structural parameters for both temperatures are summarized in the SI file (Figure S7 and Tables S1 to S5).

By DFT, the total energy of CHEAB was calculated, and it is -354 Hartree. The Mulliken charges of the CHEAB atoms were extracted (Figure 3). The charge of the hydrogen atoms of NH_2

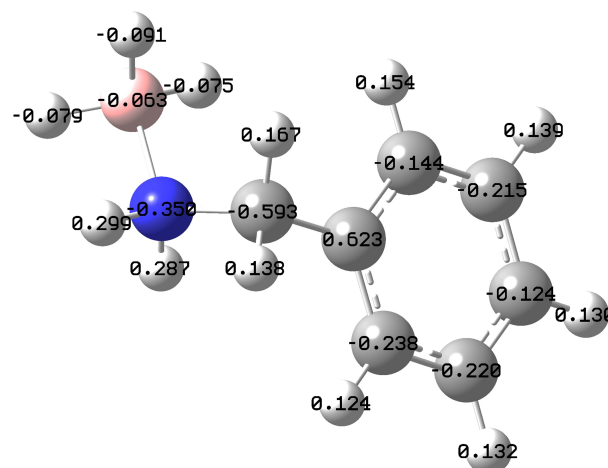


Figure 3. Mulliken charges of the elements of CHEAB.

is positive ($+0.298$ and $+0.299$), and the charge of the hydrogen atoms of BH_3 is slightly negative (between -0.091 and -0.071). This confirms the presence of both $\text{H}^{\delta+}$ and $\text{H}^{\delta-}$ in the CHEAB molecule, which is the necessary condition for the occurrence of intermolecular DHBs in solid state CHEAB.

The thermal stability of CHEAB was studied by DSC (Figure S8) and TG (Figure S9). CHEAB first melts, the onset temperature being $89\text{ }^{\circ}\text{C}$, and then decomposes. The mass loss at $218\text{ }^{\circ}\text{C}$ is 19.4 wt%, which is higher than the gravimetric hydrogen density of CHEAB (14.2 wt% H). Pure CHEAB is thus, like ammonia borane and more broadly amine boranes, not suitable for hydrogen storage,^[4,7,14] but it might have a potential if combined to the amine CHEA.^[20] Above $218\text{ }^{\circ}\text{C}$, a second decomposition (mass loss of 73.4 wt%) takes place. Various products (cyclohexene, ethylcyclohexene, methylcyclohexene,

ammonia, acetonitrile, methylcyclopentene, cyclopentene, methylcyclohexane, and cyclohexane) were detected, thereby suggesting a complex decomposition mechanism. The total mass loss at 600 °C is 96.2%. If one had to consider CHEAB as a possible ceramic precursor, the ceramic yields would be too low. CHEAB might however produce e.g. carbon-doped boron nitride in higher yields, provided a two-step process is applied as for the polymer-derived ceramics route (i.e. dehydrocoupling at < 250 °C to form a polymer, and pyrolysis at > 600 °C of the as-formed polymer).^[21]

Conclusions

In conclusion, we have synthesized pure CHEAB in high yield and in the form of (mono)crystals. CHEAB crystallizes into the orthorhombic structure (s.g. $P2_12_12_1$). The molecules are in close-packed arrangement into the unit cell, they are aligned along the *c* crystallographic axis, and they give an antiparallel NH_2BH_3 configuration where NH_2 of one molecule is facing BH_3 of another one. This is consistent with the existence of DHBs between the molecules. Like other amine boranes, pure CHEAB decomposes under heating and is not suitable for the hydrogen storage application. It however may have potential as a precursor for the production of polymers and/or carbon-doped boron nitride.

Experimental Section

In an argon-filled glove box (MBraun M200B; O_2 and H_2O < 0.1 ppm) and at ambient temperature, CHEAB is synthesized by reaction of 500 mg of (R)-(-)-1-cyclohexylethylamine ($\text{C}_6\text{H}_{11}\text{CH}(\text{CH}_3)\text{NH}_2\text{BH}_3$; 98% from Merck) in 8 mL of diethyl ether (anhydrous; Merck) and a volume of borane dimethylsulfide ($(\text{CH}_3)_2\text{S}\cdot\text{BH}_3$ at 5 M in diethyl ether; from Merck) so that the mol ratio is 1:1.1. The addition of the later is done dropwise, and the mixture is left under stirring for 24 h. Finally, the solvent and the product (Scheme 1) are removed by vacuum distillation, and the as-obtained white solid is transferred into an argon-filled vial and stored at -48 °C.

CHEAB was analyzed by digital microscopy (Keyence VHX-6000), Calvet calorimetry (C80 calorimeter from Setaram), differential scanning calorimetry (DSC 1 Mettler-Toledo), thermogravimetric (TG) analysis (TGA/DSC 2 Mettler Toledo) coupled gas chromatography (GC) and mass spectrometry (MS) detector (7890B GC-5977A MSD Agilent Technologies) such as TGA/microGC-MS (SRA Instrument) to follow H_2 and other small molecules and TGA/Storage-Interface/GC-MS for heavier volatile products, Fourier-transform infrared spectroscopy (FTIR; IS50 Thermo Fisher Scientific), Raman spectroscopy (Horiba Jobin Yvon LabRAM 1B; laser Ar/Kr 100 mW 647.1 nm), solid-state ^{11}B magic angle spinning (MAS) NMR spectroscopy (^{11}B MAS NMR; Varian VNMR4000; 128.378 MHz), ^{11}B NMR spectroscopy (Bruker AVANCE-400; probe head BBFO; CD_3CN (also containing CH_3CN and H_2O) in a sealed capillary; 5-mm tube; 128.378 MHz), ^1H nuclear magnetic resonance spectroscopy (^1H NMR; Bruker Avance-400 NMR; BBOF probe; CD_3CN in a capillary; 5-mm NMR tube). Details are also provided in the Supporting Information file.

The X-ray single-crystal structural data were collected at 173 and 297 K on a Bruker D8 VENTURE equipped with a PHOTON II CPAD detector and a microfocus X-ray source with Cu-K_α radiation ($\lambda =$

1.54178 Å) operating at 50 kV and 1 mA. A suitable crystal was chosen and mounted on a 30×300 μm MicroLoop. The single crystal X-ray diffraction allowed collecting 1829 frames. The total exposure times were 5.37 and 4.54 at 173 K and 297 K respectively. The frames were integrated with the Bruker SAINT Software package using a narrow-frame algorithm. 6976 reflections were measured in the angle range $9.78^\circ \leq 2\theta \leq 140.18^\circ$ at 173 K, whereas 3899 reflections were measured in the range $9.66^\circ \leq 2\theta \leq 140.24^\circ$ at 297 K. The crystal was kept at 173 and 297 K during data collection. Using Olex2, the structure was solved with the SHELXT structure solution program using Intrinsic Phasing and refined with the olex2.refine refinement package (see Supporting Information).^[22] Deposition Numbers 2246112 (for CHEAB at -100 °C), and 2246113 (for CHEAB at 24 °C) 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.

The Mulliken charges and the total energy of CHEAB was determined by density functional theory (DFT) calculations (gas phase geometry optimization using the DFT/B3LYP method with the 6-311++G (2d, p) basis set available in the Gaussian16 program). The optimized conformers were calculated at 25 °C.

Supporting Information Summary

The Supporting Information provides Figures S1 to S9 and Tables S1 to S6, and they concern the: enthalpy of reaction by Calvet calorimetry; FTIR, Raman, ^{11}B MAS NMR, ^{11}B NMR and ^1H NMR spectra; representation of the asymmetric unit of CHEAB; single X-ray structure elucidation of CHEAB including the standardized experimental structural parameters, the bonds lengths, the geometrical angles, the torsion angles and the intermolecular distances observed for the refined structure of CHEAB at -100 and 24 °C; onset temperature of the melting of CHEAB by DSC; and, TG and $\mu\text{GC-MS}$ analyses.

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Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords: amine borane · boranes · cyclohexylethylamine · dihydrogen bond

- [1] R. H. Crabtree, P. E. M. Siegbahn, O. Eisenstein, A. L. Rheingold, T. F. Koetzle, *Acc. Chem. Res.* **1996**, *29*, 348–354.
- [2] T. B. Richardson, S. de Gala, R. H. Crabtree, *J. Am. Chem. Soc.* **1995**, *117*, 12875–12876.
- [3] W. T. Klooster, T. F. Koetzle, P. E. M. Siegbahn, T. B. Richardson, R. H. Crabtree, *J. Am. Chem. Soc.* **1999**, *121*, 6337–6343.
- [4] A. Staubitz, A. P. M. Robverson, M. E. Sloan, I. Manners, *Chem. Rev.* **2010**, *110*, 4023–4078.
- [5] R. Kumar, A. Karkamkar, M. Bowden, T. Autrey, *Chem. Soc. Rev.* **2019**, *48*, 5350–5380.
- [6] K. M. Dreux, L. E. McNamara, J. T. Kelly, A. M. Wright, N. I. Hammer, G. S. Tschumper, *J. Phys. Chem. A* **2017**, *121*, 5884–5893.
- [7] A. Al-Kukhun, H. T. Hwang, A. Varma, *Int. J. Hydrogen Energy* **2013**, *38*, 169–179.
- [8] W. Luo, L. N. Zakharov, S. Y. Liu, *J. Am. Chem. Soc.* **2011**, *133*, 13006–13009.
- [9] A. L. Colebatch, A. S. Weller, *Chem. Eur. J.* **2019**, *25*, 1379–1390.
- [10] C. A. De Albuquerque Pinheiro, C. Rolland, P. Jehan, G. Alcaraz, *Angew. Chem. Int. Ed.* **2018**, *57*, 1519–1522.
- [11] K. T. Jackson, M. G. Rabbani, T. E. Reich, H. M. El-Kaderi, *Polym. Chem.* **2011**, *2*, 2775–2777.
- [12] X. Chen, J. C. Zhao, S. G. Shore, *Acc. Chem. Res.* **2013**, *46*, 2666–2675.
- [13] R. Mighri, U. B. Demirci, J. Alauzun, *Nanomaterials* **2023**, *13*, 734.
- [14] a) K. Turani-I-Belloto, M. J. Valero-Pedraza, R. Chiriac, F. Toche, D. Granier, D. Cot, E. Petit, P. G. Yot, J. G. Alauzun, U. B. Demirci, *ChemistrySelect* **2021**, *6*, 9853–9860; b) A. Theodoratou, K. Turani-I-Belloto, E. Petit, S. Dourdain, G. Alauzun, U. B. Demirci, *J. Mol. Struct.* **2022**, *1248*, 131484; c) K. Turani-I-Belloto, M. J. Valero-Pedraza, E. Petit, R. Chiriac, F. Toche, D. Granier, P. G. Yot, J. G. Alauzun, U. B. Demirci, *ChemistrySelect* **2022**, *7*, e202203533.
- [15] J. Smith, K. S. Seshadri, D. White, *J. Mol. Spectrosc.* **1973**, *45*, 327–498.
- [16] H. Wolff, G. Gamer, *J. Phys. Chem.* **1972**, *76*, 871–876.
- [17] R. Knitsch, M. Brinkkötter, T. Wiegand, G. Kehr, G. Erker, M. R. Hansen, H. Eckert, *Molecules* **2020**, *25*, 1400.
- [18] G. R. Eaton, *J. Chem. Educ.* **1969**, *46*, 547–556.
- [19] R. Owarzany, P. J. Leszczynski, K. J. Fijalkowski, W. Grochala, *Crystals* **2016**, *6*, 88.
- [20] G. Zhang, D. Morrison, G. Bao, H. Yu, C. W. Yoon, T. Song, J. Lee, A. T. Ung, Z. Huang, *Angew. Chem. Int. Ed.* **2021**, *133*, 11981–11835.
- [21] Y. Guo, Y. Sugahara, *Int. J. Appl. Ceram. Technol.* **2023**, *20*, 8–23.
- [22] a) O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, *J. Appl. Crystallogr.* **2009**, *42*, 339–341; b) G. M. Sheldrick, *Acta Crystallogr.* **2015**, *A71*, 3–8; c) L. J. Bourhis, O. V. Dolomanov, R. J. Gildea, J. A. K. Howard, H. Puschmann, *Acta Crystallogr.* **2015**, *A71*, 59–75.

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